

# CRYSTAL STRUCTURE ANALYSIS OF $K_3VF_6$ COMPOUND

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## Abstract

The object of research is the crystal structure of the  $\alpha$  phase of  $K_3VF_6$  compound. From the analysis of the literature it follows that there are three modifications of this compound – the  $\alpha$  phase, the  $\beta$  phase and the  $\gamma$  phase. There are also several methods for preparing this compound. One of the problem areas is the existence of a large number of diffraction spectra obtained with the Bragg-Bertrand survey geometry in the PDF-2 database for 2004. There is also no complete data on the crystal structure of this compound. A structural model is proposed for the diffraction spectrum of the compound under the number 00-037-0738.

The study used the PDF-2 database for 2004. As well as the HighScorePlus program, which allows to refine the microstructural parameters of the structural model by the Rietveld method.

As a result, it is found that this diffraction spectrum of the compound under study can correspond to the following structural model: tetragonal system with lattice periods  $a=12.3524(4)$  Å;  $b=12.3524(4)$  Å;  $c=16.7447(7)$  Å. A possible spatial symmetry group is  $I4_1/a$  (88).

Analyzing the obtained results, it can be assumed that the studied structure of the  $\alpha$  phase of the compound crystallizes in its own structural type.

**Keywords:** X-ray diffraction analysis, Bragg-Bertrand survey geometry, Rietveld method, crystal structure,  $K_3VF_6$  composition.

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## 1. Introduction

The synthesis, crystalline structure and properties of mixed secondary metals are becoming an area of interest for scientists of material science for their isoelectronic and isostructural nature with their oxide counterparts [1].

Unlike oxides, the synthesis of mixed metal fluorides is limited due to the complexity that occurs during its synthesis, and therefore many interesting properties inherent in these metal fluorides are intensively studied [1, 2].

The general methods adopted for the synthesis are high temperature solid-state synthesis, high hydrothermal pressure (100 MPa), sol-gel synthesis and a solution based on chemical synthesis.

$K_3VF_6$  compound can be obtained by light synthesis at room temperature for V (III) and potassium-containing KF secondarily.

The non-hygroscopic nature of  $K_3VF_6$  makes it an ideal electrolyte component for electrorefining or electrostriction of vanadium compared to other candidates such as  $VCl_2$  and  $VCl_3$ , as well as in electrochemical cells [3, 4].

Therefore, investigation of the crystal structure of this compound remains relevant.

The object of research is the crystal structure of the  $\alpha$  phase of  $K_3VF_6$  compound.

At the same time, it follows from the literature that there are six diffraction spectra of the indicated compound obtained by X-ray powder diffraction (Bragg-Bertrand method) of different quality.

The aim of this research is to propose a structural model for the diffraction spectrum of the  $K_3VF_6$  compound under the number 00-037-0738 in the database PDF-2 for 2004.

## 2. Materials and methods

To achieve this aim it is necessary to solve the following objectives:

- to index the diffraction spectrum of the compound at number 3 (according to **Table 1**);
- to determine the lattice and syngony periods;

- to choose a spatial symmetry group for a given spectrum of the compound;
- to propose a structural model for a given spectrum of the compound;
- to refine the microstructural parameters for the selected model by the Rietveld method.

Since part of  $\text{KHF}_2$  in the mixture decomposes without reaction with  $\text{V}_2\text{O}_3$ , the excess  $\text{KHF}_2$  necessary for the preparation of  $\text{K}_3\text{VF}_6$  compound. In a 300-gram portion of each mixture in molar ratios of 1:6, 1:6.5, 1:7, 1:8, 1:9, 1:10, 1:11 and 1: 12 were heated at 600 °C in a stream argon (200 ml/min.) using certain equipment described in detail in [5]. After cooling, the reaction products were washed with water, and the residue was examined with the naked eye and also by X-ray diffraction. Vanadium (III) oxide is black or brown, and  $\text{K}_3\text{VF}_6$  is bright green. When the reaction product is washed with water, the remaining parts of  $\text{V}_2\text{O}_3$  can be well separated from  $\text{K}_3\text{VF}_6$  in water. Mixtures in molar ratios of 1:7–1:12 gave  $\text{K}_3\text{VF}_6$  pure [5], **Table 1**.

