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Raman spectroscopic study of the hydroxy-arsenate mineral geminite $Cu(AsO_3OH)\cdot H_2O$

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ABSTRACT

The mineral geminite, an hydrated hydroxy-arsenate mineral of formula $Cu(AsO_3OH)\cdot H_2O$, has been studied by Raman and infrared spectroscopy. Two minerals from different origins were investigated and the spectra proved quite similar. In the Raman spectra of geminite, four bands are observed at 813, 843, 853 and 885 cm^{-1}. The assignment of these bands is as follows: (a) The band at 853 cm^{-1} is assigned to the AsO_4^{3-} ν_1 symmetric stretching mode (b) the band at 885 cm^{-1} is assigned to the AsO_3OH^{2-} ν_1 symmetric stretching mode (c) the band at 843 cm^{-1} is assigned to the AsO_4^{3-} ν_3 antisymmetric stretching mode (d) the band at 813 cm^{-1} is ascribed to the AsO_3OH^{2-} ν_3 antisymmetric stretching mode. Two Raman bands at 333 and 345 cm^{-1} are attributed to the ν_2 AsO_4^{3-} bending mode and a set of higher wavenumber bands are assigned to the ν_4 AsO_4^{3-} bending mode. A very complex set of overlapping bands is observed in both the Raman and infrared spectra. Raman bands are observed at 2288, 2438, 2814, 3152, 3314, 3448 and 3521 cm^{-1}. Two Raman bands at 2288 and 2438 cm^{-1} are ascribed to very strongly hydrogen bonded water. The broader Raman bands at 3152 and 3314 cm^{-1} may be assigned to adsorbed water and not so strongly hydrogen bonded water in the molecular structure of geminate. Two bands at 3448 and 3521 cm^{-1} are assigned to the OH stretching vibrations of the $(AsO_3OH)^{2-}$ units. Raman spectroscopy identified Raman bands attributable to AsO_4^{3-} and AsO_3OH^{2-} units.

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KEYWORDS: geminite, acid-arsenate, arsenate, Raman spectroscopy

INTRODUCTION

Geminite [1-4] $Cu(AsO_3OH)\cdot H_2O$ is an acid arsenate with a triclinic structure normally found in the deposits of arsenic bearing Cu-Pb deposits. The structure of geminate has been published by Cooper and Hawthorne [2]. However to the best of our knowledge no vibrational spectroscopy of this mineral has been published. According to Cooper and Hawthorne, in the structure of geminate, there are four unique Cu^{2+} sites, each coordinated by six anions in a [4 + 2]-distorted octahedral arrangement [2]. There are four unique As^{5+} sites, each coordinated by three oxygen atoms and an OH group in a distorted tetrahedral arrangement. The three As-O distances are shorter than the As-OH distance in each tetrahedron, in according to the prediction of bond-valence theory [2]. The copper polyhedra share edges to form chains parallel to [010]; As.vphi.4 tetrahedra cross-link these chains into sheets parallel to (001), and there are two symmetrical distinct but topologically identical sheets in the structure. These sheets are linked into a three-dimensional structure by a network of H bonds, accounting for the perfect cleavage and platy habit of geminite.

The aim of this paper is to report the Raman spectra of geminite, and to relate the spectra to the molecular and crystal chemistry of this arsenate type mineral. The paper follows the systematic research on Raman and infrared spectroscopy of secondary minerals containing oxy-anions formed in the oxidation zone [5-16].

EXPERIMENTAL

Minerals

The mineral geminite originated from the Jáchymov (St Joachimsthal), Ostrov, Krusné Hory Mountains (Erzgebirge), Karlovy Vary Region, Bohemia (Böhmen; Boehmen), Czech Republic. A second sample was obtained from the cap Garonne Mine, France [4]. The chemical analysis of the geminite can be found in Anthony et al. (page 212) [17].the crystal structure of this mineral has been published [2].

