Channel constituents of alkali-poor Orissan beryls: An FT-IR spectroscopic study

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The Fourier transform infrared (FT-IR) spectra of four coloured beryls, both in single crystal and powder form, were studied at room temperature to characterize the volatile constituents of beryls from Badmal mines of Orissa state. Among the four coloured beryls (colourless, blue, green and yellow), the green beryl shows predominance of type-II H_2O. Presence of sodium and cesium molecules associated with OH⁻ group and CO_2 as common channel constituent is also deciphered. Fourier self-deconvolution (FSD) technique is applied to observe weak shoulders and classify beryls based on substitution. While the c/a ratio of 0.9994–0.9996 indicates beryls to be of 'tetrahedrally substituted', the FT-IR study in the structural vibration indicates them to be of 'normal type'.

BERYL is a typical cyclosilicate mineral, whose structure was first described by Bragg and West[1]. Its honey comb structure consists of six-membered silica tetrahedral rings linked together with octahedrally co-ordinated Al^{3+} ions and tetrahedrally co-ordinated Be^{2+} ions. Oppositively facing six-fold rings of silica tetrahedra are stacked along the c crystallographic axis of beryl crystal resulting in widely spaced interconnecting channels (Figure 1 a and b). Dimension of channels varies from 2.8 Å in the plane of Si_6O_{18} rings and 5.1 Å between neighbouring rings.

Although beryl is an anhydrous mineral, it is known that a considerable amount of water (up to 2.9% by wt)[2] occupies channel sites. Apart from water molecules, the other contents of the channels are CO_2, alkali cations, e.g. Li, Na, K, Ca, Rb, Cs (refs 3–7), transition elements[8,9] such as Fe^{2+} and Fe^{3+} and rare gases like He and Ar (ref. 10). While these ions occupy channel site as a result of interstitial solid solution, existence of two important kinds of substitution in beryl gives rise to two distinct substitutional solid solution series[7]. These are 'octahedral beryl' in which dominant substitution is Fe^{2+} and Mg^{2+} for Al^{3+} in octahedral site, and 'tetrahedral beryl' in which the dominant substitution is of Li^+ for Be^{2+} in the distorted tetrahedral site. According to Wood and Nassau[8], presence of alkali ions in the channels is made possible because of the missing negative charges in the lattice due to substitution of Al^{3+} by a bivalent ion like Fe^{2+} or Mg^{2+}. These substitutions modify cell parameters. Based on c/a ratio Aurisichhio et al.[7] classified beryls into three groups; i) octahedral beryl, c/a value from 0.991 to 0.996; ii) tetrahedral beryl, c/a value from 0.999 to 1.003; and iii) normal beryl, c/a value from 0.997 to 0.998. Normal beryl includes those where substitutions occur in both tetrahedral and octahedral sites. Owing to such complex cationic substitutions, the chemical composition of beryl often deviates from the ideal formula $Be_3Al_2Si_6O_{18}$. Wood and Nassau[3] inferred that hydrogen speciation in beryl is H_2O and from the polarization dependence of the absorption inferred that it occupied two distinct sites. They accordingly called it as Type-I and Type-II water. Type-I water has its H–H vector parallel to c crystallographic axis and Type-II has its H–H vector perpendicular to c-axis (Figure 1 a). The linear CO_2 molecule, because of its size (4.9 Å), lies normal to c-axis and parallel to the larger dimension of the channel. With the help of NMR studies on cordierite, which is also characterized by large channels like beryls, Carson et al.[11] demonstrated that individual water molecules can hob between the two directions.

Hydrogen in minerals mostly occurs bonded to oxygen and the resulting OH group is highly polar. This dipole is an efficient absorber of light in the infrared region. Further, if the OH groups are structurally oriented within the host, the amount of incident radiation absorbed is strongly dependent upon the relative orientation of the OH dipole and the direction of linear polarization of light[12]. Thus infrared spectroscopy is a powerful technique to investigate the volatile constituents in minerals.

In the present study, we have carried out Fourier transform infrared spectroscopic (FT-IR) studies to characterize the channel constituents in four samples of coloured beryls from Badmal area in Orissa state. An attempt is also made to observe the influence of octahedrally and tetrahedrally substituted cations on the IR absorption spectrum of beryl crystals by applying Aurisichhio et al.[13] criteria.

Experimental methods

Infrared absorption spectra were obtained at room temperature from doubly polished thin slices of beryls

(colourless, blue, green and yellow), orienting either parallel or normal to c-axis. FT-IR spectra in the frequency range 4000–2000 cm^{-1}, for single crystal and 4000–400 cm^{-1} for powder samples were recorded using NICOLET Magna IR 550 FT-IR spectrometer. Spectral resolution was set at 4 cm^{-1}. Background effects due to atmospheric H_2O and CO_2 were subtracted. In the case of slice cut parallel to c-axis, IR spectra were taken for both polarization using a grid polarizer deposited on a substrate of KRS-5, enclosed in a stainless steel ring. Samples were prepared as compressed disks of powdered beryl and KBr (2 to 5 wt%) to minimize polarization effects. Disks were prepared taking the same amount of mechanically ground powder and compacted under 10^{10} Pa pressure.

Single crystal X-ray diffraction was carried out on Enraf Nonius CAD-4. Graphite monochromate radiation MoKα ($\lambda = 0.7107$ Å) was used as source. Twenty-five reflections were used for least square refinement of cell parameters in the $20 < \vartheta$ range $< 25°$ using CAD4-PC software[14].

Analyses of four coloured beryl samples were carried out by Electron Probe Micro Analysis using SX-50 Microprobe housed at the Department of Geology, Mysore University[15]. Operating conditions were 15 kV accelerating voltage, 20 mA beam current and 10 s counting time.

Results and discussion

The IR absorption spectra of four beryls under study, i.e. colourless (goshenite), blue (aquamarine), green beryl and yellow (heliodor) are shown in Figure 2 a–d. The cell parameters determined from single crystal are; $c = 9.199$ (2) Å, $a = 9.204$ (3) Å, $c/a = 0.9994$, $V = 675.55$ (4) Å^3 and for powder are; $c = 9.199$ Å, $a = 9.208$ Å, $c/a = 0.9996$, and $V = 674.76$ Å^3. The above c/a ratio is indicative of tetrahedrally substituted beryl.

Wood and Nassau[8] assigned 3800–3400 cm^{-1} (OH-stretching mode) frequency range absorption bands to two types of H_2O molecules; Type-I with its 2-fold axis of symmetry parallel to 6- fold axis and type-II with its 2-fold axis perpendicular to 6- fold axis. Our polarization studies on Orissan beryls confirm the existence of these two types of water molecules.

