

AB Initio Structure Determination of $\text{Bi}_{0.5} \text{Mn}_{0.125} \text{Mo}_{0.5} \text{O}_{0.67} \text{U}_{0.32}$ Mixed Valency From Quartenary of Bi_2O_3 - V_2O_5 - UO_2 - Mn_2O_7 - MoO_4

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ABSTRACT

This paper is intended the ab initio structure determination of the heavy metal framework mixed valence $\text{Bi}_{0.5} \text{Mn}_{0.125} \text{Mo}_{0.5} \text{O}_{0.67} \text{U}_{0.32}$ from precession electron diffraction intensities. The metal framework of the compound was solved in this investigation via direct methods from Powder XRD. A subsequent (kinematical) least-squares refinement with electron intensities yielded slightly improved co-ordinates for the 6 heavy atoms in the structure. Chemical analysis of several crystallites by EDX is in agreement with the formula $\text{Bi}_{0.5} \text{Mn}_{0.125} \text{Mo}_{0.5} \text{O}_{0.67} \text{U}_{0.32}$. Moreover, the structure was independently determined by Rietveld refinement from X-ray powder data obtained from a multi-phasic sample. The compound having triclinic crystal system space group $P-1$ and centrosymmetry structure with refined lattice parameters $a=5.8655, b=4.4099, c=17.6031, \alpha=82.0712, \beta=88.251$ and $\gamma=74.4651$. Comparison of the framework structure from electron diffraction with the result from Rietveld refinement shows an average agreement for the heavy atoms within 0.09 \AA . The titled compound was prepared from mixture of Bi_2O_3 - V_2O_5 - UO_2 - Mn_2O_7 - MoO_4 by solid state reaction with full thermal decomposition at 1000°C . $R_{wp} = 0.0680, R_p = 0.030$ and $\text{GOF} = 0.31$ and the structure factors $F_0 = 3023$ and $F_c = 3021$.

KEY-WORDS:- *Multiphase, mixture, heavy, centrosymmetry, structure, XRD, frame work, Refinement*

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I. INTRODUCTION

The fields of solid-state chemistry and materials science are searching for and discovering new functional materials. In materials chemistry, the crystal structure determination is the first step to understand and interpret physical properties of an unknown material. Moreover, it also guides people on how to modify the material and hence improve the performance. Nowadays, the most successful technique for structure determination is through single crystal X-ray diffraction, from which a sufficient number of independent reflections against the structural parameters can be extracted in 3D reciprocal space. Several mature analysis methods, such as the direct method,¹ Patterson method,² charge-flipping algorithm³ and maximum entropy method⁴ can be applied to accurately solve the structure. This technique requires synthesizing a high quality single crystal at a micrometre level, which might be difficult in some fields, for example in ceramic chemistry [1]. Experimentally, the chance to get polycrystalline materials is generally larger than to get single crystals. In this case, powder X-ray diffraction (PXRD) becomes a popular technique but with this technique, the possibility to determine an unknown structure dramatically decreases, because 3D reflections are compressed into 1D with an inevitable overlapping problem, especially when the unit cell is big. The situation will become worse when the PXRD is collected on a multi-phase sample, which is not uncommon in the preliminary stage of searching new materials, especially in the cases of hydrothermal (or solvothermal) syntheses of zeolitic or MOF materials [2]. The difficulty could further increase when the target phase is not the highly dominant one in the composition. To determine the structure of an unknown phase in a multi-phase polycrystalline sample would be very helpful for the researchers, saving time to optimize the syntheses conditions. Our understanding of the properties of materials is almost always based on structural information on the atomic scale. Such information is commonly obtained by the wide spread method of X-ray crystallography. However, due to the relatively weak interaction of matter with X-rays, this method is insufficient to investigate extremely small volumes or individual crystals at the scale. Materials with pronounced twinning or new compounds that only exist as part of a complex multi-phase powder sample are thus extremely difficult to treat with this standard method for structure determination [3]. It should be noted that these problem cases also include many technologically relevant products such as small precipitates in a metallic matrix, catalysts, pharmaceuticals, pigments and thin films, which a priori exist only in small quantities or rarely grow as large crystals. Hence, ample motivation exists to develop alternative approaches capable for structural analysis of extremely small volumes and

crystallites. However, the only real alternative to X-rays is fast electrons, since their interaction with matter is several orders of magnitude stronger than that of X-rays. Electron diffraction structure analysis (EDSA) makes it, thus, possible to obtain structural information at the atomic level even for the steadily growing number of nanocrystalline materials[4-7]. On the other hand, structure analysis with electron data is rarely straightforward and fully automated, as it is the case with X-ray data. because now-a-days powder X-ray diffraction has been routinely used a non-destructive fingerprinting technique. It has also been used in studies related to structural phase transitions at variable temperature and pressure. The aim of this work is determine structure of titled compound by ab initio method with the help of powder XRD