**Table 1**  
Details of the  $\text{K}_3\text{VF}_6$  crystal structure

No.	Compound	Syngony, SSG	Lattice periods, Å°	Card No. in the pdf-2 base	Diffraction spectrum quality
1	$\text{K}_3\text{VF}_6$	Cubic, F	$a=8.7$ $b=8.7$ $c=8.7$	00-022-0868	Indexed [6]
2	$\text{K}_3\text{VF}_6$	Not known	Lattice periods are not defined	00-028-0841	Low spectrum accuracy [7]
3	$\text{K}_3\text{VF}_6$	Monoclinic	$a=12.1120$ $b=17.4740$ $c=12.0190$ $\beta=92.59$	00-037-0738	Highest Spectrum Quality [8]
4	$\text{K}_3\text{VF}_6$	Monoclinic	$a=17.136$ $b=12.368$ $c=5.479$ $\beta=91.485$	00-037-0739	Indexed [8]
5	$\text{K}_3\text{VF}_6$	Tetragonal, I	$a=13.708$ $b=13.708$ $c=8.752$	00-038-0541	Indexed [8]
6	$\text{K}_3\text{VF}_6$	Tetragonal, I	$a=13.570$ $b=13.570$ $c=8.671$	00-038-0542	Indexed [8]

According to [8], the diffraction spectrum 00-037-0738 in the PDF-2 database of  $\text{K}_3\text{VF}_6$  compound obtained at 298 0K as  $\alpha$  phase, numbered 00-037-0739 at 453 K, as  $\beta$  phase, and are indexed in monoclinic syngony, and 00-038-0541  $\gamma$  phase is formed at 483 K, and is indexed in tetragonal syngony.

An Internet source [13] indicates that the structure of the 3 phase of  $\text{K}_3\text{VF}_6$  belongs to the structural type  $[\text{NH}_4]_3\text{FeF}_6$  of the space group Fm-3m (225).

Spectrum number 3 in the Table 1, in my opinion, deserves special attention due to its high quality of shooting (as indicated in the card of this spectrum given in the PDF-2 database for 2004), as well as the lack of literature data on a detailed study of the structure of  $\text{K}_3\text{VF}_6$  compound. According to [8], this diffraction spectrum is indexed in monoclinic syngony (**Fig. 1**).

As a result of the research, it is found that this system is a simple quasibinary system and for KCl-K it has a eutectic temperature of 604 °C with a composition of 55 mol % KCl, and for KCl- $\text{K}_3\text{VF}_6$  the eutectic point is at 741 °C and 7.5 mol %  $\text{K}_3\text{VF}_6$ . It is also found that KF- $\text{K}_3\text{VF}_6$  is a simple eutectic type at a temperature of 821 °C and 7.5 mol %  $\text{K}_3\text{VF}_6$ . The system and the

triple eutectic point at 594 °C at the point KCl 54.8, KF 44,  $K_3VF_6$  1.2 mol.%. Thus, the eutectic points have a much higher temperature than the temperature of formation of the  $K_3VF_6$  phases according to [8].

Also in [10], the importance of F – as a stabilizing ligand for V (III) and Mn (III) is emphasized, both in aqueous solution and in solid compounds. Many fluorine and mixed fluorine complexes have been synthesized from an aqueous solution.  $VF(C_5H_7O_2)_2$ ,  $Na_3VF_6$  and  $K_3VF_6$  compounds are obtained on the basis of electron transfer processes, including vanadium (V) and harmful reducing agents.

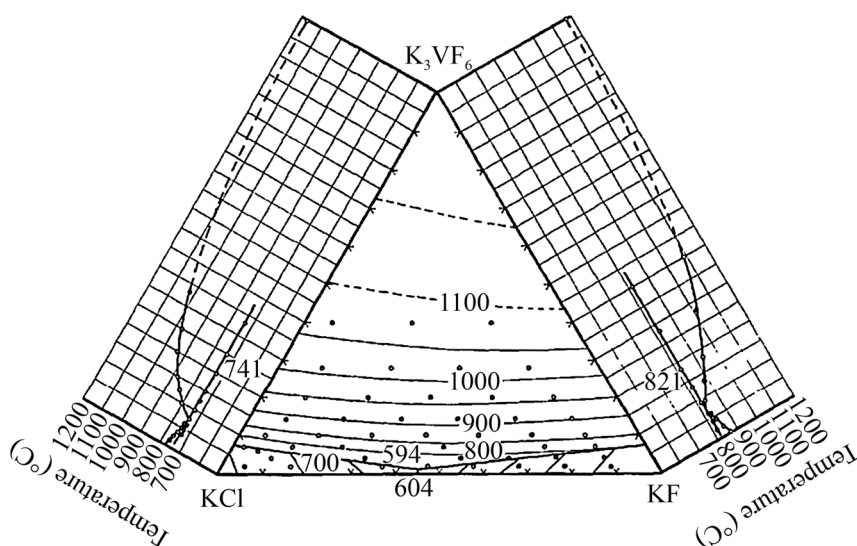


Fig. 1. System status diagram KCl-KF- $K_3VF_6$

In [11], the creation of the  $K_2VF_6$  compound by fluorination with  $K_3VF_6$  is noted. However, there is no information on the crystal structure of the input compound.

According to [12],  $K_3VF_6$  is obtained by the interaction of powder V with  $KHF_2$ . Green crystalline  $K_3VF_6$  is separated by sublimation from Reacting V metal and from KF, which is formed in the reaction and is detected by chemical analysis. Previously,  $K_3VF_6$  is prepared by reacting  $VF_3$  with KF. Vanadium powder is heated with excess  $KHF_2$  in a platinum crucible to 600 °C, and slowly rises to 800 °C. The product, a pale green melt, is then heated for a long time at 1200 °C in a graphite crucible. Bright green crystals condense in the upper parts of the crucible. All excess KF and a small portion of the green product crystallized on colder thermal screens.

