

Crystal structure and vibrational properties of new luminescent hosts K_3YF_6 and K_3GdF_6

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Abstract

The luminescence hosts K_3YF_6 and K_3GdF_6 were obtained in a single-crystal form. Their crystal structure was determined from single-crystal X-ray diffraction data. Both crystals adopt monoclinic system with space group $P2_1/n$, $Z = 2$. Lattice parameters for K_3YF_6 are refined to the following values $a = 6.3376(13) \text{ \AA}$, $b = 6.5435(13) \text{ \AA}$, $c = 9.0390(18) \text{ \AA}$, $\beta = 90.65(3)$ and for K_3GdF_6 $a = 6.3759(13) \text{ \AA}$, $b = 6.5922(13) \text{ \AA}$, $c = 9.1200(18) \text{ \AA}$, $\beta = 90.80(3)$. The vibrational analysis, IR and Raman spectroscopy at room temperature, was applied to these compounds in order to study the site symmetry of Y^{3+} and Gd^{3+} ions.

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1. Introduction

There are a lot articles on phase diagrams of double-fluoride systems with alkaline and lanthanide fluorides [1–5] but the data concerning K_3LnF_6 compounds (where $Ln = Sm\text{--}Lu$, Y) are of the greatest controversy.

In the articles published in the 1970s there was an examination of K_3LnF_6 compositions [6]. The authors presented the symmetry of the system and lattice parameters based on the powder diffraction measurements. Later de Kozak in his papers [4,5] described a binary composition K_3GdF_6 , where he presented a phase diagram and XRD data. In the 1990s, a publication by Fedorov appeared [7]. He demonstrated the experimental phase equilibrium data concerning binary systems of alkali metal fluorides and lanthanide fluorides. This work was actually a summary of previous achievements in this field, so the data concerning K_3GdF_6 were taken from the paper by de Kozak [4,5]. However, at the same time Fedorov emphasized that the data for K_3LnF_6 are often contradictory.

In the papers [4,5,7], the authors claim that K_3GdF_6 compound decomposes upon temperature decrease (below $495 \text{ }^\circ\text{C}$) according to the reaction $K_3GdF_6 \rightarrow K_2GdF_5 + KF$. However, they do not show strong evidence for it and some details of obtaining the compounds $KF\text{--}GdF_3$ of different stoichiometry are not very clear. Still, in powder diffraction files [8] K_3GdF_6 exists.

As it is described in the literature [7] it is possible to obtain K_3YF_6 but it seems impossible to grow congruently melting compounds K_3RF_6 for $R = Sm, Eu, Gd, Tb$. This resulted in the fact that in subsequent papers the physicochemical properties of K_3GdF_6 are omitted in distinction to $KGdF_4$, K_2GdF_5 , KGd_2F_7 and KGd_3F_{10} [9–12].

Our interest in multicomponent fluorides arises from their spectroscopic properties. Such compounds have excellent transparency in the range from IR to far UV. This quality comes from low-energy phonons and high ionicity. To use this unique feature, called wide optical window or large band gap, a small concentration of RE^{3+} ions (activators) is incorporated into matrices. It enables to record absorption peaks attributed to RE^{3+} electronic transitions in UV and even in VUV spectral region. It is also possible to record the luminescence from $4f^{n-1}5d$ level after the excitation with high energetic photon [13].

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The system K_3GdF_6 has many advantages compared to K_3YF_6 . It is possible to obtain the process called quantum cutting in this compound. After the excitation by the high-energy VUV photons one can observe the emission of two visible photons. This is well documented in the pairs Gd–Eu and Gd–(Er, Tb) [14,15].

These properties could be applied to Hg-free fluorescent lamps excited by the vacuum ultraviolet emission (150–180 nm) from a discharge in a noble-gas mixture of Xe and Ne emitting visible light after the VUV excitation, color plasma display panels (PDPs), scintillator materials and others [16].

This paper contains the description of crystal structure of two crystals: K_3GdF_6 and K_3YF_6 . Both of them were synthesized in our laboratory. The most important fact was that we were able to obtain them in the crystal form, not to mention our success in growing K_3GdF_6 —a compound which does not exist according to some scientists [4,5,7]. The vibrational characteristic of these fluorides was also under our consideration.

This publication can be a base for their future precision characteristics in the field of laser spectroscopy.