II. MATERIAL AND METHOD

All chemicals used were analytical grade. A polycrystalline sample of Bi_{0.5} Mn_{0.125} Mo_{0.5} O_{0.67} U_{0.3}. was synthesized by a standard solid state reaction using a mixture of high purity reagents of Bi₂O₃-V₂O₅ -UO₂- Mn₂ O₂- MoO₄ contained mixed valence as the starting materials in the molar ratio of 1 : 1 : 1. The mixture was ground carefully, homogenized thoroughly with methanol (99%) in an agate mortar and then packed into an alumina crucible and calcined at 1000°C in air for 30h with several intermediate grindings. Finally the product was pressed into pallets and sintered at 100 K/h. Powder X-ray diffraction (XRD) data were collected at room temperature in the angular range of 2 θ =10 to 90 with scan step width of 0.02° and a fixed containing time of 15 s using Philips powder diffractometer with graphite monochromatic CuK α radiation. The powder was rotated during the data collection to minimize preferred Orientation effect if any. The program TREOR in CRYSFIRE was used to index the powder pattern which give orthorhombic cell system.SIRPOW92 was used to locate the positional parameters of constituent atoms. The full pattern is fitting and peak decomposition in the space group P-1 using check cell program. The structural parameters were refined by the Reitveld method using the GSAS program which gave at 1000°C. Rwp = 0.0680, Rp = 0.030 and GOF=0.31 the structure factors F0 = 3023 and Fc = 3021. The density is determined by Archimedes principle.

III. RESULTS AND DISCUSSIONS

In materials chemistry, the crystal structure determination is the first step to understand and interpret physical properties of an unknown material. Moreover, it also guides people on how to modify the material and hence improve the performance[4-8]. Nowadays, the most successful technique for structure determination is through single crystal X-ray diffraction, from which a sufficient number of independent reflections against the structural parameters can be extracted in 3D reciprocal space. Several mature analysis methods, such as the direct method,1 Patterson method,2 charge-flipping algorithm3 and maximum entropy method4 can be applied to accurately solve the structure. This technique requires synthesizing a high quality single crystal at a micrometer. Experimentally, the chance to get polycrystalline materials is generally larger than to get single crystals. In this case, powder X-ray diffraction (PXRD) becomes a popular technique but with this technique, the possibility to determine an unknown structure dramatically decreases, because 3D reflections are compressed into 1D with an inevitable overlapping problem, especially when the unit cell is big. The situation will become worse when the PXRD is collected on a multi-phase sample, which is not uncommon in the preliminary stage of searching new materials, especially in the cases of hydrothermal (or solvothermal) syntheses of zeolitic or MOF materials'XRD data for the sample of **Bi_{0.5} Mn_{0.125} Mo_{0.5} O_{0.67} U_{0.32}** synthesized at its nominal composition is shown in Figure 1. All peaks could be indexed to a triclinic unit cell with *P-1* symmetry, analogous to the **Bi_{0.5} Mn_{0.125} Mo_{0.5} O_{0.67} U_{0.32}** structure using CRYSFIRE PACKAGE[10]. In the diffraction pattern there is a slight anisotropic peak broadening moving to higher angles. This slight peak broadening could be caused by ordering within the cationic layers but relative disorder of the cations between layers. The sloping background observed in XRD at low angles out to ~30° 2 θ is qualitatively indicative of stacking faults, a common feature of honeycomb oxides. In an ideal layered mixed-metal oxide, all the cationic planes are stacked along the c-axis by a unique translational stacking vector. However it has been shown that two other stacking vectors with very similar energy (1-2 meV) can occur in a structure, leading to so called "stacking faults". Stacking faults in the honeycomb oxides occur due to the relatively weak coupling between the layers In practice, the cationic layers are never perfectly stacked along the c-axis when a layered structure has the *P-1* space group, although higher temperature thermal treatment during synthesis can decrease the frequency of stacking faults[11].