The diffraction spectrum for the study is generated using the HighScorePlus 3.0 program [14] and the attached pdf-2 database for 2004 in the UDF format.

An analysis of the proposed structural model of this spectrum is also performed using the HighScorePlus 3.0 program by the Rietveld method. Obtaining initial data for studying the crystal structure of the compound, exclusively data taken from the PDF-2 database for 2004 are used.

### 3. Research results

The diffraction spectrum of the compound is indexed in tetragonal system with lattice periods  $a=12.3524(4)$  Å;  $b=12.3524(4)$  Å;  $c=16.7447(7)$  Å. The space group of symmetry I 41/a (88) is possible.

An analysis of the proposed structural model of this spectrum is also performed using the HighScorePlus 3.0 program by the Rietveld method.

The correct system of points and their coordinates for this spectrum are specified in Table 2.

**Table 2**  
K<sub>3</sub>VF<sub>6</sub> microstructural parameters

Atom	The correct point system	x	y	z	Position fill ratio	$U_{iso}^a$
K1	16f	0.2313(8)	0.7620(8)	0.4875(5)	1.000000	0.2(2)
K2	16f	0.239(1)	0.016(1)	0.1285(9)	1.000000	4.0(5)
K3	16f	0.045(2)	0.061(3)	0.146(2)	0.500000	10(1)
K4	16f	0.149(2)	0.658(2)	0.509(1)	0.500000	0(1)
V1	8e	0.000000	0.250000	0.3777(8)	1.000000	5(1)
V2	4b	0.000000	0.250000	0.625000	1.000000	10(1)
V3	4a	0.000000	0.250000	0.125000	1.000000	8(1)
F1	16f	0.620(2)	−0.910(2)	−0.291(1)	1.000000	0(1)
F2	8d	0.000000	0.000000	0.500000	1.0(2)	0(1)
F3	16f	0.123(2)	0.378(2)	0.258(1)	1.000000	0(1)
F4	16f	0.311(3)	0.666(2)	0.400(2)	1.000000	10(1)
F5	8e	0.000000	0.250000	−0.006(7)	0.341316	10(6)
F6	16f	0.252(2)	0.530(3)	0.485(2)	1.000000	8(1)
F7	16f	0.090(3)	0.159(2)	0.259(2)	1.000000	7(1)
F8	8e	0.000000	0.250000	0.689(4)	0.658684	10(2)

The values of interplanar distances and intensities observed and calculated by this model are given in **Table 3**.

**Table 3**  
The values of interplanar distances and intensities, which are observed and calculated

$d_{cal}$ (Å°)	$d_{obs}$ (Å°)	$I_{cal}$	$I_{obs}$	$H$	$K$	$L$	$d_{cal}$ (Å°)	$d_{obs}$ (Å°)	$I_{cal}$	$I_{obs}$	$H$	$K$	$L$
1	2	3	4	5	6	7	8	9	10	11	12	13	14
9.93	–	1.20	–	0	1	1	1.58	–	1.43	–	4	1	9
6.17	–	0.54	–	0	2	0	1.57	–	1.12	–	5	6	1
6.04	–	0.23	–	1	1	2	1.57	–	0.09	–	6	5	1
5.24	5.24	2.31	0.93	1	2	1	1.56	–	1.07	–	2	2	10
5.24	–	0.51	–	2	1	1	1.56	–	0.21	–	0	7	5
5.08	–	0.82	–	0	1	3	1.54	1.54	0.38	6.21	1	6	7
4.96	4.96	90.8	80.9	0	2	2	1.54	–	0.01	–	6	1	7
4.36	4.36	24.3	24.2	2	2	0	1.54	–	5.94	–	0	8	0
4.18	4.18	3.77	5.09	0	0	4	1.53	–	0.08	–	1	3	10
3.99	4.00	1.64	1.09	0	3	1	1.53	–	0.00	–	3	1	10
3.92	3.92	0.18	1.09	1	2	3	1.52	1.52	0.22	1.05	1	8	1
3.92	–	0.89	–	2	1	3	1.52	–	0.02	–	8	1	1
3.87	–	0.04	–	2	2	2	1.52	–	0.26	–	4	7	1
3.77	3.78	2.64	2.18	1	1	4	1.52	–	0.86	–	7	4	1
3.53	3.53	0.40	1.11	1	3	2	1.52	–	0.00	–	5	6	3
3.53	–	2.81	–	3	1	2	1.52	–	0.24	–	6	5	3
3.46	3.46	1.90	1.08	0	2	4	1.51	–	0.03	–	0	8	2
3.35	3.35	2.37	4.17	2	3	1	1.51	–	0.01	–	2	7	5
3.35	–	1.95	–	3	2	1	1.51	–	0.18	–	7	2	5