Raman spectroscopy

Crystals and single crystal samples of geminite were placed on a polished metal surface on the stage of an Olympus BHSM microscope, which is equipped with 10x, 20x, and 50x objectives. The microscope is part of a Renishaw 1000 Raman microscope system, which also includes a monochromator, a filter system and a CCD detector (1024 pixels). The Raman spectra were excited by a Spectra-Physics model 127 He-Ne laser producing highly polarised light at 633 nm and collected at a nominal resolution of 2 cm^{-1} and a precision of ± 1 cm^{-1} in the range between 200 and 4000 cm^{-1}. Repeated acquisition on the crystals using the highest magnification (50x) were accumulated to improve the signal to noise ratio in the spectra. Spectra were calibrated using the 520.5 cm^{-1} line of a silicon wafer. Previous studies by the authors provide more details of the experimental technique. Alignment of all crystals in a similar orientation has been attempted and achieved. However, differences in intensity may be observed due to minor differences in the crystal orientation [9-16, 18-25].

Infrared spectroscopy

Infrared spectra of dussertite sample were recorded by micro diffuse reflectance method (DRIFTS) on a Nicolet Magna 760 FTIR spectrometer (range 4000-600 cm^{-1}, resolution 4 cm^{-1}, 128 scans, 2 level zero-filtering, Happ-Genzel apodization), equipped with Spectra Tech InspectIR micro FTIR accessory. Each sample of amount less than 0.050 mg was mixed without using pressure with KBr. Samples were immediately recorded together with the same KBr as a reference.

Spectral manipulation such as baseline correction/adjustment and smoothing were performed using the Spectracalc software package GRAMS (Galactic Industries Corporation, NH, USA). Band component analysis was undertaken using the Jandel 'Peakfit' software package that 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 Lorentzian-Gaussian cross-product function with the minimum number of component bands used for the fitting process. The Gaussian-Lorentzian 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

Arsenate vibrations

The free arsenate ion has tetrahedral symmetry and thus should have four bands of which two are infrared active with theoretical values of ν_3 (F_2) 887 cm^{-1} and ν_4 (F_2) 463 cm^{-1}. The Raman active modes are observed at 837 (A_1) and 349 (E) cm^{-1}. Upon coordination of the arsenate ion to the copper atom, then the symmetry of the arsenate ion reduces to C_{3v} and may further reduce to C_{2v}. The implication is that all bands will be both infrared and Raman active. The Raman spectra of the tetrahedral anions in aqueous systems are well known. The symmetric stretching vibration of the arsenate anion (ν_1) is observed at 810 cm^{-1} and coincides with the position of the asymmetric stretching mode (ν_3). The symmetric bending mode (ν_2) is observed at 342 cm^{-1} and the out-of-plane bending modes (ν_4) is observed at 398 cm^{-1}. Of all the tetrahedral oxyanions spectra, the positions of the arsenate vibrations occur at lower wavenumbers than any of the other naturally occurring mineral oxyanions spectra. Farmer lists a number of spectra of arsenates including the basic copper arsenates olivenite and euchroite. [26] The effect of the arsenate ion in a crystal will be to remove the degeneracy and allow splitting of the bands according to factor group analysis. The ν_1 and ν_3 infrared bands of olivenite and euchroite were observed at 860, 828, 790 cm^{-1} and 830 and 770 cm^{-1} respectively. The bending modes were found at 493 and 452 cm^{-1} for olivenite and at 475 and 410 cm^{-1} for euchroite. No ν_2 bands were shown. This is no doubt related to the fact the bands are found below 400 cm^{-1}, which makes the measurement by infrared spectroscopy difficult. Two OH stretching vibrations were observed at 3580 and 3440 cm^{-1} for olivenite. A number of bands were listed which were unassigned.