IR spectra in the region of 4000–2000 cm^{-1} were collected from 1.0 mm size doubly polished crystals of beryls, cut normal to c-axis. In $goshenite$ (Figure 2 a), the fundamental symmetric stretching motion (v_1) of type-I H_2O is observed at 3555 cm^{-1} and asymmetrical stretching motion (v_3) is observed at 3699 cm^{-1}. The type-II water molecule shows v_1-mode at 3606 cm^{-1} and v_3-mode at 3662 cm^{-1}. $Heliodor$ (Figure 2 b) shows v_1-mode of type-I at 3560 cm^{-1} and v_3-mode at 3698 cm^{-1}. Type-II H_2O, v_1-mode is observed at 3607 cm^{-1} and
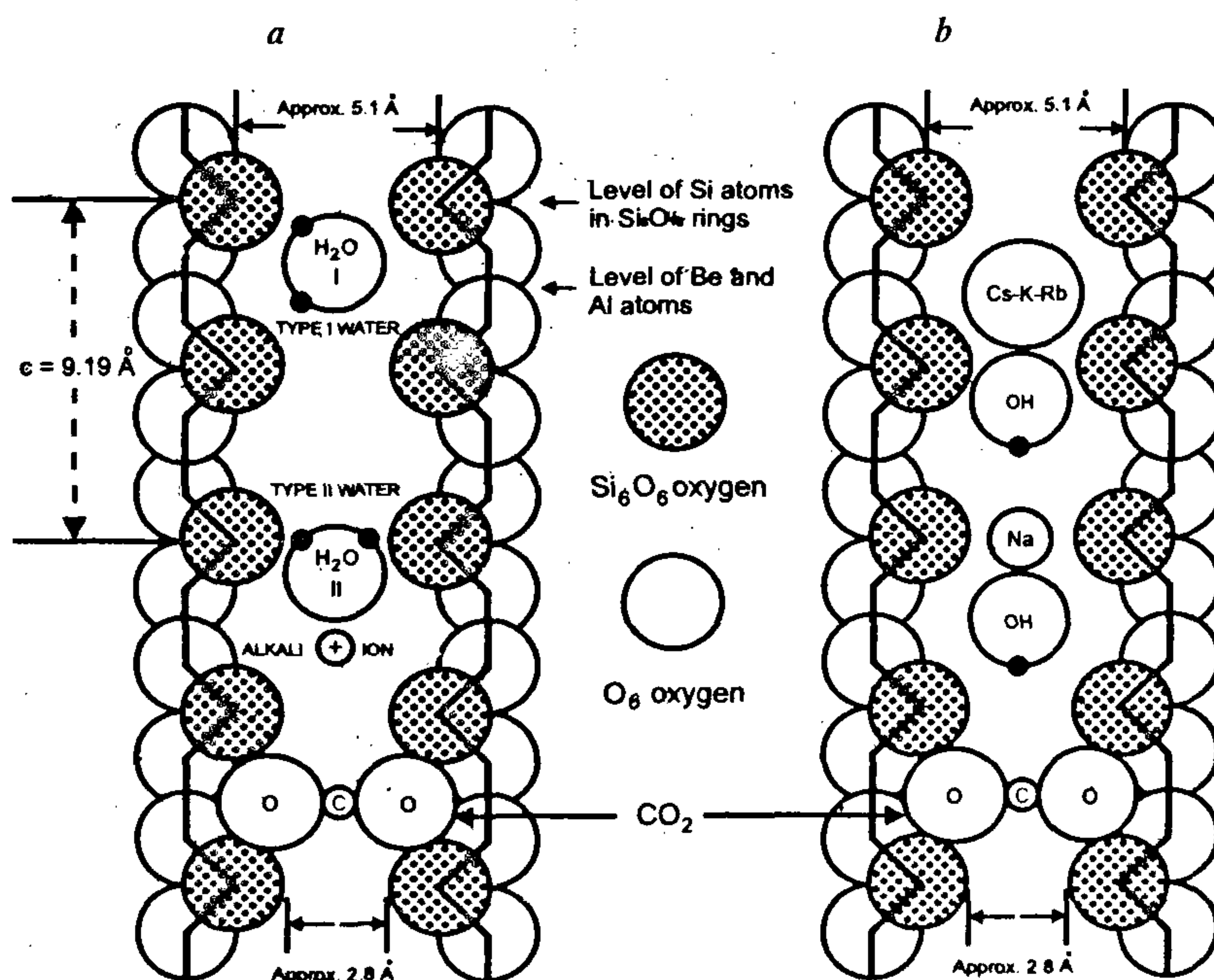

Figure 1. Schematic drawing parallel to c-axis showing the channel configuration of beryl (modified after Aurisichhio et $al.$[13]). a, With type-I H_2O, type-II H_2O and CO_2 molecules; b, With OH$^-$ group and associated with larger alkali ions.

v_3-mode at 3661 cm^{-1}. Similarly in *aquamarine* (Figure 2 *c*), the v_1-mode of type-I water molecules is observed at 3552 cm^{-1} while the v_3-mode is discerned at 3698 cm^{-1}. For type-II water, v_1-mode is seen at 3606 cm^{-1} and v_3-mode at 3660 cm^{-1}. *Green beryl* (Figure 2 *d*), shows type-I, v_1-mode at 3555 cm^{-1} and v_3-mode at 3699 cm^{-1}. Type-II water molecule, v_1-mode is observed at 3606 cm^{-1} and v_3-mode at 3660 cm^{-1}. The IR absorbance observed above 3850 cm^{-1} bands commonly arises from linear combination and multiple overtones. According to Aines and Rossman[16], absence of these higher vibration modes indicates the molecular species to be OH. This is particularly important if the bending mode vibrations expected around 1600 cm^{-1} is not seen, due to the interference from silicate absorption, when thick samples are used, as in the present study described above.

The bending mode vibration of H_2O is commonly observed in the region of $1400-1600 \text{ cm}^{-1}$ (refs 3, 16). However, in our study on single crystal, no such lines were observed, probably due to lack of transmittance in the region above 2000 cm^{-1}, as a result of thickness of sample as described above. However, these lines were able to be observed when the samples were further thinned to $100 \text{ }\mu\text{m}$ (Figure 2 *c*). As observed in Figure 2 *c*, the type-II vibration frequency is slightly greater, since the adjacent alkali ions rotate the water molecules in the channel. This tighter bonding with charged alkali ions results in higher rotational combination frequency of the type-II spectrum, which in turn causes higher deformation frequency and symmetric bending[17]. All the three vibration modes of Orissan beryls described above are summarized in Table 1.