Continuation of Table 3

1	2	3	4	5	6	7	8	9	10	11	12	13	14
3.31	3.31	8.97	7.26	0	3	3	1.51	—	0.05	—	3	7	4
3.23	3.23	2.13	2.05	0	1	5	1.51	—	0.09	—	7	3	4
3.08	3.08	41.2	33.6	0	4	0	1.51	1.51	11.8	10.3	4	4	8
3.02	3.02	100.	100.	2	2	4	1.51	—	0.14	—	0	1	11
2.94	2.94	0.13	1.98	1	4	1	1.50	—	0.14	—	4	5	7
2.94	—	4.21	—	4	1	1	1.50	—	0.07	—	5	4	7
2.91	2.91	0.62	1.99	3	2	3	1.49	—	0.05	—	2	8	0
2.91	—	2.84	—	2	3	3	1.49	—	0.20	—	8	2	0
2.89	—	0.08	—	0	4	2	1.48	—	0.17	—	3	5	8
2.86	2.87	0.32	2.19	1	2	5	1.48	—	0.43	—	5	3	8
2.86	—	3.61	—	2	1	5	1.48	—	0.02	—	0	5	9
2.85	—	0.08	—	1	3	4	1.48	—	0.26	—	3	4	9
2.85	—	0.00	—	3	1	4	1.48	—	0.62	—	4	3	9
2.76	2.76	1.01	2.19	2	4	0	1.48	—	0.00	—	1	7	6
2.76	—	1.52	—	4	2	0	1.48	—	0.04	—	5	5	6
2.74	—	2.06	—	3	3	2	1.48	—	0.26	—	7	1	6
2.65	—	1.02	—	1	1	6	1.47	1.47	0.37	2.08	1	8	3
2.63	2.63	1.66	2.17	1	4	3	1.47	—	0.38	—	4	7	3
2.63	—	0.45	—	4	1	3	1.47	—	0.40	—	7	4	3
2.62	—	1.27	—	2	4	2	1.47	—	0.98	—	8	1	3
2.62	—	2.18	—	4	2	2	1.47	—	0.30	—	2	8	2
2.59	2.59	1.51	2.18	0	3	5	1.47	—	0.28	—	8	2	2
2.54	—	2.66	—	0	2	6	1.47	—	0.01	—	0	4	10
2.48	2.48	25.5	21.8	0	4	4	1.46	—	0.12	—	0	6	8
2.44	2.45	0.00	11.4	0	5	1	1.46	—	0.01	—	2	1	11
2.44	—	4.45	—	3	4	1	1.46	—	0.37	—	1	2	11
2.44	—	0.93	—	4	3	1	1.45	1.45	1.01	2.02	4	6	6
2.39	2.39	2.92	10.4	0	2	5	1.45	—	0.88	—	6	4	6
2.39	—	4.84	—	2	3	5	1.45	—	0.27	—	3	6	7
2.38	—	0.13	—	3	3	4	1.45	—	0.00	—	6	3	7
2.35	—	5.72	—	2	2	6	1.45	1.45	0.01	3.06	6	6	0
2.34	2.34	1.08	9.49		1	7	1.45	—	0.58	—	3	3	10
2.32	2.32	1.26	2.09	1	5	2	1.44	1.44	0.61	2.03	0	8	4
2.32	—	0.09	—	5	1	2	1.44	—	0.08	—	2	5	9
2.30	2.30	7.10	5.27	2	4	4	1.44	—	0.09	—	5	2	9
2.30	—	0.04	—	4	2	4	1.44	—	0.01	—	3	8	1
2.27	2.27	1.99	7.31	5	2	1	1.44	—	0.03	—	8	3	1
2.27	—	0.31	—	2	5	1	1.43	1.43	0.01	3.04	6	6	2
2.27	—	2.10	—	1	3	6	1.43	—	0.06	—	2	4	10
2.27	—	2.20	—	3	1	6	1.43	—	0.31	—	4	2	10
2.25	2.25	2.40	8.44	0	5	3	1.42	—	0.01	—	5	6	5
2.25	—	1.04	—	3	4	3	1.42	—	0.15	—	6	5	5
2.25	—	6.72	—	4	3	3	1.42	1.42	0.15	3.04	2	6	8
2.23	2.23	4.73	6.19	1	4	5	1.42	—	0.17	—	6	2	8