Raman spectroscopy of geminite

According to Myneni *et al.* [27, 28] and Nakamoto [29], $(AsO_4)^{3-}$ is a tetrahedral unit, which exhibits four fundamental vibrations: the ν_1 symmetric stretching vibration (A_1) 818 cm^{-1}, Raman active; the doubly degenerate ν_2 symmetric bending vibration (E) 350 cm^{-1}, Raman active; the triply degenerate ν_3 antisymmetric stretching vibration (F_2) 786 cm^{-1}, and the triply degenerate ν_4 bending vibration (F_2) 405 cm^{-1} , both infrared and Raman active. Protonation, metal complexation, and/or adsorption on a mineral surface should cause change

in $(AsO_4)^{3-}$ symmetry from T_d to lower symmetries, such as C_{3v}, C_{2v}or C_1. This loss of degeneracy causes splitting of degenerate vibrations of $(AsO_4)^{3-}$ and the shifting of the As-OH stretching vibrations to different wavenumbers. Such chemical interactions reduce $(AsO_4)^{3-}$ tetrahedral symmetry, as mentioned above, to either C_{3v}/C_3 (corner-sharing), C_{2v}/C_2 (edge-sharing, bidentate binuclear), or C_1/C_s (corner-sharing, edge-sharing, bidentate binuclear, multidentate) [27, 28]. In association with $(AsO_4)^{3-}$ symmetry and coordination changes, the A_1 band may shift to different wavenumbers and the doubly degenerate E and triply degenerate F modes may give rise to several new A_1, B_1, and/or E vibrations [27, 28]. In the absence of symmetry deviations, $(AsO_3OH)^{2-}$ in C_{3v} symmetry exhibit the ν_s As-OH and ν_{as} and ν_s $(AsO_3OH)^{2-}$ vibrations together with corresponding the δ As-OH in-plane bending vibration, δ As-OH out-of-plane bending vibration, ν_s $(AsO_3OH)^{2-}$ stretching vibration and δ_{as} $(AsO_3OH)^{2-}$ bending vibration [30-32]. Keller [30] assigned observed the following infrared bands in $Na_2(AsO_3OH)\cdot 7H_2O$ 450 and 360 cm^{-1} to the δ_{as} (ν_4) $(AsO_3OH)^{2-}$ bend (*E*), 580 cm^{-1} to the δ As-OH out-of-plane bend, 715 cm^{-1} to the ν As-OH stretch (A_1), 830 cm^{-1} to the ν_{as} $(AsO_3OH)^{2-}$ stretch (*E*), and 1165 cm^{-1} to the δ As-OH in plane bend. In the Raman spectrum of $Na_2(AsO_3OH)\cdot 7H_2O$, Vansant *et al.* [31] attributed observed Raman bands to the following vibrations 55, 94, 116 and 155 cm^{-1} to lattice modes, 210 cm^{-1} to ν (OH…O) stretch, 315 cm^{-1} to $(AsO_3OH)^{2-}$ rocking, 338 cm_{-1} to the δ_s $(AsO_3)^{2-}$ bend, 381 cm^{-1} to the δ_{as} $(AsO_3OH)^{2-}$ bend, 737 cm^{-1} to the ν_s As-OH stretch (A_1), 866 cm^{-1} to the ν_{as} $(AsO_3OH)^{2-}$ stretch (*E*).

The Raman spectra of the mineral geminite from Jáchymov, The Czech Republic, in the 650 to 1000 cm^{-1} region are displayed in Fig. 1. In the spectral region from 750 to 950 cm^{-1} a complex set of overlapping bands is observed. Four bands are observed at 813, 843, 853 and 885 cm^{-1}. The assignment of these bands is as follows: (a) The band at 853 cm^{-1} is assigned to the AsO_4^{3-} ν_1 symmetric stretching mode (b) the band at 885 cm^{-1} is assigned to the AsO_3OH^{2-} ν_1 symmetric stretching mode (c) the band at 843 cm^{-1} is assigned to the AsO_4^{3-} ν_3 antisymmetric stretching mode (d) the band at 813 cm^{-1} is ascribed to the AsO_3OH^{2-} ν_3 antisymmetric stretching mode.

The infrared spectrum of geminate from (a) France and (b) Jáchymov, The Czech Republic in the 600 to 1000 cm^{-1} region is shown in Fig. 2. The infrared spectrum compliments the Raman spectrum. The infrared spectrum exhibits a complex set of overlapping bands. Remarkable similarity exists for the two spectra from geminate samples

from two different origins. Infrared bands are found at 740, 760, 804, 830, 850 and 877 cm^{-1}. The two infrared bands at 850 and 877cm^{-1} seem to correspond with the Raman bands at 853 and 885 cm^{-1} assigned to the AsO_4^{3-} ν_1 symmetric stretching mode and the AsO_3OH^{2-} ν_1 symmetric stretching mode respectively.