In all the four FT-IR spectra, it is observed that the type-II H_2O is dominant. A similar observation has also been made by Panjikar[18] on beryls from Orissa. This indicates that in these beryls there is increased substitutions for octahedral Al and tetrahedral Be sites, coupled with incorporation of alkalis into channels[13]. Therefore, type-II H_2O is directly related to the incorporation of alkalis into channel[13,19]. This substitution results in type-II H_2O becoming dominant due to the dipole effect of H_2O. Oxygens of these water molecules are attracted by alkali ions and turn their 2-fold axis parallel to the 6-fold axis of beryl crystal.

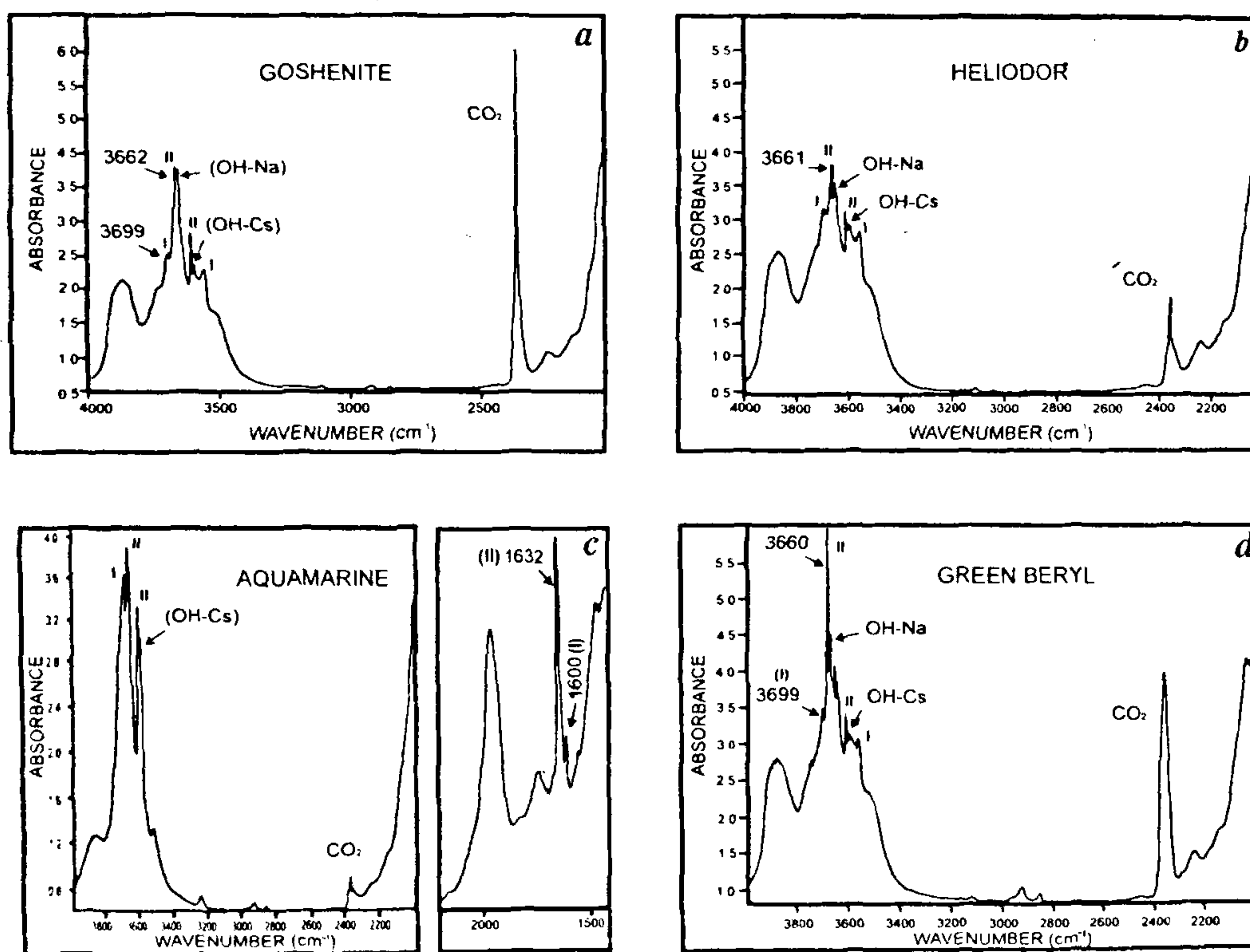A significant observation seen in the above four spectra of beryls is the presence of variable amount of type-II

Figure 2. Unpolarized (single crystal) FT-IR spectrum beryl crystals showing presence of type-I, type-II H_2O and CO_2 molecular species parallel to c-axis. *a*, colourless beryl (goshenite); *b*, yellow beryl (heliodor); *c*, blue beryl (aquamarine) showing both stretching and bending motion of H_2O; *d*, green beryl.

water in different coloured beryls, although collected from the same locality (Badmal pegmatite mines). As explained earlier, the type-II water molecule is dominant in all the four beryls, however, among the four coloured beryls, the IR spectrum shows that type-II water molecule is predominant in green beryl (Figure 2 d) compared to other coloured beryls. Whereas the IR spectrum in Figure 2 c, of blue beryl shows that type-I water molecule is predominant in blue beryl in comparison with other coloured beryls. This result indicates that among the four coloured beryls, green beryl is richer in alkalis. A similar type of observation was also made by Blak et al.[20]. As shown in Table 2, the alkali content is the same in almost all crystals. This suggests that a major amount of alkalis in blue and alkalis in minor amount in colourless and yellow beryl are situated in interstitial sites between Be^{2+} and Al^{3+} or in substitutional sites, while in green beryls the alkalis are in the structural channel. The effects of alkalis on colouration in beryls have been completely ruled out by many authors[8,9,21].

In addition to the common type-I and type-II water molecules, a small amount of water molecules is also observed around 3120, 2820 and 2925 cm^{-1} in both polarization directions, without any bending vibration counterpart. The above inference is in contrast to the observation of Charoy et al.[22] of above lines being oriented only parallel to the c-axis. Such frequency is assigned to H_2O with a compact structure of ice[23].