Continuation of Table 3

1	2	3	4	5	6	7	8	9	10	11	12	13	14
2.23	–	1.14	–	4	1	5	1.42	–	0.01	–	0	3	11
2.19	–	0.12	–	1	2	7	1.41	–	0.02	–	0	7	7
2.19	–	0.22	–	2	1	7	1.41	1.41	0.12	1.02	5	7	2
2.18	2.18	63.5	54.7	4	4	0	1.41	–	0.00	–	7	5	2
2.12	–	0.45	–	2	5	3	1.41	1.41	0.08	1.02	2	8	4
2.12	–	0.01	–	5	2	3	1.41	–	0.50	–	8	2	4
2.11	2.11	0.05	2.09	4	4	2	1.40	–	0.02	–	3	7	6
2.09	2.09	0.79	28.2	1	5	4	1.40	–	0.00	–	7	3	6
2.09	–	0.07	–	5	1	4	1.39	–	0.07	–	3	8	3
2.09	–	25.0	–	0	0	8	1.39	–	0.24	–	8	3	3
2.07	–	0.01	–	0	4	6	1.39	–	0.02	–	0	0	12
2.06	–	1.25	–	0	3	7	1.39	1.39	0.25	1.02	1	8	5
2.05	–	0.01	–	0	6	0	1.39	–	0.77	4.10	4	7	5
2.05	–	0.93	–	3	5	2	1.39	–	0.25	3.07	7	4	5
2.05	–	0.07	–	5	3	2	1.39	–	0.09	2.05	8	1	5
2.03	2.03	0.63	1.09	1	1	8	1.39	–	0.03	–	2	3	11
2.01	2.01	0.67	2.16	1	6	1	1.39	–	0.11	–	3	2	11
2.01	–	0.19	–	6	1	1	1.38	–	0.24	–	2	7	7
2.01	–	0.58	–	3	3	6	1.38	–	0.18	–	7	2	7
1.99	–	0.11	–	0	6	2	1.38	1.38	1.00	4.10	4	8	0
1.98	1.98	2.14	18.8	0	5	5	1.38	–	0.86	–	8	4	0
1.98	–	1.45	–	3	4	5	1.37	–	0.06	–	1	1	12
1.98	–	11.8	–	4	3	5	1.37	–	0.39	–	1	5	10
1.98	–	0.02	–	0	2	8	1.37	–	0.01	–	5	1	10
1.96	1.96	0.30	2.15	2	4	6	1.37	1.37	3.50	3.07	6	6	4
1.96	–	1.50	–	4	2	6	1.37	–	0.23	–	1	6	9
1.96	–	0.06	–	2	3	7	1.37	–	0.32	–	6	1	9
1.96	–	1.18	–	3	2	7	1.36	–	0.03	–	0	9	1
1.95	1.95	1.29	2.12	2	6	0	1.36	–	0.36	–	4	8	2
1.95	–	3.83	–	6	2	0	1.36	–	0.29	–	8	4	2
1.93	1.93	3.10	3.18	4	4	4	1.36	–	0.03	–	0	2	12
1.91	–	0.89	–	4	5	1	1.35	–	0.00	–	5	7	4
1.91	–	0.29	–	5	4	1	1.35	–	0.00	–	7	5	4
1.90	1.90	0.00	2.08	1	6	3	1.35	–	0.00	–	1	4	11
1.90	–	0.18	–	6	1	3	1.35	–	0.01	–	4	1	11
1.90	–	0.76	–	2	6	2	1.35	–	0.01	–	0	8	6
1.90	–	2.04	–	6	2	2	1.34	–	0.01	–	1	9	2
1.89	1.89	0.04	3.15	5	2	5	1.34	–	0.02	–	9	1	2
1.89	–	1.26	–	2	5	5	1.34	–	0.26	–	1	7	8
1.88	–	0.90	–	3	5	4	1.34	–	0.01	–	7	1	8
1.88	–	1.12	–	5	3	4	1.34	–	0.06	–	5	5	8
1.88	–	1.19	–	2	2	8	1.33	–	0.04	–	4	5	9
1.86	1.86	0.03	1.10	1	4	7	1.33	–	0.39	–	5	4	9
1.86	–	0.39	–	4	1	7	1.33	1.33	0.58	2.05	2	9	1