2. Experimental

2.1. Preparation

The compounds K_3GdF_6 and K_3YF_6 were synthesized using commercially available KF, YF_3 and GdF_3 (Aldrich 99.99%, anhydrous). The reagents were mixed in two batches corresponding to the ratios 3KF:1GdF₃ and 3KF:1YF₃ in the dry glove-box using mortar and pestle. Binary mixture was put into a platinum crucible and covered with platinum cap. Then it was placed in a quartz tube, and after filling the tube with dry argon, the sample was heated up to 1050 °C for about 24 h under the insignificant argon overpressure. After that, the heater was turned off and the temperature was falling down at the pace 3°/min to room temperature.

2.2. Crystallographic studies

Single crystal X-ray diffraction was performed on Kuma KM4CCD diffractometer with graphite-monochromated MoK α radiation. A flat, colorless sample of dimensions $0.2 \times 0.3 \times 0.1 \text{ mm}^3$ for K_3YF_6 and $0.25 \times 0.25 \times 0.1 \text{ mm}^3$ for K_3GdF_6 was chosen under polarizing microscope. The Crys. Alis. software version 1.170.32 (Oxford Diffraction) was used for the data processing. The structure was solved by direct methods and refined by the full-matrix least-squares method by means of SHELX-97 program package—G. M. Sheldrick, SHELX-97, Program for crystal structure determination, University of Cambridge, Cambridge, UK, 1997. Analytical absorption correction for the sample was applied.

2.3. Vibrational studies

Transmission far-infrared spectra were recorded on Bruker IFS-88 spectrometer. It was measured for suspensions in Nujol with resolution 2 cm^{-1} at room temperature. FT-Raman spectra were also measured at room temperature on FRA-106 attachment to Bruker IFS-88 equipped with laser Nd:YAG (1064 nm) with the same resolution.

3. Results

3.1. Crystal structure

The room temperature structure of K_3LnF_6 (where $\text{Ln} = \text{Gd}$ or Y) is monoclinic with space group $P2_1/n$. Lattice parameters for K_3YF_6 are refined to the following values $a = 6.3376(13) \text{ \AA}$, $b = 6.5435(13) \text{ \AA}$, $c = 9.0390(18) \text{ \AA}$, $\beta = 90.65(3)$ and for K_3GdF_6 $a = 6.3759(13) \text{ \AA}$, $b = 6.5922(13) \text{ \AA}$, $c = 9.1200(18) \text{ \AA}$, $\beta = 90.80(3)$. Crystallographic data and details of the structure determination of both crystals are given in Table 1. Further details of the crystal structure investigations can be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (fax: +49 7247 808 666, e-mail: crysdata@fiz.karlsruhe.de) on quoting the depository numbers CSD 416295 and 416296.

The crystal structure of K_3LnF_6 (where $\text{Ln} = \text{Gd}$ or Y) is composed of distorted LnF_6 and $\text{K}(1)\text{F}_6$ octahedra and $\text{K}(2)\text{F}_4$ tetrahedra, Fig. 1.

The LnF_6 and $\text{K}(1)\text{F}_6$ octahedra are linked together by apical F(3) fluorine atoms and form chains which expand along c direction, although they are tilted away from c -axis. Equatorial F(1) and F(2) fluorine atoms are shared by LnF_6 and $\text{K}(1)\text{F}_6$ octahedra in (a,b) plane. The highly disordered $\text{K}(2)\text{F}_4$ tetrahedra join together octahedral chains, Fig. 2. The Ln and $\text{K}(1)$ atoms occupy positions in the center of octahedra with C_i local symmetry. All fluorine ligands and $\text{K}(2)$ atoms occupy general positions. The neighboring Gd atoms are located in average distance of 6.475 \AA whereas average distance between neighboring Y atoms is equal to 6.429 \AA . Atomic coordinates and equivalent isotropic displacement parameters are given in Table 2. Selected bond lengths are presented in Table 3.

The average Gd–F distance of 2.21 \AA correlates well with the value of 2.23 \AA calculated in K_2GdF_5 [17] and value of 2.32 \AA in PtGdF_7 [18]. Also, average Y–F distance of 2.16 \AA fits reasonably well within the value of 2.24 \AA in K_2YF_5 [19].