In the Raman spectrum, a band at 743 cm^{-1} is observed and is assigned to an OH deformation mode. Distinct infrared bands are also observed at 739 cm^{-1} (France) and 740 cm^{-1} (Jácymov). These bands are also attributed to the OH deformation mode. The Raman spectra of geminite in the 100 to 600 cm^{-1} region are displayed in Fig. 3. Raman bands are observed at 333, 345, 421, 451, 481 and 479 cm^{-1}. The two bands at 333 and 345 cm^{-1} are attributed to the ν_2 AsO_4^{3-} bending mode. The set of higher wavenumber bands are assigned to the ν_4 AsO_4^{3-} bending mode. Intense Raman bands are observed on the far low wavenumber region at 136, 161, and 182 cm^{-1}. It is considered that these bands are related to hydrogen bonding of the water molecule in the geminate structure. Other low intensity bands are observed at 213, 244, 284 and 310 cm^{-1}. It is suggested that these bands are related to CuO stretching and bending bands.

The Raman spectrum of geminate from 1000 to 3600 cm^{-1} is displayed in Fig. 4. In the higher wavenumber region of the Czech sample, Raman bands are observed at 2288, 2438, 2814, 3152, 3314, 3448 and 3521 cm^{-1}. For the geminite sample from France, Raman bands are observed at 2289, 2433, 2737, 2855, 3525, 3305, 3377, 3449 and 3521 cm^{-1}. There is good correspondence between the Raman spectra of the two geminite samples.

What this spectrum does is describe water in different molecular environments. The two Raman bands at around 2288 and 2438 cm^{-1} are ascribed to very strongly hydrogen bonded water. The broader Raman bands at 3152 and 3314 cm^{-1} may be assigned to adsorbed water and not so strongly hydrogen bonded water in the molecular structure of geminate. The two bands at 3448 and 3521 cm^{-1} are assigned to the OH stretching vibrations of the $(AsO_3OH)^{2-}$ units. The infrared spectra of geminate in the 1000 to 4000 cm^{-1} are displayed in Fig. 5. Infrared bands are found in similar positions to those shown in the Raman spectrum. Importantly two infrared bands are observed at 1631 and 1670 cm^{-1} (France) and 1632 and 1689 cm^{-1} (Czech). These two bands are assigned to water HOH bending modes. The fact that two bands are observed provides good evidence for two different types of water molecules in the geminate structure. One water molecule is water

involved in very strongly hydrogen bonding and is reflected in the bending mode at ~1689 cm^{-1}; the second is more weakly hydrogen bonding and is reflected in the water bending mode at 1632 cm^{-1}. These bands are associated with the OH stretching vibrations. The water OH stretching bands at 2337, 2403 and 2469 cm^{-1} (France) and 2268, 2396 and 2452 cm^{-1} (Czech) are attributed to very strongly hydrogen bonded water. The broader infrared bands at 2820, 3037, 3190 and 3311 cm^{-1} (France) and 2763, 2966, 3176, 3305, 3330 and 3454 cm^{-1} (Czech) are assigned to the more weakly hydrogen bonded water molecules. The two infrared bands at 3452 and 3520 cm^{-1} (France) and 3520 and 3565 cm^{-1} (Czech) are assigned to the OH stretching bands of the OH units of $(AsO_3OH)^{2-}$ units.