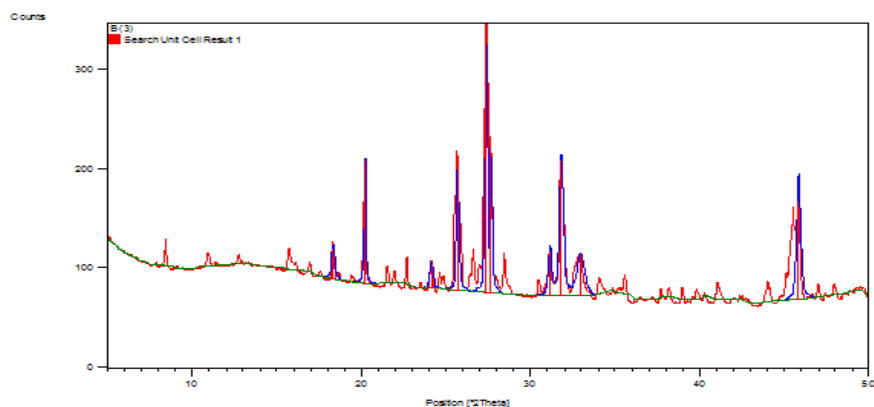


Figure 1. Powder XRD graphical spectra of $\text{Bi}_{0.5}\text{Mn}_{0.125}\text{Mo}_{0.5}\text{O}_{0.67}\text{U}_{0.32}$

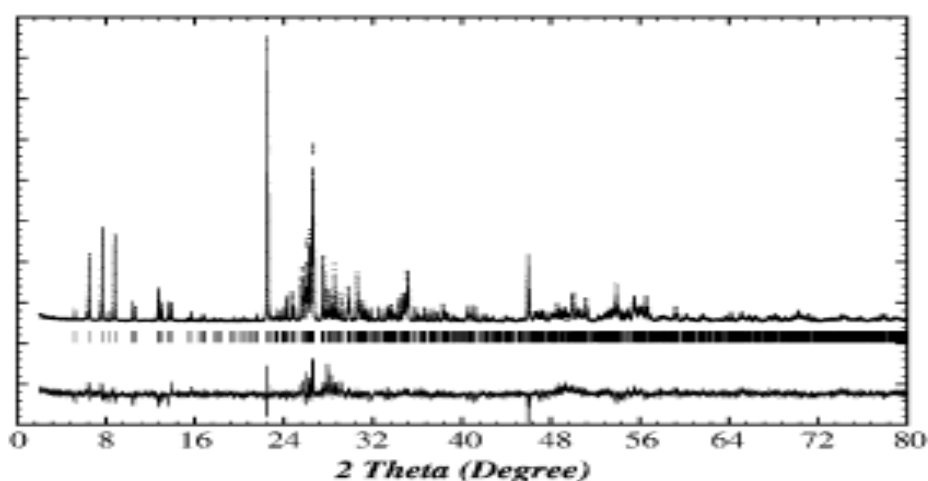


Figure 2. Graphical representation of the result from Rietveld refinement with X-ray powder data. Vertical bars indicate positions of the Bragg reflections for $\text{Bi}_{0.5}\text{Mn}_{0.125}\text{Mo}_{0.5}\text{O}_{0.67}\text{U}_{0.32}$. Dots mark the observed intensities and the solid line gives the calculated intensity curve. The deviations between the observed and the calculated intensities from the refined model are shown by the difference plot in the lower part of the diagram.