The expected valence of metal and fluorine atoms (Table 4) has been calculated from the received structure models using the bond-valence method [20]. The valence of Y and Gd is calculated to be +3.00 which is identical with the formal +3 charge of these cations. The valences of all F and K1 atoms are also close to formal charge -1 and $+1$ of F and K ions. The only significant problem indicated by the bond-valence sum is an apparent under-bonding of potassium at K2 position in both compounds. One possible explanation is that the lower value of K2 valence is

Table 1
Crystallographic data and data collection parameters for K_3YF_6 and K_3GdF_6

Crystal data	K_3YF_6	K_3GdF_6
Formula	K_3YF_6	K_3GdF_6
Temperature (K)	293	293
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
Z, Pearson code	$2, mP20$	$2, mP20$
Lattice parameters (Å)	$a = 6.3376(13)$ $b = 6.5435(13)$ $c = 9.0390(18)$ $\beta = 90.65(3)$	$a = 6.3759(13)$ $b = 6.5922(13)$ $c = 9.1200(18)$ $\beta = 90.80(3)$
Volume (Å ³)	374.82	383.29
Formulae per unit cell	2	2
Formula weight	320.21	388.55
Calculated density (g/cm ³)	2.84	3.367
Crystal size (mm)	$0.2 \times 0.3 \times 0.1$	$0.25 \times 0.25 \times 0.1$
Absorption coefficient μ (mm ⁻¹)	9.48	10.31
Data collection		
Radiation type λ (Å)	MoK α /0.71069	MoK α /0.71069
2θ range for data collection (deg)	59.13	58.86
Limiting indices	$-8 < h < 8$ $-8 < k < 7$ $-11 < l < 12$	$-8 < h < 8$ $0 < k < 9$ $0 < l < 12$
Criterion for observed reflections	$I > 2\sigma(I)$	$I > 2\sigma(I)$
No. of reflections collected/unit	4468/971	4443/992
$R(int)$	0.0511	0.0613
Refinement		
Refinement on	F^2	F^2
Program used	ShelXL97	ShelXL97
Data/parameters	971/50	992/50
S_{all}	1.000	1.004
Final R_{obs} , $R_{w obs}$	0.0297, 0.0370	0.0292, 0.0602
Final R_{all} ,	0.0575	0.0507
$\Delta\rho$ (e/Å ³)	0.13	0.24
Extinction method	SHELXL	SHELXL
Extinction coefficient	0.0052(7)	0.0055(8)

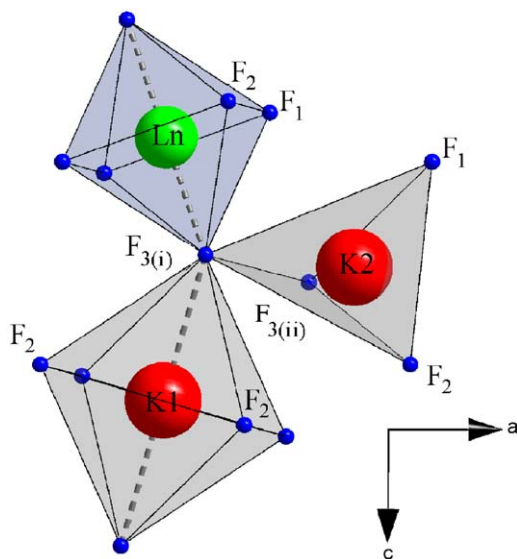


Fig. 1. The coordination polyhedra of Ln, K(1) and K(2) atoms along b direction.

connected with distorted surrounding of this cation. When such distortion occurs the equal-valence rule does not apply [21].

3.2. Vibrational studies

There are several articles devoted to vibrational studies of fluoride compounds [22–26]. Our assignment of the bands observed in IR and Raman spectra, which was partly based on them, confirms the symmetry of the title compounds. We were able to measure K_3LnF_6 (where $Ln = Gd$ or Y) crystals only in the powder form as it was impossible to obtain monocrystals large enough to use in polarized light experiment.

If we considered LnF_6^{3-} complex ion as an ideal octahedron of O_h symmetry we would obtain six modes, of which five are active in vibrational spectra. The modes marked as ν_1 , ν_2 and ν_5 are Raman active, whereas ν_3 and ν_4 are observed in IR. The mode ν_6 is not active neither in Raman nor in IR. Since the crystals are monoclinic at room temperature, the space group $P2_1/n$ (centrosym-

metric), only sites of C_i (2) and C_1 (4) symmetry are allowed. The Ln ions occupy C_i sites—the fact being clearly noticeable in oscillation spectroscopy. That is why we can observe all vibrations of the LnF_6^{3-} groups which are now distorted octahedra. The tentative assignment of the bands along with the wavenumbers is gathered in Table 5. We consider hereafter for simplicity the bands as of LnF_6^{3-} ideal octahedra keeping in mind that the modes are actually no longer of O_h symmetry. In the correlation table (Table 6) the splitting of the bands due to the lowering of symmetry of the LnF_6^{3-} from O_h to C_i is presented. Each degenerate mode can be split into either A_g or A_u due to the site group splitting.