CONCLUSIONS

The Raman and infrared spectra of geminite from Jáchymov, Czech Republic and cap, Garonne Mine, France were obtained and related to the structure of the mineral. Raman bands attributable to the stretching and bending vibrations of the $(AsO_4)^{3-}$ and $(AsO_3OH)^{2-}$ units are obtained. Well defined OH stretching bands are found as well as bands attributable to adsorbed water. The Raman spectrum shows a complex set of overlapping bands in the OH stretching region. It is proposed that the OH units in the geminite structure are non-equivalent. A complex set of overlapping bands are observed in the OH stretching region. Raman bands are assigned to very strongly hydrogen bonded water molecules and to less strongly hydrogen bonded water. Raman bands are attributed to the OH stretching vibrations of the (AsO_3OH) units. Corresponding bands are observed in the infrared spectra.

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Fig.1 Raman spectra of geminite from the (a) Czech Republic and (b) France in the 650 to 1000 cm^{-1} region.

Fig. 2 Infrared spectrum of geminite from (a) Czech Republic and (b) France in the 600 to 1000 cm^{-1} region.

Fig. 3 Raman spectra of geminite from the (a) Czech Republic and (b) France in the 100 to 600 cm^{-1} region.

Fig.4 Raman spectra of geminite from the (a) Czech Republic and (b) France in the 1000 to 3600 cm^{-1} region.

Fig. 5 Infrared spectrum of geminite from (a) Czech Republic and (b) France in the 1000 to 4000 cm^{-1} region.

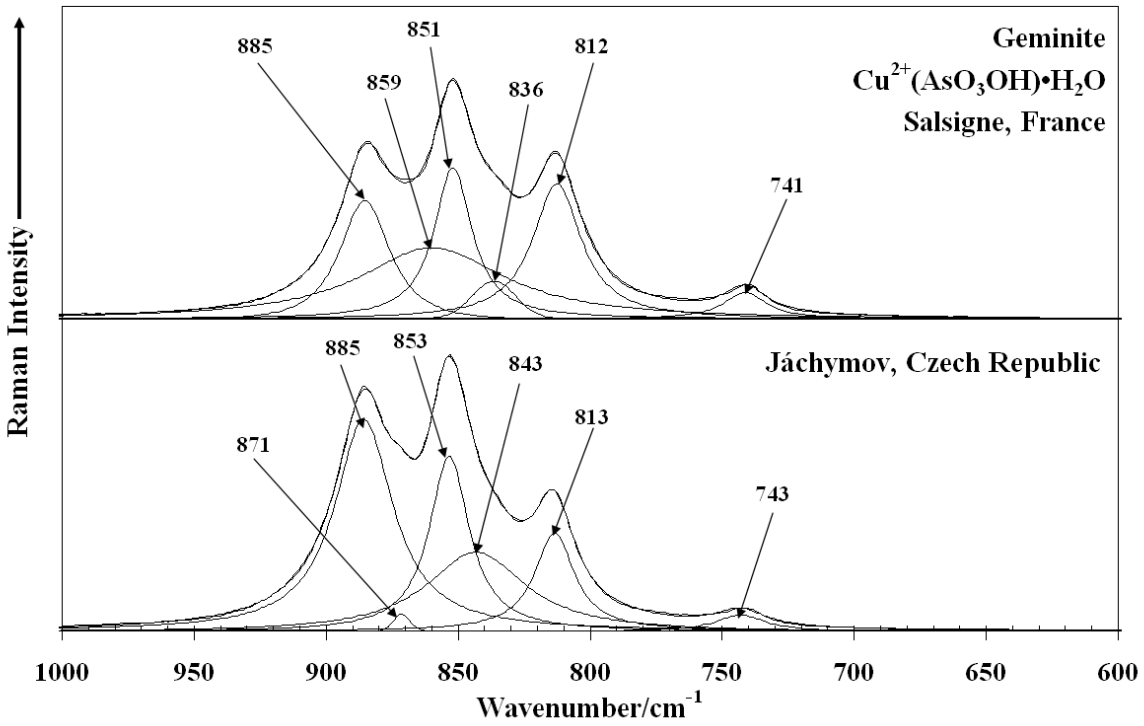
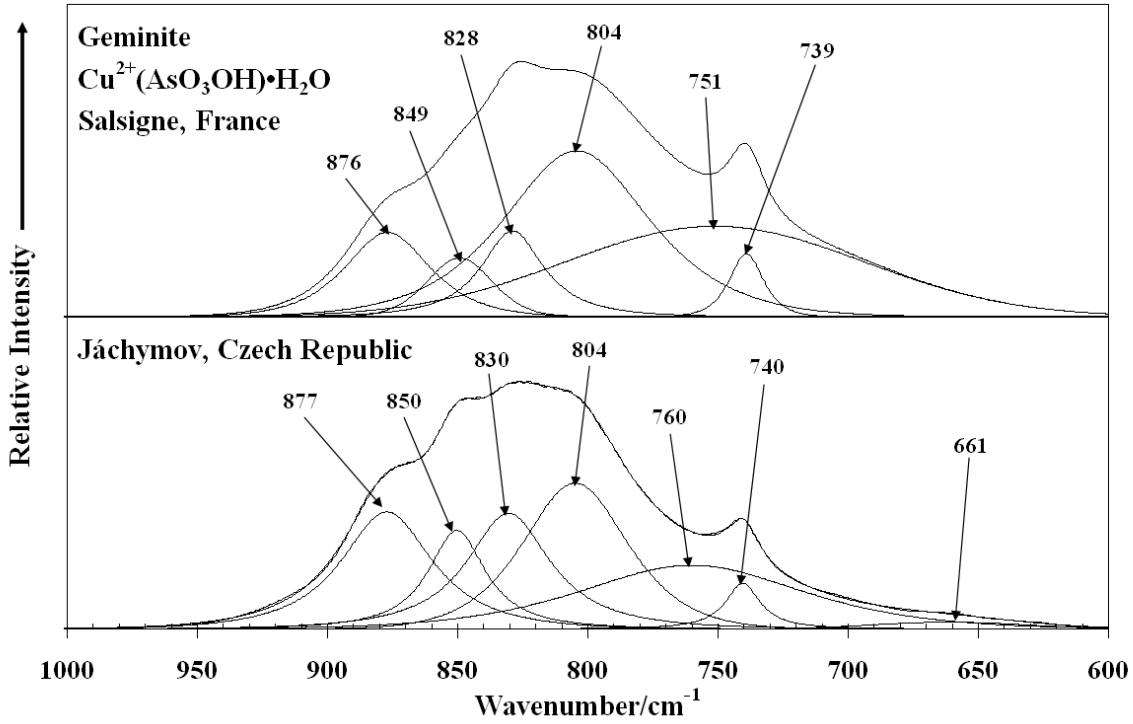