Continuation of Table 3

1	2	3	4	5	6	7	8	9	10	11	12	13	14
1.84	1.84	0.58	2.12	0	6	4	1.33	—	0.05	—	6	7	1
1.84	—	0.26	—	1	3	8	1.33	—	0.48	—	7	6	1
1.84	—	0.58	—	3	1	8	1.33	—	1.01	—	9	2	1
1.83	—	0.01	—	0	1	9	1.33	—	0.09	—	0	9	3
1.83	—	0.85	—	3	6	1	1.32	—	2.35	—	2	2	12
1.83	—	0.00	—	6	3	1	1.32	—	0.00	—	4	4	10
1.82	1.82	1.00	2.13	1	5	6	1.32	—	0.00	—	3	8	5
1.82	—	0.61	—	5	1	6	1.32	—	0.02	—	8	3	5
1.82	—	0.02	—	4	5	3	1.32	—	0.06	—	4	6	8
1.82	—	1.22	—	5	4	3	1.32	—	0.31	—	6	4	8
1.76	1.76	18.1	25.9	2	6	4	1.31	—	0.23	—	2	8	6
1.76	—	14.6	—	6	2	4	1.31	—	0.12	—	8	2	6
1.76	—	0.83	—	2	1	9	1.31	—	0.05	—	5	6	7
1.76	—	0.04	—	1	2	9	1.31	—	0.02	—	6	5	7
1.75	—	0.59	—	0	7	1	1.31	—	0.08	—	1	3	12
1.74	—	0.15	—	3	6	3	1.31	—	0.02	—	3	1	12
1.74	—	0.00	—	6	3	3	1.31	—	0.23	—	3	5	10
1.73	1.73	1.02	11.4	1	6	5	1.31	—	0.06	—	5	3	10
1.73	—	0.86	—	6	1	5	1.31	—	0.14	—	4	8	4
1.73	—	8.94	—	0	4	8	1.31	—	0.17	—	8	4	4
1.71	1.71	0.66	2.11	4	4	6	1.30	—	0.18	—	3	6	9
1.71	—	0.23	—	4	3	7	1.30	—	0.01	—	6	3	9
1.71	—	0.42	—	0	5	7	1.30	—	0.20	—	5	8	1
1.71	—	0.01	—	3	4	7	1.30	—	0.11	—	8	5	1
1.71	—	0.16	—	4	6	0	1.30	—	0.02	—	6	7	3
1.71	—	0.46	—	6	4	0	1.30	—	0.09	—	2	9	3
1.70	—	0.00	—	1	7	2	1.30	—	0.00	—	7	6	3
1.70	—	0.27	—	7	1	2	1.30	—	0.11	—	9	2	3
1.70	—	0.67	—	5	5	2	1.29	—	0.00	—	0	6	10
1.69	—	0.39	—	3	3	8	1.29	—	0.14	—	1	9	4
1.69	—	1.35	—	0	3	9	1.29	—	0.05	—	9	1	4
1.68	—	0.63	—	2	7	1	1.29	—	0.45	—	0	5	11
1.68	—	1.04	—	7	2	1	1.29	—	0.06	—	3	4	11
1.68	—	0.06	—	3	5	6	1.29	—	0.00	—	4	3	11
1.68	—	0.06	—	5	3	6	1.29	—	0.06	—	6	6	6
1.68	—	0.20	—	0	7	3	1.29	—	0.16	—	1	8	7
1.67	1.67	4.42	7.24	4	6	2	1.29	—	0.01	—	4	7	7
1.67	—	2.48	—	6	4	2	1.29	—	0.23	—	7	4	7
1.67	1.67	1.84	3.02	4	5	5	1.29	—	0.00	—	8	1	7
1.67	—	0.00	—	5	4	5	1.28	—	0.04	—	9	3	2
1.66	—	0.71	—	2	4	8	1.28	—	0.01	—	3	9	2
1.66	—	0.01	—	4	2	8	1.28	—	0.07	—	7	3	8
1.65	1.65	0.20	4.17	0	6	6	1.28	—	0.11	—	3	7	8
1.65	—	0.37	—	2	5	7	1.28	—	0.11	—	0	1	13

Continuation of Table 3

1	2	3	4	5	6	7	8	9	10	11	12	13	14
1.65	–	3.04	–	5	2	7	1.28	–	0.08	–	0	7	9
1.64	–	0.57	–	1	1	10	1.27	–	0.12	–	5	7	6
1.63	–	1.10	–	2	3	9	1.27	–	0.10	–	7	5	6
1.63	–	1.08	–	3	2	9	1.27	–	0.33	–	5	8	3
1.62	–	0.04	–	2	7	3	1.27	–	0.04	–	8	5	3
1.62	–	0.00	–	7	2	3	1.27	–	0.16	–	0	4	12
1.61	1.61	1.37	4.17	0	2	10	1.27	–	0.38	–	2	6	10
1.61	–	0.44	–	3	6	5	1.27	–	0.54	–	6	2	10
1.61	–	0.02	–	6	3	5	1.26	–	0.11	–	0	9	5
1.61	–	0.30	–	1	7	4	1.26	–	0.00	–	2	5	11
1.61	–	0.04	–	7	1	4	1.26	–	0.36	–	5	2	11
1.61	–	0.03	–	5	5	4	1.58	–	1.43	–	4	1	9
1.60	–	0.39	–	2	6	6	1.57	–	1.12	–	5	6	1
1.60	–	0.88	–	6	2	6	1.57	–	0.09	–	6	5	1
1.59	–	0.00	–	3	7	2	1.56	–	1.07	–	2	2	10
1.59	–	0.00	–	7	3	2	1.56	–	0.21	–	0	7	5
1.58	1.58	0.04	1.07	4	6	4	1.54	1.54	0.38	6.21	1	6	7
1.58	–	0.01	–	6	4	4	1.54	–	0.01	–	6	1	7
1.58	–	0.00	–	5	1	8	1.54	–	5.94	–	0	8	0
1.58	–	0.02	–	1	5	8	1.53	–	0.08	–	1	3	10
1.58	–	0.34	–	1	4	9	1.53	–	0.00	–	3	1	10
1.58	–	1.43	–	4	1	9	1.52	1.52	0.22	1.05	1	8	1

The disagreement factor is  $R=9.8845\%$ .