Apart from internal vibrations, the crystals have also external (librations and translations, ν_L and ν_T) and acoustic modes.

Table 3
Selected bond lengths

K_3GdF_6		K_3YF_6	
Gd–F1	2.2069(33)	Y1–F1	2.1534(19)
Gd–F2	2.2144(31)	Y1–F2	2.1665(18)
Gd–F3	2.2142(33)	Y1–F3	2.1635(18)
K1–F1	2.5726(32)	K1–F1	2.5792(19)
K1–F2	2.6485(33)	K1–F2	2.6503(19)
K1–F3	2.6407(34)	K1–F3	2.6264(19)
K2–F1	2.6746(38)	K2–F1	2.6781(23)
K2–F2	2.6467(37)	K2–F2	2.6376(22)
K2–F3 ⁱ	2.6481(37)	K2–F3 ⁱ	2.6510(21)
K2–F3 ⁱⁱ	2.7065(36)	K2–F3 ⁱⁱ	2.7119(20)
K1–K2 ⁱ	3.7191(15)	K1–K2 ⁱ	3.7064(9)
K1–K2 ⁱⁱⁱ	3.8668(18)	K1–K2 ⁱⁱⁱ	3.8373(14)
K1–K2 ^{iv}	4.0494(18)	K1–K2 ^{iv}	4.0076(14)
K1–K2 ^v	4.257(1)	K1–K2 ^v	4.220(1)
Gd–Gd ^{vi}	6.498(67)	Y1–Y1 ^{vi}	6.442(67)
Gd–Gd ^{vii}	6.435(68)	Y1–Y1 ^{vii}	6.391(67)
Gd–Gd ^{viii}	6.376(1)	Y1–Y1 ^{viii}	6.338(1)
Gd–Gd ^{ix}	6.592(1)	Y1–Y1 ^{ix}	6.543(1)

Symmetry codes: K_3LnF_6 . (i) $x, 1-y, -z$; (ii) $-0.5+x, 1.5-y, -0.5+z$; (iii) $0.5+x, 0.5-y, -0.5+z$; (vi) $-0.5+x, 0.5-y, -0.5+z$; (v) x, y, z ; (vii) $-0.5-x, -0.5+y, 0.5-z$; (viii) $0.5-x, 0.5+y, 0.5-z$; (ix) $x, 1+y, z$.

Table 4
Bond-valence sums [20] for final structure models of K_3GdF_6 and K_3YF_6

K_3YF_6		K_3GdF_6	
Y	3.00	Gd	3.00
K1	1.11	K1	1.17
K2	0.76	K2	0.79
F1	0.96	F1	0.99
F2	0.90	F2	0.94
F3	0.99	F3	0.99

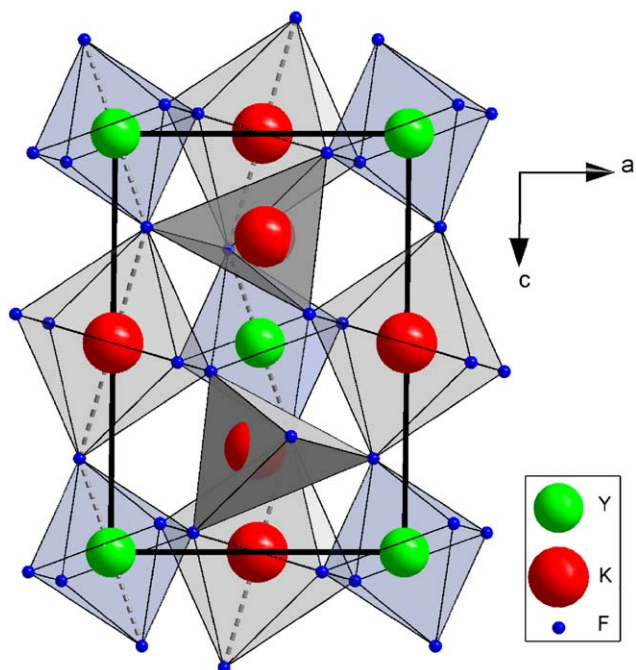


Fig. 2. Projection of a unit cell along b direction in K_3LnF_6 (where $Ln = Gd$ or Y). The bottom layer of polyhedra was removed for view's clarity.