All the samples studied show strong and sharp dichroic lines around 2360 cm^{-1}. This peak corresponds to the asymmetric stretching mode of CO_2 vibration, since except v_1-mode, all other vibration modes (v_3 and v_2) are IR active, whereas v_1 is Raman active[22]. Identical bands of CO_2 were also observed by Wood and Nassau[8], from natural beryl, but are absent in synthetic ones. In all the IR spectra (Figure 2 a–d) the base of the peak is slightly enlarged and asymmetrical. Two side bands at 2450 cm^{-1} and 2250 cm^{-1} observed on either side of the CO_2 peak are attributed to the combination bands of both stretching and coupled hindered motion of CO_2 (ref. 16). The polarization studies confirm that, CO_2

molecules are isotropically distributed in the (0001) plane, in accordance with the observations of earlier workers[8,15,22].

Aurisichhio et al.[13] observed that the type-II band at 3663 cm^{-1} is a combination of two bands, when observed at 77 K. One due to type-II H_2O at 3663 cm^{-1} and other at 3658 cm^{-1} due to OH–Na association. However, in our present FT-IR study at room temperature, the 3658 cm^{-1} band is seen at 3654 cm^{-1} (Figure 2 a–d) as separate low peak apart from 3663 cm^{-1} type-II H_2O. This indicates that the 3654 cm^{-1} band is not associated with H_2O, but a separate hydrous species, possibly (OH)⁻ group. This is in accordance with the expectations of Aurisichhio et al.[13]. Several authors[3,24–26] had also suggested the possible presence of OH⁻ group in the beryl lattice. However, its definite assignment in the beryl structure was debatable. We favour its presence in the channel site, in accordance with Aurisichhio et al.[13], since (OH)⁻ group can also be similarly attracted to the alkali cation as the dipolar H_2O (Figure 1 b).

Aurisichhio et al.[13] attributed that, if both Na and Cs are present, the abosorption band is close to 3595–3597 cm^{-1}. Thus the 3600 cm^{-1} band becomes a combination of three absorption frequencies; type-II H_2O, OH–Na, and OH–Cs. In the present study, although the Cs content (Table 1) is not analysed, in the IR spectra a low weak absorbance at 3595 cm^{-1} (Figure 2 a–d) is seen close to the 3606 cm^{-1} (v_1, type-II band). This peak, we presume, is of OH–Cs as envisaged by Aurisichhio et al.[13].

Structural vibration (1200–400 cm^{-1})

As shown in Figure 3, the powder FT-IR spectrum of yellow beryl indicates dominant type-I (v_3-mode) water molecule as compared to type-II (v_3-mode) when observed in single crystal spectrum (Figure 2 b). The above behaviour of water molecule is also observed in other coloured beryls. Increase in v_3, type-I H_2O intensity in powder spectra indicates increase in proportion of type-I water molecule at the expense of v_3, type-II water

Table 1. Vibrational bands of molecular species from single crystal and KBr pellets experiments

Molecular species	Vibrational mode	Goshenite	Aquamarine	Heliodor	Green beryl	KBr pellets	Wood and Nasssau[3]
H_2O Type-I	v_1	3555	3552	3555	3555	3555	3555
	v_2	1600	1596	1603	1602	1560	1595
	v_3	3699	3690	3698	3699	3698	3694
H_2O Type-II	v_1	3606	3606	3607	3606	3598	3592
	v_2	1632	1633	1633	1633	1630	1628
	v_3	3662	3660	3661	3660	3660	3655
OH–Na		3654	–	3653	3654	3654	–
OH–Cs		3595	3594	3595	3595	–	–
CO_2		2360	2360	2360	2360	2362	2360

molecule (Figure 3). The above observation elucidates that, on crushing there is probable release or movement of alkali ions from their present position, thereby causing the type-II H_2O to rotate back to type-I H_2O. This, however, is a tentative explanation and detailed work is warranted to explain the above phenomenon.

Structural vibrations in the region of 1200–400 cm⁻¹, as shown in Figure 4 a, were studied using powdered samples of beryls. Several authors[27,28] assigned 1200 cm⁻¹ to Si–O–Si ring vibration. Later, Aurisichhio et al.[13] confirmed this by correlating with c/a ratio, bond lengths and site occupancies. The IR spectra of all the four Orissan beryls (Figure 4 a) show the 1201 cm⁻¹ vibration. Aurisichhio et al.[13] attributed such vibrational bands to 'normal type' beryl. Increased substitution for Al and Be, coupled with the incorporation of alkalis may result in shifting and broadening of this band due to slight changes in the silicon tetrahedron geometry[13].

As shown in Figure 4 a, a low absorbance peak is seen at 1026 cm⁻¹ and also small shoulder at 1070 cm⁻¹. These peaks indicate that the Orissan beryls are octahedrally (1026 cm⁻¹) and tetrahedrally (1070 cm⁻¹) substituted[13]. The intensity of 1070 cm⁻¹ band appears when the extent of substitution in tetrahedral site attains 0.19 atoms per formula[13]. Plysunina[20] considered the above bands to be associated with variation in the length of the external bonds, Si–O–Al and Si–O–Be respectively. Thus the above observation indicates Orissan beryls to be of 'normal type'.

The absorption band at 960 cm⁻¹ is due to another type of Si–O–Si ring vibration[28,29]. The absorption band at 808 cm⁻¹ is attributed to Be–O(2) vibration[13,28,29]. The 745 and 683 bands are also due to Be–O (refs 27, 28).