**Table 4** shows the interatomic distances of the  $K_3VF_6$  compound under the number 00-037-0738 in the pdf-2 database for 2004.

**Table 4**

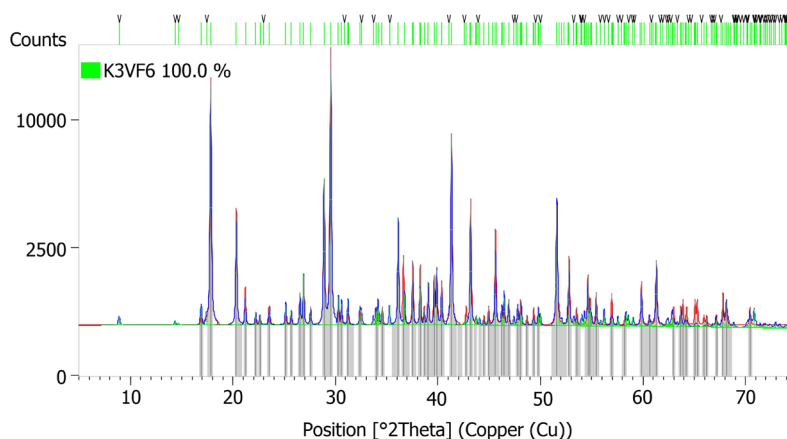
Interatomic distances of the test compound

Atom1	Atom2	Distance, Å°	Atom1	Atom2	Distance, Å°	Atom1	Atom2	Distance, Å°
1	2	3	4	5	6	7	8	9
K1	K5	1.677	–	F7	3.364	V2	F5	2.045
–	F1	1.830	–	K5	3.437	–	F7	2.534
–	F4	2.128	–	F1	3.479	–	F4	2.590
–	F3	2.210	–	F7	3.482	–	F1	2.827
–	K3	2.298	–	F3	2.899	–	K5	2.883
–	F7	2.536	–	F1	2.961	–	F3	2.969
–	F6	2.884	–	F5	3.028	–	F8	3.067
–	F8	2.972	–	F6	3.030	F4	F7	1.946
–	F5	3.326	–	F7	3.329	–	K1	2.128
–	F6	3.347	–	F4	3.400	–	F6	2.326
–	V3	3.424	–	F4	3.452	–	K2	2.390
K5	K1	1.677	–	K3	3.475	–	V2	2.590
–	F6	2.070	–	F5	3.497	–	K5	2.710



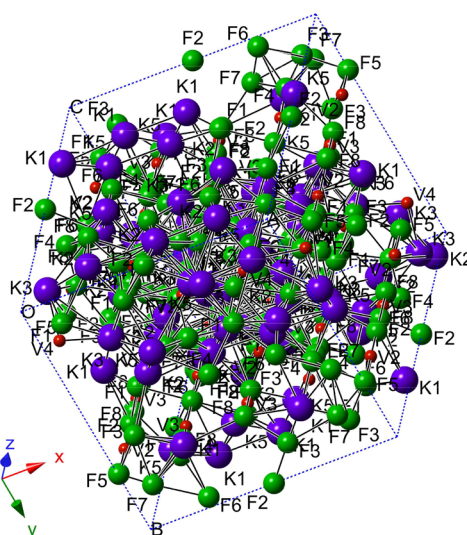
Continuation of Table 4

1	2	3	4	5	6	7	8	9
–	F8	2.462	V3	F8	1.073	–	K5	2.767
–	K3	2.617	–	K5	3.117	–	F2	2.819
–	F1	2.682	–	K1	3.424	–	F5	3.000
–	F2	2.687	V4	F5	2.187	–	F3	3.069
–	F4	2.710	–	K3	2.433	–	K3	3.400
–	F4	2.767	–	F7	2.744	–	K3	3.452
–	V2	2.883	–	F3	3.123	–	F6	3.482
–	K2	2.992	F5	F7	1.577	F6	K2	1.983
–	V3	3.117	–	V2	2.045	–	K5	2.070
–	F7	3.228	–	V4	2.187	–	K2	2.307
–	K2	3.437	–	F3	2.190	–	F1	2.311
F1	K1	1.830	–	F4	3.000	–	F4	2.326
–	F6	2.311	–	K3	3.028	–	F3	2.470
–	K2	2.380	–	K1	3.326	–	F7	2.528
–	F2	2.636	–	K3	3.497	–	K1	2.884
–	K5	2.682	F2	F1	2.636	–	K3	3.030
–	F3	2.738	–	K5	2.687	–	F2	3.146
–	V2	2.827	–	F4	2.819	–	K1	3.347
–	K3	2.961	–	F6	3.146	–	F4	3.482
–	F8	3.003	–	F8	3.252	F7	F5	1.577
–	F3	3.405	F3	F5	2.190	–	F4	1.946
–	K2	3.479	–	K1	2.210	–	K3	2.322
K2	F6	1.983	–	F6	2.470	–	F6	2.528
–	F6	2.307	–	F7	2.663	–	V2	2.534
–	F1	2.380	–	F7	2.732	–	K1	2.536
–	F4	2.390	–	F1	2.738	–	F3	2.663
–	K3	2.471	–	K2	2.868	–	F3	2.732
–	F3	2.868	–	K3	2.892	–	V4	2.744
–	K5	2.992	–	F3	2.899	–	F7	3.151
–	F3	2.998	–	F1	2.961	–	K5	3.228
–	F7	3.364	–	F5	3.028	–	K3	3.329
–	K5	3.437	–	F6	3.030	–	K2	3.364
–	F1	3.479	–	F7	3.329	–	K2	3.482
–	F7	3.482	–	F4	3.400	F8	V3	1.073
K3	K1	2.298	–	K3	2.899	–	F8	2.147
–	F7	2.322	–	V2	2.969	–	K5	2.462
–	V4	2.433	–	K2	2.998	–	K1	2.972
–	K2	2.471	–	F4	3.069	–	F1	3.003
–	K5	2.617	–	V4	3.123	–	V2	3.067
–	F3	2.892	–	F1	3.405	–	F2	3.252