STRUCTURE SOLUTIONS

X-Ray powder diffraction and Rietveld refinements: X-Ray powder diffraction (XRD) patterns were collected on a PANalytical X'Pert-MPD diffractometer using graphite-monochromated $\text{Cu-K}\alpha$ radiation. The data were collected at 40 kV, 30 mA with a step size of 0.02° in the 2θ range of $5-80^\circ$. Rietveld refinements of the XRD patterns were performed using GSAS-II. The backgrounds were fit with Chebyshev function, and Bragg-Brentano was selected as diffractometer type. Lattice constants, instrument parameters, atomic coordinates, thermal parameters, and peak profile functions were refined in order to achieve a simulated diffraction pattern that matched the experimental data. The completed refinements provided phase quantification and lattice parameter determination. The observed, difference and calculated patterns of the newly synthesized novel oxide obtained by Rietveld refinement has been shown in Fig. 1. The structure in packing form shown in Figure 2. In the structure Bi is bonded with Mn, Mo bonded with U-O, Bi with O each forming closed type structure. The bonding between different constituent atoms has been discussed in more details in table 2. In conclusion, the structure of $\text{Bi}_{0.5}\text{Mn}_{0.125}\text{Mo}_{0.5}\text{O}_{0.67}\text{U}_{0.3}$ has been solved by the *ab initio* approach using powder X-ray diffraction data. $\text{Bi}_{0.5}\text{Mn}_{0.125}\text{Mo}_{0.5}\text{O}_{0.67}\text{U}_{0.3}$ was found to crystalline in orthorhombic crystal system with space group P-1 which displays an unusual spiral chain structure along the c-axis and packing form on three-dimensional axis. The data were analysed with Diamond package with help of CIF file. Rietveld refinement of triclinic crystal system $\text{Bi}_{0.5}\text{Mn}_{0.125}\text{Mo}_{0.5}\text{O}_{0.67}\text{U}_{0.3125}$ against XRD data for structural determination proved difficult, due to a combination of preferred orientation of the plate-like crystallites in flat-plate geometry [13]. In other words, the extracted peak intensities for phase 1 could be reliable. Note that if the relative intensities differed a lot from the simulated patterns of the known phases, additional manual partitioning of those overlapping peaks would be necessary to get reliable intensities for the unknown phase 1. After considering the multiplicity and Lorentz-polarization correction, the intensities of phase 1 were used for the structure solution as below.

Moreover, the reflection conditions indicated possible space groups $Pcnb$. The initial structure model was obtained using a charge flipping algorithm with the program Superflip.8 from Jana package [12-13]. Random phases were used at the beginning of the charge-flipping iteration, and overlapping peaks were re-partitioned using a histogram match to improve the convergence. The iteration converged with an R factor of 29% and the final electron density shows a P-1 symmetry with a 5% error. The program of EDMA was then used to automatically assign atomic positions. Four unique heavy atomic positions were found and the heaviest one was assigned as Bi while the others were considered as Bi and U. Due to the existence of heavy atoms, all oxygen positions were ambiguous in the electron density map of this stage [14,15]. To locate the oxygen atoms, a Monte-Carlo based simulated annealing process with the program TOPAS was applied. For each annealing process, various atomic coordinates were randomly assigned as the initial positions of the oxygen atoms [15]. The annealing process was restarted after finding a few oxygen positions, until all oxygen positions were found to be reasonable.

Crystallographic data after refinement

Phase data	
Formula sum	$\text{Bi}_{0.5}\text{Mn}_{0.125}\text{Mo}_{0.5}\text{O}_{0.67}\text{U}_{0.32}$
Formula weight	246.2 g/mol
Crystal system	triclinic
Space-group	P -1 (2)
Cell parameters Index	$a=5.8655 \text{ \AA}$ $b=8.4099 \text{ \AA}$ $c=17.6031 \text{ \AA}$ $\alpha=82.0712^\circ$ $\beta=88.2518^\circ$ $\gamma=74.4636^\circ$ $0 \leq h \leq 2, 0 \leq k \leq 3, -1 \leq l \leq 0$
Cell ratio	$a/b=0.6975$ $b/c=0.4778$ $c/a=3.0011$
Cell volume	828.59 \AA^3
Z	2
Calc. density	0.986743 g/cm^3
Meas. density	0.867
Pearson code	aP56
Formula type	$\text{N}_2\text{O}_2\text{P}_5\text{Q}_6\text{R}_{13}$
Wyckoff sequence	i28

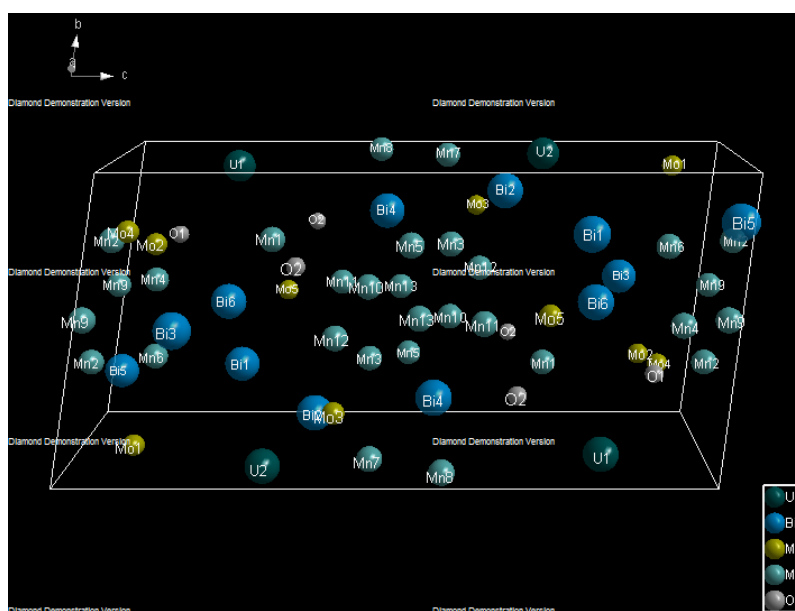


Figure 4. Representation of the powellite-type crystal structures of $\text{Bi}_{1.2}\text{Mn}_{2.5}\text{Mo}_{0.21}\text{U}_{2.516}\text{O}_{0.67}$.