According to Aurisichhio et al.[13], presence of shoulders at 700 and 560 cm⁻¹ bands is evidence of tetrahedrally
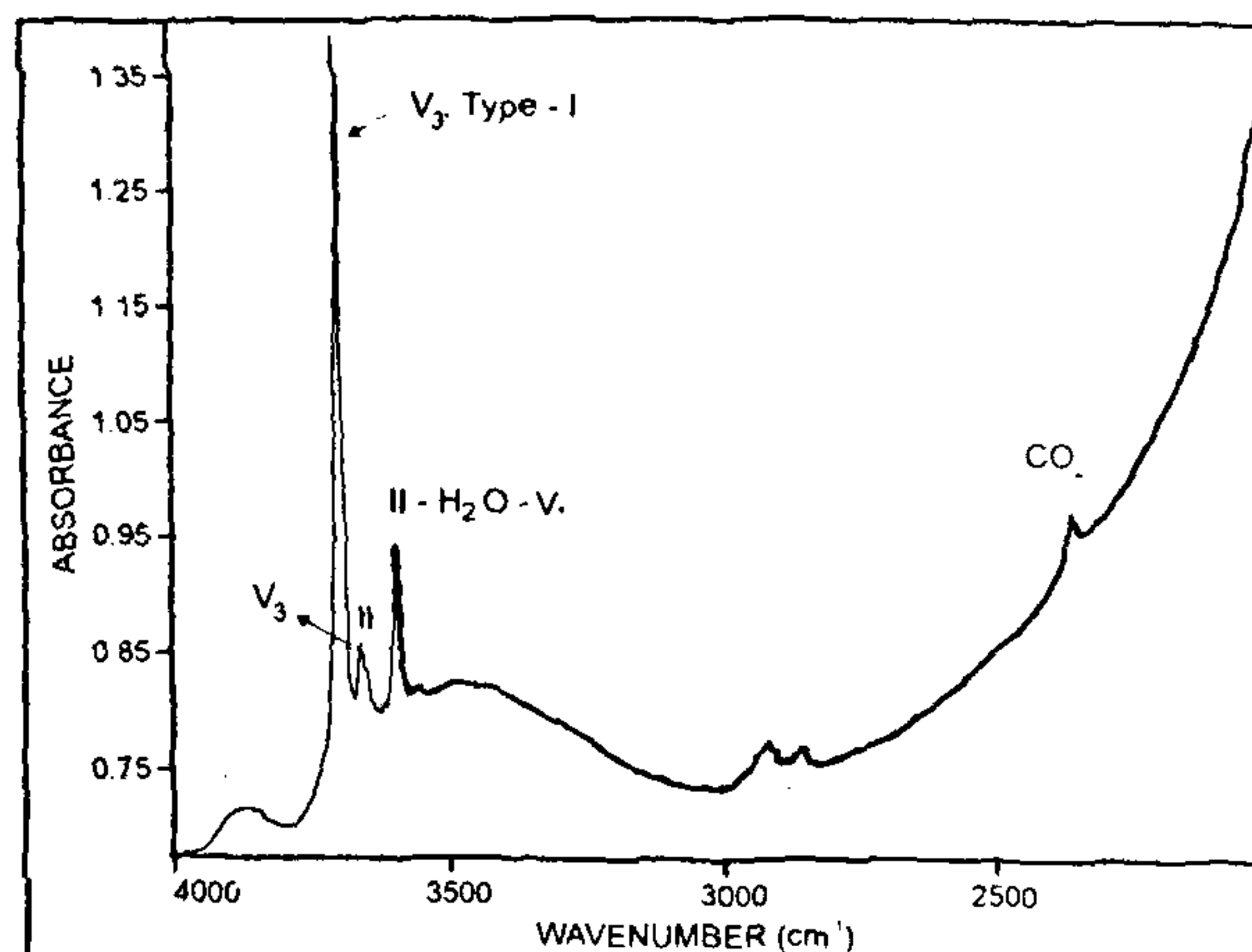