Geminite
Cu2+(AsO3OH)•H2O
Salsigne, France
885
851
859
836
812
741
Jáchymov, Czech Republic
885
853
843
813
871
743
Raman Intensity
1000
950
900
850
800
750
700
650
600
Wavenumber/cm-1

Figure 1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Cu2+(AsO3OH)•H2O
Salsigne, France
828
804
739
751
849
876
Jáchymov, Czech Republic
830
804
740
877
850
760
661
Relative Intensity
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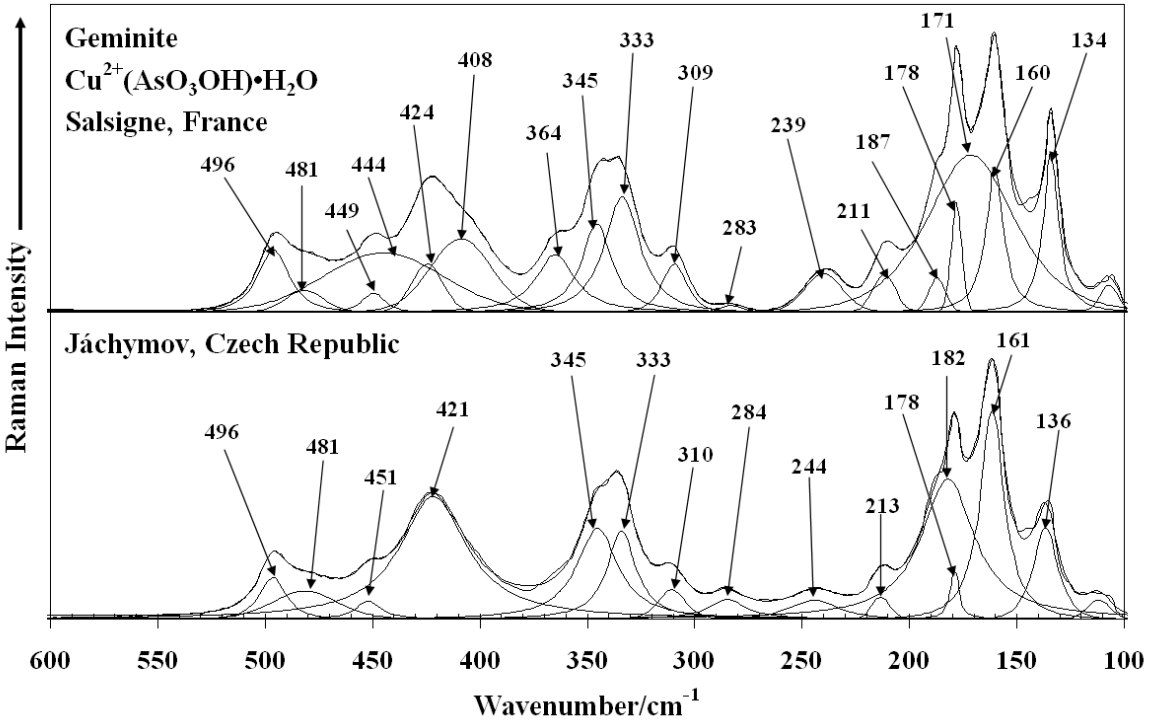
950
900
850
800
750
700
650
600
Wavenumber/cm-1

Figure 2


Geminite
Cu2+(AsO3OH)•H2O
Salsigne, France
Jáchymov, Czech Republic
Raman Intensity
Wavenumber/cm-1
600
550
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450
400
350
300
250
200
150
100
496
481
444
449
424
408
364
345
333
309
283
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211
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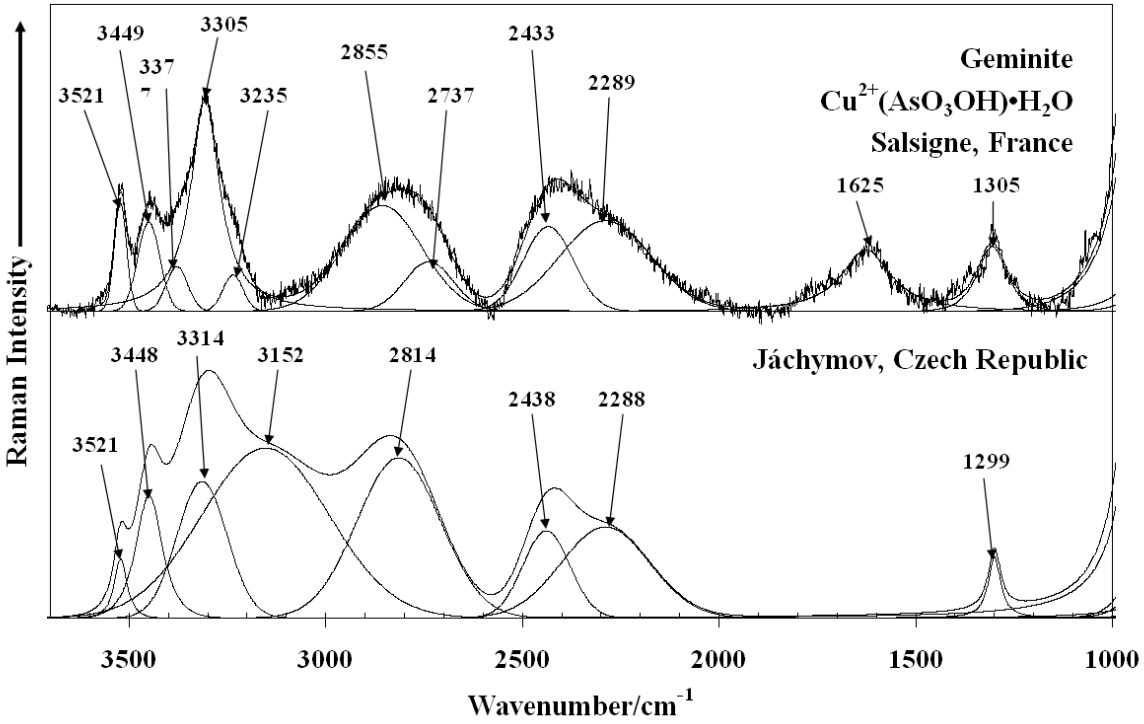
Figure 3