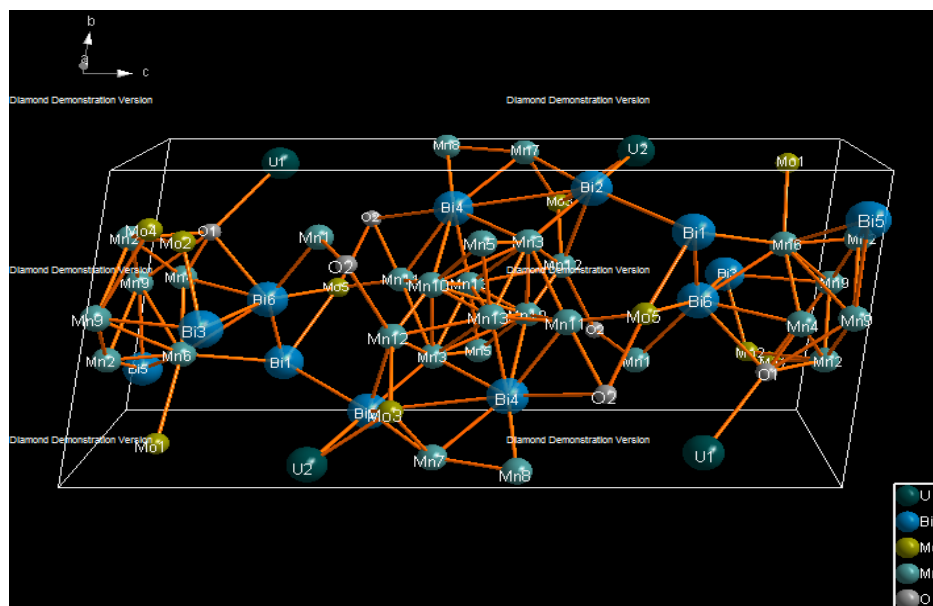


Figure 3. Three dimension polyhedral structure of $\text{Bi}_{1.2}\text{Mn}_{2.5}\text{Mo}_{0.21}\text{U}_{2.516}\text{O}_{0.67}$.

TABLE 1.
Atomic Coordinate

Atom	Ox.	Wyck.	Site	S.O.F.	x/a	y/b	z/c	U [\AA^2]
U1	+4	2i	1		0.86850	0.92384	0.17673	0.0380
U2	+4	2i	1		0.16943	0.02727	0.30740	0.0380
Bi1	+3	2i	1		0.77510	0.22684	0.23207	0.0380
Bi2	+3	2i	1		0.50431	0.11850	0.37059	0.0380
Bi3	+3	2i	1		0.07887	0.48531	0.14433	0.0380
Bi4	+3	2i	1		0.66554	0.78970	0.44176	0.0380
Bi5	+3	2i	1		0.04639	0.83759	-0.01822	0.0380
Bi6	+3	2i	1		0.58731	0.48514	0.20254	0.0380
Mo1	+4	2i	1		0.66337	0.94810	-0.09498	0.0380
Mo2	+4	2i	1		0.15332	0.75162	0.09777	0.0380
Mo3	+4	2i	1		0.04801	0.22914	0.40690	0.0380
Mo4	+4	2i	1		0.07331	0.80441	0.05725	0.0380
Mo5	+4	2i	1		0.97849	0.45457	0.28698	0.0380
Mn1	+4	2i	1		0.38322	0.73194	0.26703	0.0380
Mn2	+4	2i	1		0.45336	0.71701	0.00624	0.0380
Mn3	+4	2i	1		0.58127	0.28961	0.44694	0.0380
Mn4	+4	2i	1		0.75862	0.53070	0.06853	0.0380
Mn5	+4	2i	1		0.85050	0.25172	0.51035	0.0380
Mn6	+4	2i	1		0.43889	0.69926	-0.09601	0.0380
Mn7	+4	2i	1		0.28486	0.01907	0.47016	0.0380
Mn8	+4	2i	1		0.86192	0.98498	0.41615	0.0380
Mn9	+4	2i	1		0.79956	0.50475	0.00320	0.0380
Mn10	+4	2i	1		0.59870	0.43134	0.56950	0.0380
Mn11	+4	2i	1		0.82020	0.51249	0.38114	0.0380
Mn12	+4	2i	1		0.26547	0.42298	0.39025	0.0380
Mn13	+4	2i	1		0.72986	0.51311	0.48199	0.0380
O1	-2	2i	1		0.70333	0.70120	0.10113	0.0380
O2	-2	2i	1		0.99290	0.70569	0.32089	0.0380