Figure 3. Powder FT-IR spectrum of yellow beryl showing the presence of molecular species.
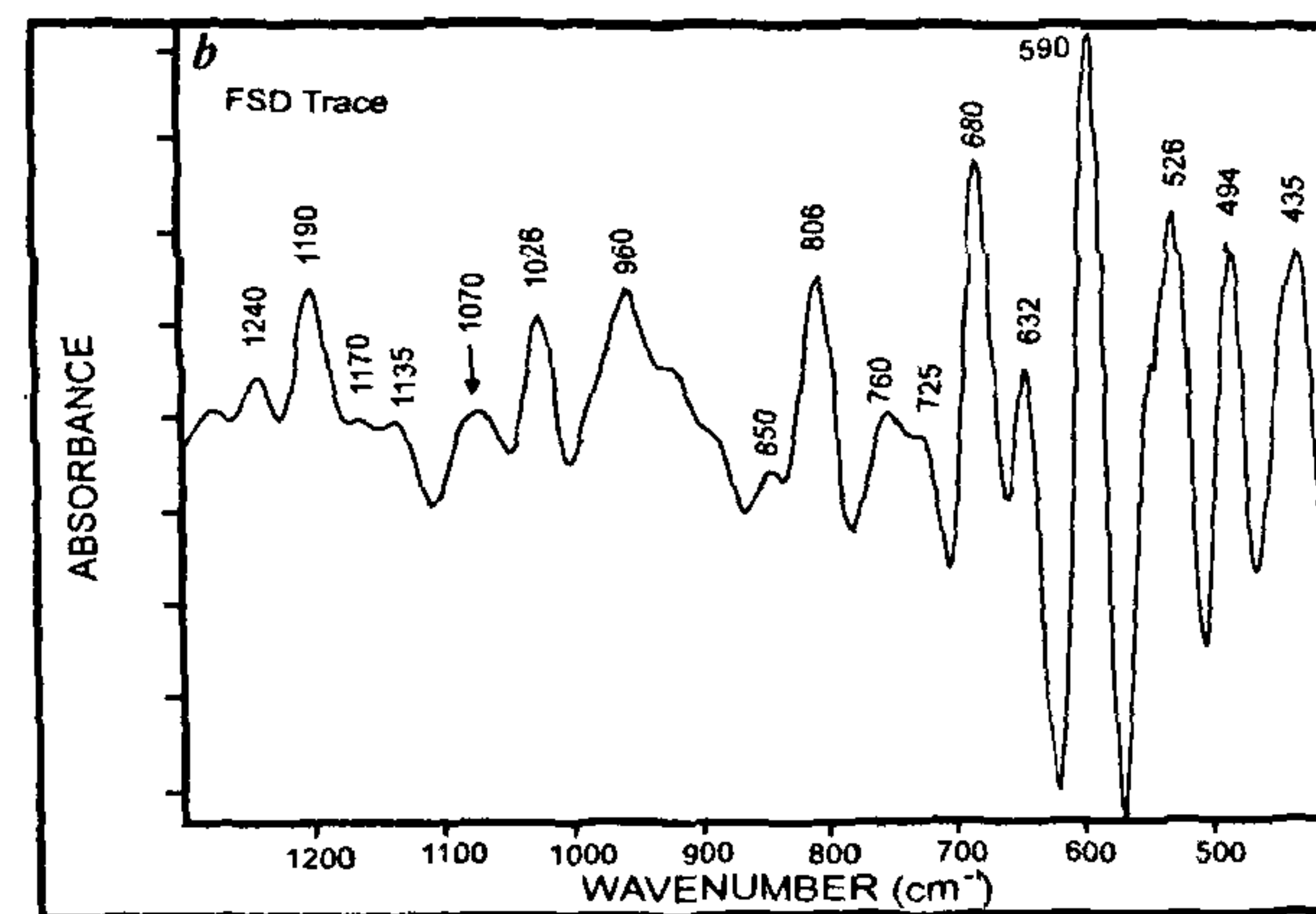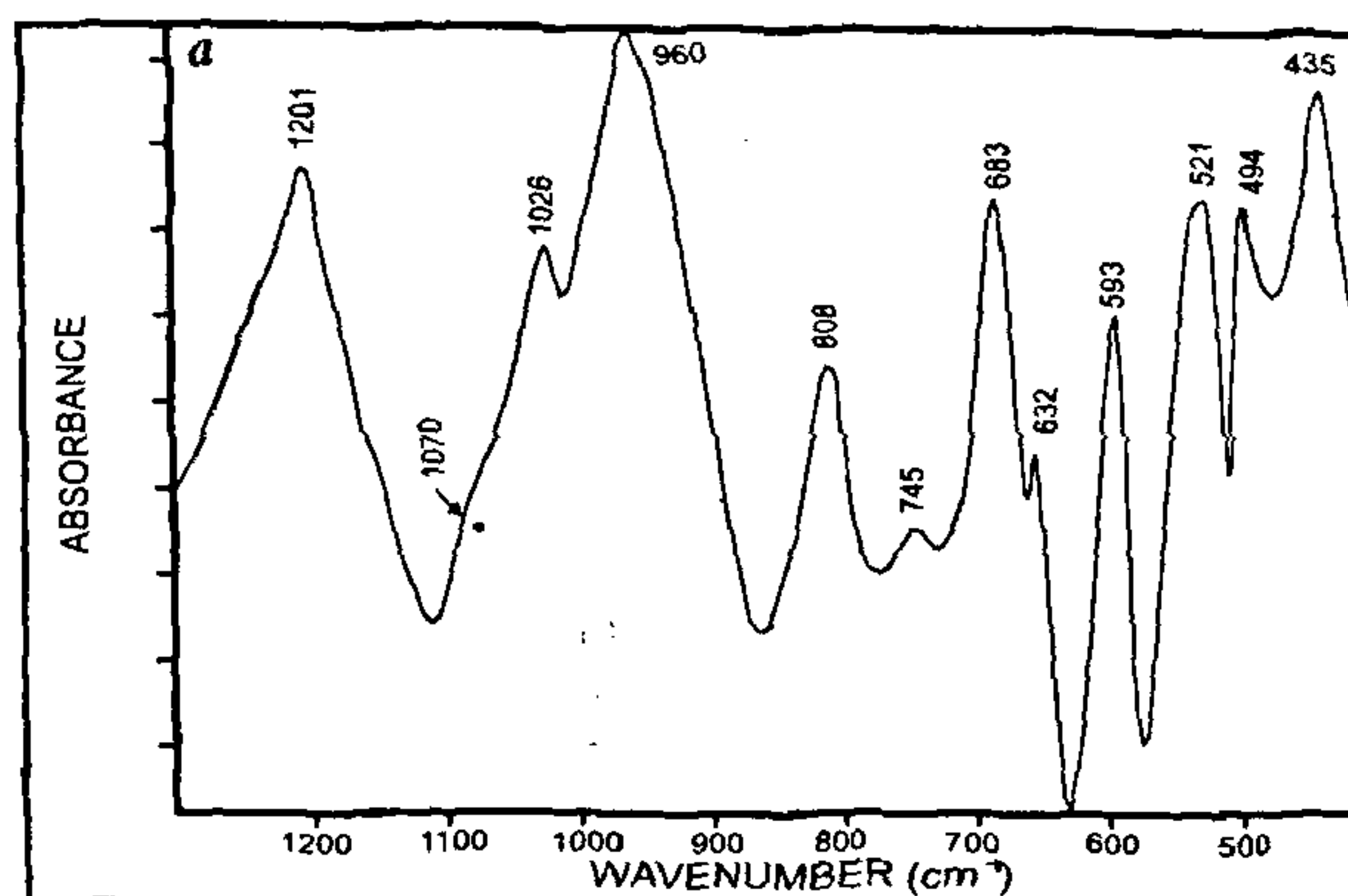

Figure 4. a, Normal powder FT-IR spectrum of beryl in the region of structural vibration (1200–400 cm⁻¹). b, Fourier self-deconvolution (FSD) spectrum of beryl in the region of structural vibration (1200–400 cm⁻¹).

Table 2. Electron probe micro analyses of four coloured beryls

Wt%	Green beryl	Yellow beryl	Blue beryl	Colourless beryl
SiO_2	66.943	67.088	63.287	65.698
Al_2O_3	17.607	18.146	17.743	17.740
FeO'	0.680	0.543	0.519	0.546
MnO	0.006	0.006	–	0.025
MgO	0.026	0.001	0.009	0.093
CaO	0.015	0.013	0.032	0.020
K_2O	0.018	0.010	0.002	0.019
Na_2O	0.159	0.229	0.234	0.206
F	0.250	0.166	0.042	0.308
Cr_2O_3	0.039	–	0.040	0.027
TiO_2	–	–	–	0.068
V_2O_3	0.010	–	0.050	0.034
NiO	0.005	0.132	0.040	0.020
CoO	0.025	–	–	0.056
BeO	n.d	n.d	n.d	n.d
Li, Cs, Rb	n.d	n.d	n.d	n.d
H_2O	n.d	n.d	n.d	n.d
Total	85.783	86.740	81.978	84.860

n.d – not determined.

substituted beryl. As shown in Figure 4 a, these two bands were not discerned in a normal FT-IR spectrum. However, on carrying out Fourier self-deconvolution (FSD) (Figure 4 b), both the above bands are seen as shoulders in all four beryls (Figures 5 and 6). An increase in absorbance of IR intensity is possible when there is progressive entry of cations into tetrahedral site, but not in octahedral site[13]. However, in the samples under study only a small shoulder is observed which indicates that there is partial substitution of cations in the tetrahedral site. As shown in Figure 5 a, the FSD trace of colourless beryl (goshenite) indicates 700 cm⁻¹ as shoulder around 698 cm⁻¹. Similarly FSD trace of other coloured beryls (Figure 5 b–d) also indicates shoulders close to 700 cm⁻¹. Thus the band near 700 cm⁻¹ in all four beryl crystals indicates Orissan beryls to be partially 'tetrahedrally substituted'.