Raman Intensity
3521
3449
3377
3305
3235
2855
2737
2433
2289
1625
1305
Geminite
Cu2+(AsO3OH)•H2O
Salsigne, France
3521
3448
3314
3152
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Jáchymov, Czech Republic
3500
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2000
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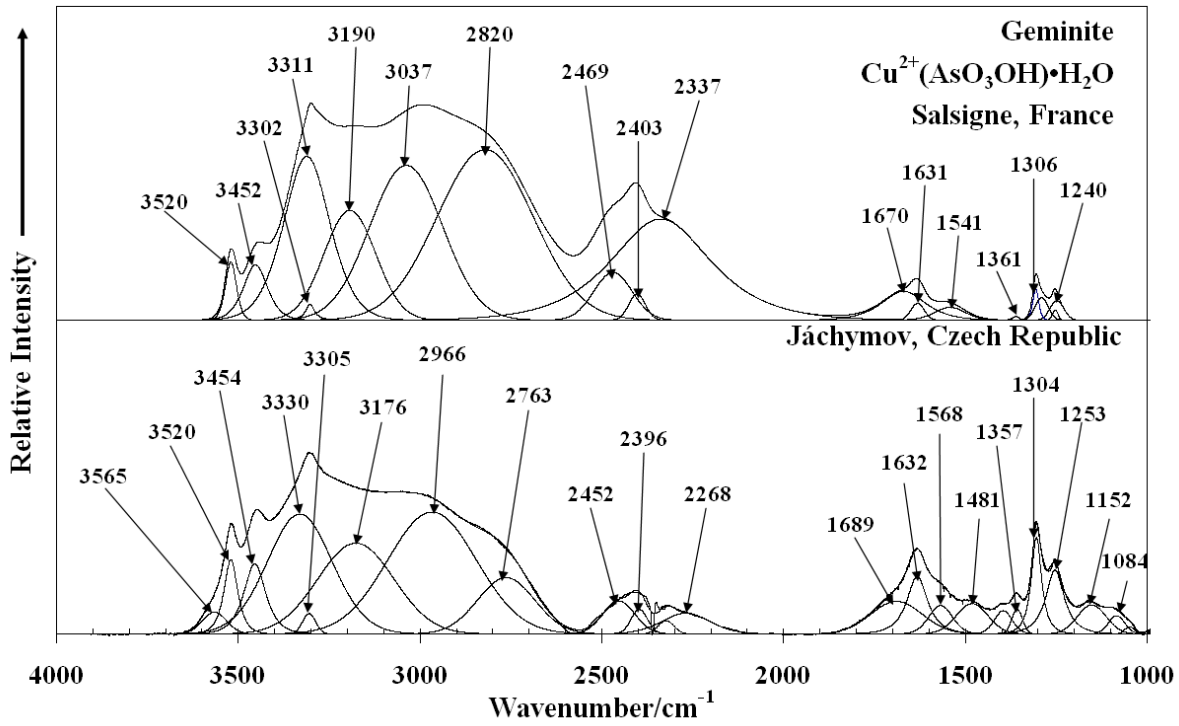
1000
Wavenumber/cm-1

Figure 4


Geminite
Cu2+(AsO3OH)•H2O
Salsigne, France
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Jáchymov, Czech Republic
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3305
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2268
1689
1632
1568
1481
1357
1304
1253
1152
1084
Relative Intensity
4000
3500
3000
2500
2000
1500
1000
Wavenumber/cm-1

Figure 5