Table 2
Positional parameters for K_3YF_6 and K_3GdF_6

Atom	Wyckoff position	Site symmetry	Sof.	x	y	z	U_{equ}
K_3GdF_6							
Gd	2a	$-I$	1	0	0	0	0.01640(15)
K1	2b	$-I$	1	1/2	1/2	0	0.02174(32)
K2	4e	I	1	0.012688(20)	0.549398(20)	-0.254660(16)	0.03283(32)
F1	4e	I	1	0.214763(53)	0.324099(52)	0.548496(42)	0.04505(96)
F2	4e	I	1	0.331615(53)	0.776461(50)	0.570230(42)	0.04490(98)
F3	4e	I	1	0.386128(53)	0.560871(52)	0.273779(36)	0.03637(83)
K_3YF_6							
Y	2a	$-I$	1	0	0	0	0.01564(16)
K1	2b	$-I$	1	1/2	1/2	0	0.02029(23)
K2	4e	I	1	0.01253(14)	0.54757(11)	-0.25335(10)	0.03133(22)
F2	4e	I	1	0.22045(31)	0.32549(30)	0.54659(25)	0.04155(64)
F1	4e	I	1	0.33320(30)	0.77259(29)	0.56821(23)	0.04155(64)
F3	4e	I	1	0.39053(30)	0.55744(28)	0.27627(20)	0.03638(58)

Table 5
The assignment of the bands in IR and Raman spectra of K_3YF_6 and K_3GdF_6

Compound	$\nu_1 (A_{1g}) (R)$	$\nu_2 (E_g) (R)$	$\nu_3 (F_{1u}) (IR)$	$\nu_4 (F_{1u}) (IR)$	$\nu_5 (F_{2g}) (R)$	$\nu_6 (F_{2u}) (IR)$	$\nu_T (F_{2g}) (R)$
K_3YF_6	463 (s)		441 sh	290 sh	244 (w)	105 (m)	135 (w)
	450 (w)		404 (s)	266 (w)	216 (s)		133 (w)
			389 (s)	211 (s)	190 (w)		120 (s)
			350 sh	167 (m)			112 (w)
				141 (m)			
				137 (m)			
K_3GdF_6	465 (sh)		370 (s)	293 (m)	228 (sh)	101 (w)	139 (w)
	457 (s)		360 (s)	278 (m)	218 (sh)		133 (w)
				214 (s)	210 (s)		121 (m)
				192 (sh)	201 (sh)		112 (w)
				153 (w)			
				136 (w)			

The numbers are given in cm^{-1} .

Table 6
The correlation table for LnF_6^{3-} octahedra

	O_h	C_i
ν_1	A_{1g}	A_g
ν_2	E_g	$2A_g$
ν_5	F_{2g}	$3A_g$
ν_3	F_{1u}	$3A_u$
ν_4	F_{1u}	$3A_u$
ν_6	F_{2u}	$3A_u$

In the K_3LnF_6 IR spectrum (Fig. 3) there is a broad band with maximum at ca. 404 cm^{-1} for Y^{3+} and at 370 cm^{-1} for Gd^{3+} assigned to ν_3 mode. In this work, as well as in some others [23,24], this band is regarded as $Ln-F-K$ stretching vibration. It possesses a shoulder at higher frequency (ca. 441 cm^{-1}) in K_3YF_6 which could be due to the site splitting. We do not observe such shoulders in Gd-containing crystal, probably because Gd–Gd distance is bigger than the one of Y–Y. This implies weaker interactions between the ions and, in consequence, since the distorted octahedra have one fluorine atom in common, Gd atom does not have such a great impact on the other atoms. Another broad band at 211 cm^{-1} (Y^{3+}) and 214 cm^{-1} (Gd^{3+}) was attributed to ν_4 (F_{1u}) ($Ln-F-K$ bending) mode, however, we cannot explain a very rich structure of this mode. Most probably it originates from the overlapping of lattice vibrations with the bending modes of LnF_6^{3-} in the crystal. The peak with maximum at 105 and 101 cm^{-1} , respectively, belongs to the libration of the polyhedra (ν_6 (F_{2u}) type).