As shown in Figure 4 b, on carrying out FSD, the 1070 cm⁻¹ seen as shoulders in a normal FT-IR spectrum (Figure 4 a) is clearly resolved as small absorbance peak. Similarly FSD spectrum indicates that the 1021, 960 and 745 cm⁻¹ bands are not single bands, but denote a combination of bands (Figure 4 b).

Two absorption bands at 521 and 494 cm⁻¹ (Figure 4 a) are assigned to Al-O(2) vibration. The slight shift of the present bands from Aurisicchio et al.[13] assignment (525 and 500 cm⁻¹) indicates the degree of distortion of the polyhedron. This occurs when larger bivalent ions like Fe^{2+} or Mg^{2+} substitute for Al ion[30]. This substitution results in increase of volume of the polyhedron to accommodate the ions with larger ionic radii. Thus it leads to elongation of Me–O distances, resulting in distortion of the polyhedron. On further detailed FSD analysis between 620 and 520 cm⁻¹, the 521 cm⁻¹ band observed in normal FT-IR spectrum (Figure 4 a) is seen resolved into 555 and 524 cm⁻¹ bands (Figure 6 a). The 555 cm⁻¹ band is closer to 560 cm⁻¹ band observed by Aurisichhio et al.[13] which they have assigned to substitutional tetrahedral site. In the blue beryl FSD spectrum (Figure 6 b), it is observed that 521 cm⁻¹ band (Figure 4 a) is a combination of two absorption bands at 556 and 521 cm⁻¹. As shown in Figure 6 c, the FSD spectrum of green beryl indicates to be a composite of 558, 536 and 518 cm⁻¹ bands. Similarly in the heliodor FSD spectrum (Figure 6 d), it is observed to be a combination of three bands at 557, 536 and 520 cm⁻¹.

The 521 cm⁻¹ and 494 cm⁻¹ IR bands observed in normal and FSD spectra in the four samples, indicate Orissan
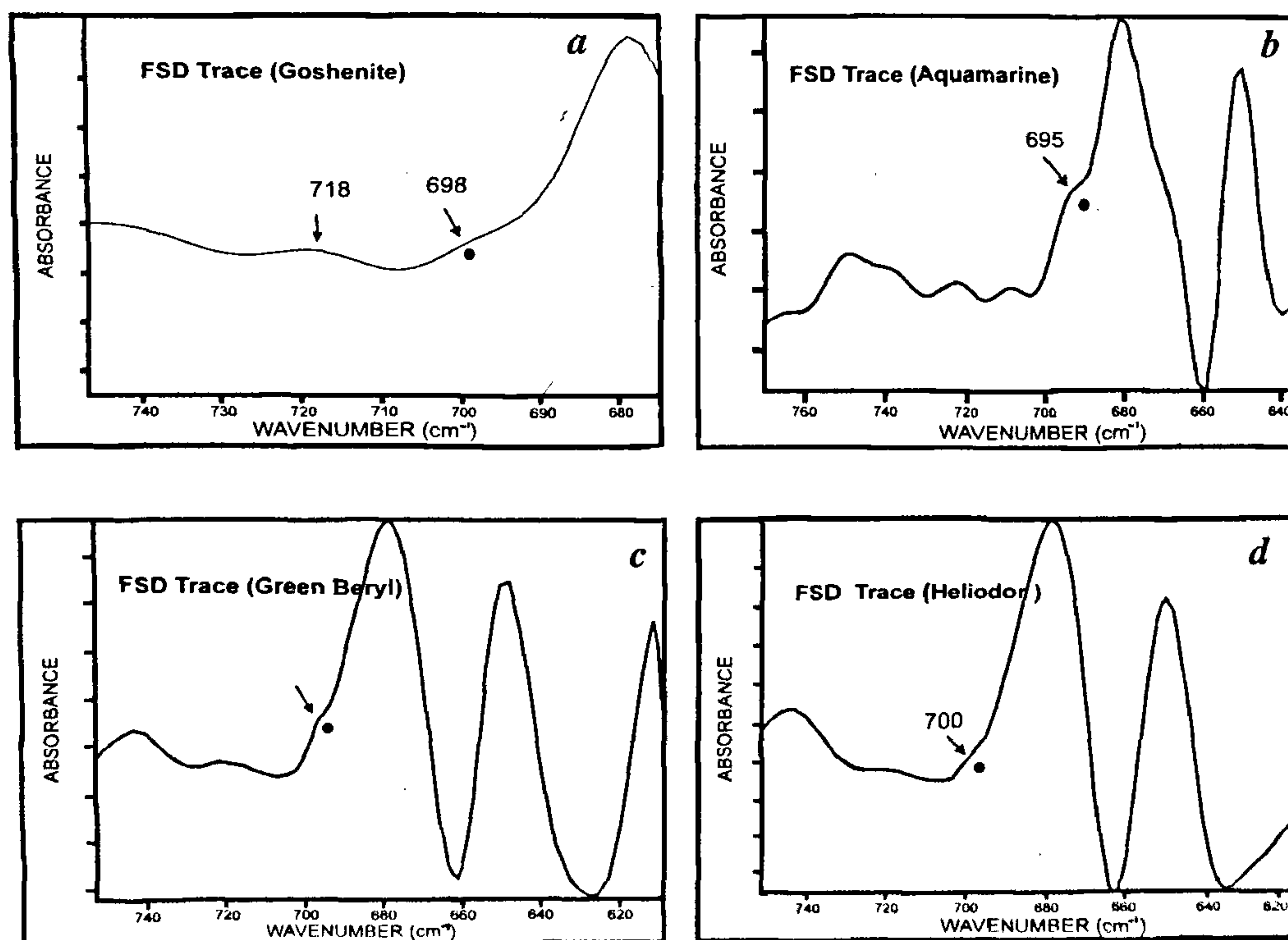