TABLE2. SELECTED BOND ANGLES

Atom 1	Atom Code	Atom2	Atom code	Bond angle
Mo1	Mn5	11555011	Mn1 7555011	80.540
Mn5	11555011	Mn9	15666021	135.819

	Mn1	7555011	Mn9	15666021	130.670
Mo2	U2	9555011	O6	24555011	69.154
	U	29555011	O3	21555011	137.232
	U	29555011	O8	26666021	25.618
	O6	24555011	O3	21555011	114.957
	O6	24555011	O8	26666021	59.165
	O3	21555011	O8	26666021	116.563
Mo3	O8	26555011	Mn7	13655011	156.870
Mo4	Mn11	17555011	Mn10	16555011	29.089
	Mn11	17555011	O4	22755021	142.514
	Mn8	14555011	O3	21666021	171.871
	Mo6 U	30756021	O2	20645011	64.498
	U	30756021	O4	22545011	93.848
	U	30756021	Mn6	12545011	115.453
	O2	20645011	O4	22545011	71.837
	O2	20645011	Mn6	12545011	57.898
	O1	19645011	Mn6	12545011	137.682
	O3	21756021	Mn6	12545011	100.243
	O7	25555011	Mo1	1555011	162.547
	O4	22555011	O2	20655011	80.116
	O4	22555011	Mn12	18555011	82.783
	O4	22555011	Mo6	6565011	64.755
	Mn10	16555011	O2	20655011	73.309
	O2	20655011	Mo6	6565011	52.054
	Mn12	18555011	Mo6	6565011	76.471
	Mn4	10555011	O2	20555011	83.359
	Mn4	10555011	Mo3	3455011	164.688

	O2	20555011	Mo3	3455011	82.392
	O4	22555011	Mo4	4555011	155.192
	U	30666021	Mn11	17455011	144.665
	O5	23555011	Mo6	6465011	80.361
	Mn7	13555011	Mn6	12455011	129.981
	Mn7	13555011	O3	21666021	88.159
	Mn7	13555011	Mn11	17455011	70.502
	Mn6	12455011	O3	21666021	111.168
	Mn6	12455011	Mn11	17455011	67.934
	O3	21666021	Mn11	17455011	144.897
O3	Mo2	2555011	O2	20666021	130.805
	Mo2	2555011	U	30555011	108.725

IV. CONCLUSION

The determination of crystal structure from powder X-ray diffraction data appears to hold promise for the future especially in materials science and drugs and pharmaceuticals fields where the growth the o diffraction quality crystals becomes a serious rate limiting step. $Bi_{1.2}Mn_{2.5}Mo_{0.21}U_{2.5.16}O_{0.67}$.has been successfully synthesized by high temperature solid state reaction in open air atmosphere for the first time.. Its structure features an intricate 3D framework composed of 1D neodymium oxygen chains connected with Cd cations located in the internal space. The phase purity was verified by XRD measurement coupled with Rietveld refinement. Preliminary structural work on the new $Bi_{0.5}Mn_{0.125}Mo_{0.5}O_{0.67}U_{0.3}$.phase leads to the triclinic system, space group $P-1$ with unit-cell parameters Unit cell dimensions $a=5.8655 \text{ \AA}$ $b=8.4099 \text{ \AA}$ $c=17.6031 \text{ \AA}$ $\alpha=82.0712^\circ$ $\beta=88.2518^\circ$ $\gamma=74.4636^\circ$ indexes $0 \leq h \leq 2, 0 \leq k \leq 3, -1 \leq l \leq 0$, and experimental density by Archimedes principle is 1.495 g/cm^3 Density, calculated 1.545 g/cm^3 . In summary, this work shows how the substitution of Bi for Mn and Mo for U in columnar structural-type oxides is progressively carried out with a simplification of the structure[16].

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