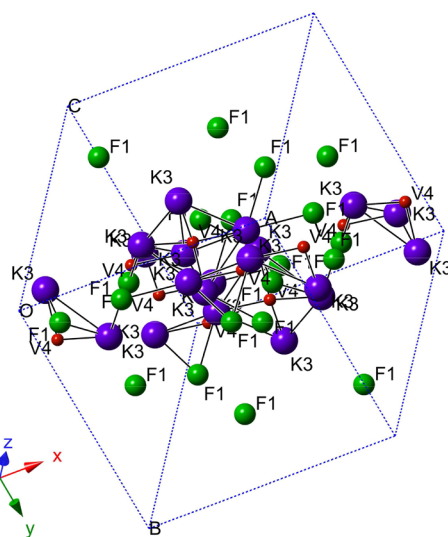


**Fig. 2.** The resulting diffraction patterns

**Fig. 2** shows the resulting diffractograms calculated from the proposed microstructural parameters of the K<sub>3</sub>VF<sub>6</sub> compound



**Fig. 3.** Crystal structure image of K<sub>3</sub>VF<sub>6</sub> compound



**Fig. 3** shows the crystal structure of the  $K_3VF_6$  tetragonal syngony of the space group I 41/a, belonging to its own type of structure. As can be seen from **Fig. 4**, the V atoms form triangular-shaped bonds with fluorine and potassium atoms.

#### 4. Discussion of results

As follows from the literature review, the test compound has a simple state diagram of the KCl-KF- $K_3VF_6$  system, which means that other compounds are not formed during the  $K_3VF_6$  synthesis. Various methods of synthesis of this compound make it possible to obtain the test compound in its pure form, and the diffraction patterns are completely consistent with the single-phase structure of this substance.

In addition, the literature indicates the existence of three polymorphic modifications of this compound [8], such as alpha, beta, and gamma phases.

The crystal structure of the  $\beta$  phase is studied in [13], and belongs to the  $[NH_4]_3FeF_6$  structural type.

The crystal structure of the  $\alpha$  phase is studied in this work and assigned to the intrinsic structural type of tetragonal syngony. So, at temperatures between 298 K and 453 K (for which the captured diffractograms [8] also exist), there is a polymorphic transformation from a tetragonal lattice to a cubic one.

The interatomic distances calculated for the proposed structural model of the  $K_3VF_6$   $\alpha$  phase indicate the reliability of the existence of this structural model.

From the fragment of the structure in **Fig. 4**, it follows that the vanadium atoms form a tri-cycle bond with the potassium and fluorine atoms.

#### 5. Conclusions

The X-ray phase analysis does not reveal the presence in the test compound of several phases, are presented in **Table 1**. So let's conclude that the connection is single-phase.

Using the HighScorePlus Treor program module, X-ray diffraction patterns of the  $K_3VF_6$  compound are indexed under the number 00-037-0738 in the PDF-2 database for 2004.

The diffraction spectrum of the compound is indexed in tetragonal system with lattice periods  $a=5.929$  Å;  $b=8.294$  Å;  $c=5.914$  Å. The space group of symmetry I 41/a is possible.

Using the HighScorePlus 3.0 program, the Rietveld method is used to study the diffraction spectrum of the compound under the number 00-037-0738. The structural model is proposed: the spatial symmetry group I 41/a (88), with lattice periods  $a=12.3524(4)$  Å;  $b=12.3524(4)$  Å;  $c=16.7447(7)$  Å.

The disagreement factor is  $R=9.88451$  %. The microstructural parameters are given in **Table 2**.

The crystal structure of this compound may belong to a new type of structure.

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