In the case of Raman spectrum of YF_6^{3-} ideal octahedron we should observe four peaks according to some works [8,9] at 466 (ν_1), 380 (ν_2), 218 (ν_5) and 120 (ν_T) cm^{-1} , whereas in our experiments three bands are observed (see Fig. 4) for both Y^{3+} and Gd^{3+} crystals. The band at 463 and at 457 cm^{-1} , respectively, is $Ln-F$ symmetric stretching vibration (ν_1) and the band at 216 cm^{-1} (Y^{3+}) and at 210 cm^{-1} (Gd^{3+}) is thought to be

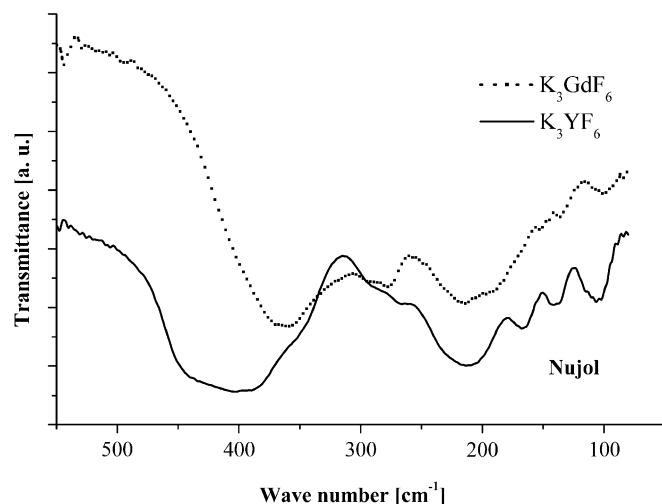


Fig. 3. Far-infrared absorption spectrum of K_3YF_6 and K_3GdF_6 taken at room temperature.

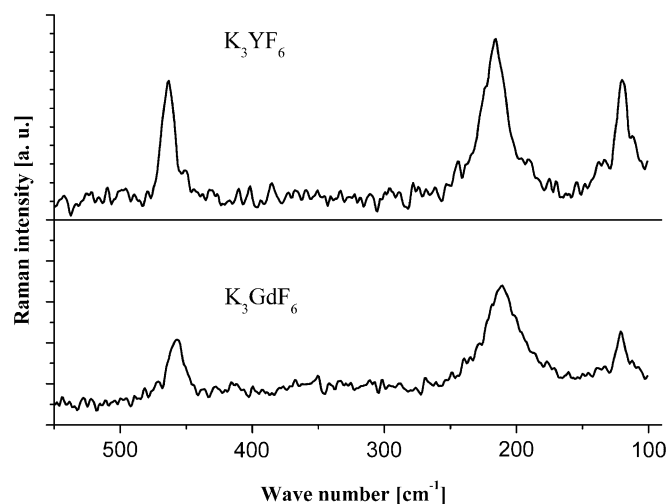


Fig. 4. Raman spectrum of K_3YF_6 and K_3GdF_6 taken at room temperature.

ν_5 (F_{2g}) mode of the polyhedra. The last peak at 120 and at 121 cm^{-1} , respectively, is assigned to lattice vibration. We are not able to observe ν_2 band, which should be at around 380 cm^{-1} for both crystals [22], because its intensity is too small. All the bands are asymmetrical and broadened which suggests again interactions in the crystal (especially in Y^{3+} -containing sample) due to the distance between lanthanide atoms. We can neglect the influence of potassium polyhedra because the average K–F distance is far longer than that of Gd–F. Hence, the impact of potassium polyhedra on the vibrations of Gd polyhedra should be little. The asymmetric shape of the bands confirms also low site symmetry of LnF_6^{3-} polyhedra.

5. Conclusions

We succeeded in obtaining K_3YF_6 and K_3GdF_6 in the form of single crystals, thereby giving a very strong evidence for the existence of the latter compound. The conditions of growth have been found but we need some improvements as far as crystals of good optical quality are concerned.

It has been proved, with the use of single-crystal X-ray diffraction and vibrational spectroscopy, that the crystal structures of both compounds are the same. We also determined the site symmetry of the lanthanide ions.

These materials are suitable for the design of lumino-phores. They possess low-energy phonons, the maximum is at around 450 cm^{-1} . This property plays an important role in nonradiative (multiphonon) relaxation for most of lanthanide excited states improving the light emission. Our present aim is to obtain crystals containing rare earth dopants and study them using laser spectroscopy. We would like to carry out research on absorption, luminescence and lifetimes of excited states of the doped ions.

Acknowledgments

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