Figure 5. Powder FSD spectrum in the range (760–620 cm⁻¹) indicating the presence of shoulders close to 700 cm⁻¹. a, colourless beryl (goshenite); b, blue beryl (aquamarine); c, green beryl and d, yellow beryl (heliodor).
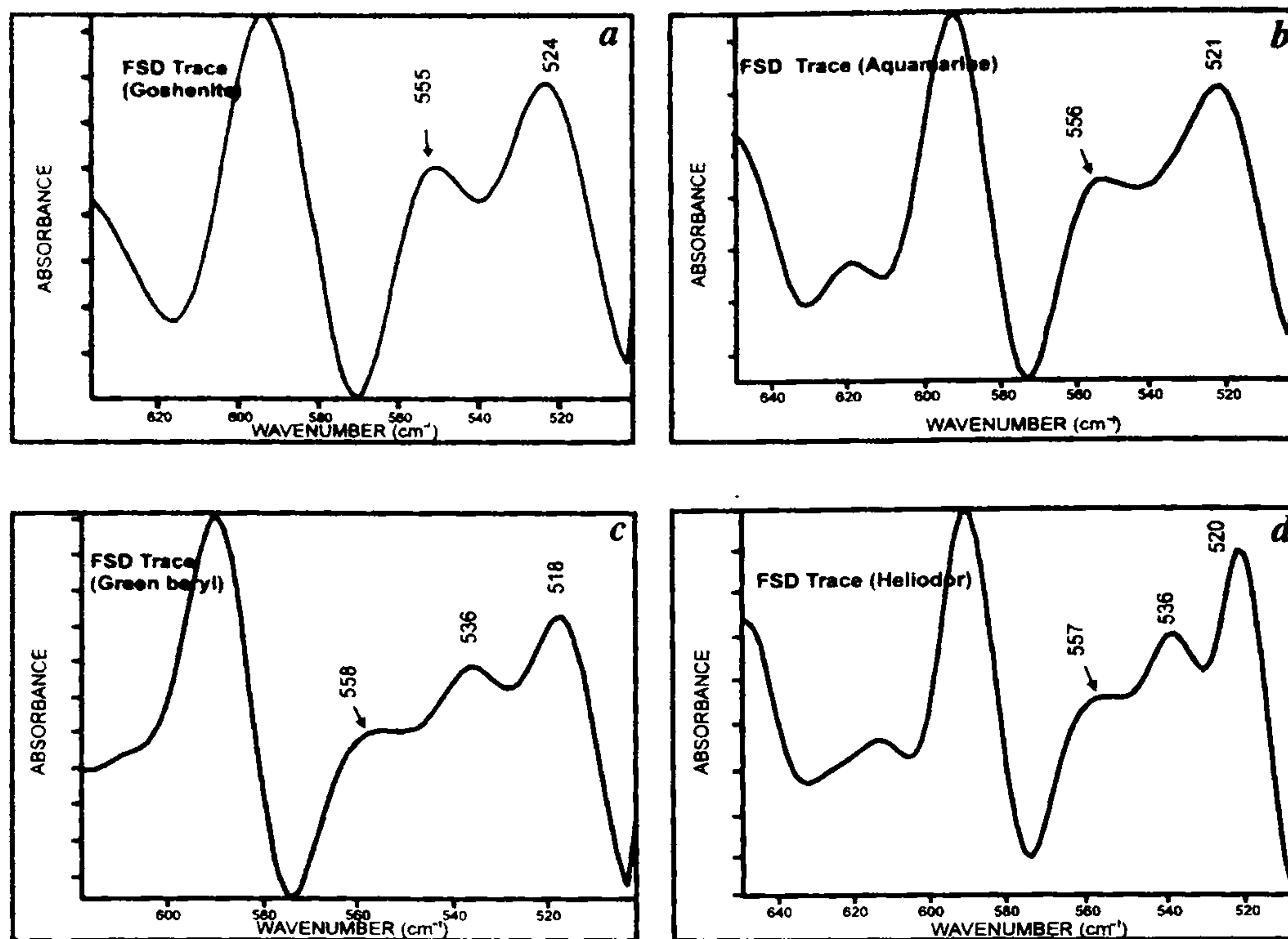
Figure 6. Powder FSD spectrum in the range (620–520 cm⁻¹) indicating presence of shoulder. *a*, colourless beryl (goshenite); *b*, blue beryl (aquamarine); *c*, green beryl and *d*, yellow beryl (heliodor).

beryls to be both of 'octahedrally' and 'tetrahedrally' substituted. Therefore it can be called as 'normal beryl'[7].

Conclusion

Thus the Orissan beryls of Badmal mines are more of 'normal type' containing both octahedral and tetrahedral substitutions. However, the $c/a = 0.9994-0.9996$ ratio indicates these beryls to be tetrahedrally substituted. The $0.9994-0.9996$ c/a ratio is close to 0.998 ascribed[7] for normal type beryl. Therefore, beryls from Badmal mines can be considered to be as normally substituted. From the normal FT-IR spectra, the beryls of Orissa can be concluded to be of 'octahedrally substituted'; however, on deconvolution reveals to be 'normally substituted'. Thus deconvolution studies on normal FT-IR spectra provide further improved resolution of infrared spectra. This again coupled with low temperature studies would provide greater insight on the type of substitutions in beryl.

The presence of H_2O and CO_2 in the structural channels of beryl crystals reflects the volatile constituents of parent melts. Dominant presence of type-II H_2O indicates major proportion of alkalis occupying channel site. This,

however, does not indicate beryls from Orissa to be of high alkaline affinity, as evidenced from chemical analysis. The low alkali content in these beryls indicates that they might have precipitated from acidic fluids.

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MEETINGS/SYMPOSIA/SEMINARS

National Symposium on Plant Diversity Pattern and Bioresource Technology

Date: 6–7 March 1998
Place: Gorakhpur

The following aspects related to the community organization, plant diversity patterns, conservation of dwindling plant resources and recent ecological and biotechnological approaches for conservation and management of bioresources are proposed to be discussed in the symposium: **Diversity patterns**: Survey and measurement of plant diversity patterns in natural and agricultural systems, vegetation structure and community organization, changes in green plant and microbial diversity in relation to microclimate. **Conservation**: *Ecological approach*: Monitoring of plant resources, temporal organization and exploitation of minor resources, technology for nature reserves, *in situ* conservation of threatened species; *Biotechnological approach*: Crop improvement through biotechnology, *ex situ* conservation of wild plants; microbial technologies; *Biological control*: Biology and control of noxious weeds and pests of agricultural systems. **Bioresource technology**: Biofertilizers and biopesticides, integrated pest management. **Diversified approach in sugarcane production**: Application of diversified methods in improving yield and quality of sugarcane.

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National Symposium on Sustainable Aquaculture

Date: 20–21 February 1998
Place: New Delhi

Themes include: Social and environmental relevance of aquaculture: Environmental problems, Social implications, Management options. Biology, methodology and techniques of aquaculture: Fishes, crustaceans and molluscs, seaweeds and micro algae. Aquaculture economics: Production technology, technology transfer, Biotechnology issues and risk assessment, Finance and human resource development. Future of aquaculture: Role of NGOs and Government, Policy and planning.

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