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Phase study in the Na–Sr–U–O system: Characterization of new phase

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ABSTRACT

In continuation with the earlier work for phase studies of Rb–Sr–U–O and Cs–Sr–U–O systems, the sub-solidus phase relations in Na–Sr–U–O quaternary system were determined at 850 °C in air atmosphere. A novel quaternary phase $Na_8Sr_2U_6O_{24}$ in the Na–Sr–U–O system was synthesized by heating the respective oxides at 850 °C in air. XRD data of $Na_8Sr_2U_6O_{24}$ was indexed on a cubic system with lattice parameter $a = 0.8326$ nm and was found isostructural with $K_8Sr_2U_6O_{24}$ and $Rb_8Sr_2U_6O_{24}$. A pseudo-ternary phase diagram of Na_2O–SrO–UO_3 was drawn using the new quaternary compound and various phase fields were established by X-ray powder diffraction analysis. The structure of $Na_8Sr_2U_6O_{24}$ was derived from the powder data and structural parameters were refined by the Rietveld profile method.

1. Introduction

The knowledge of the interaction of fuel-fission products is important in understanding the behaviour of the fuel during the operation of nuclear reactors. The operation of defective uranium–plutonium oxide pins in sodium cooled fast breeder reactors i.e. liquid metal fast breeder reactor (LMFBR) results in chemical reactions between the fuel-coolant and fuel-fission products. After pin failure, the oxide fuel along with high yield fission products such as strontium, molybdenum and cesium comes in contact with sodium coolant. Thus UO_2 fuel matrix can interact with sodium or strontium and other fission products to form ternary and polynary uranium compounds which may affect the fuel behaviour. The knowledge of phase equilibria and other thermodynamic properties of the alkali and alkaline earth metals are necessary for the advancement of the technology of nuclear energy production.

Alkali metal–uranium–oxygen and alkaline earth metal–uranium–oxygen systems have been investigated extensively, establishing the existence of several uranates and polyuranates depending on conditions like metal to uranium ratio, oxygen potential, temperature of reaction etc. [1–7]. The phase equilibria of alkali metal oxides and their interaction with other oxides relevant to nuclear fuels, fission products and structural materials have been considered in detail in a review by Lindemer et al. [8]. The high temperature reactions of uranium oxides in molten salts and in the solid state, to form alkali metal uranates, and their composition and properties have been reviewed by Griffith and Volkovich [9].

As no work was reported on alkali metal–alkaline earth metal–uranium–oxygen quaternary system, we have carried out the phase studies of the Rb–Sr–U–O and Cs–Sr–U–O systems in air at 900 °C [10,11]. Two novel quaternary phases $Rb_2Sr_2U_4O_{15}$ and $Rb_8Sr_2U_6O_{24}$ were prepared and characterized in Rb–Sr–U–O system, where as a single phase $Cs_2Sr_2U_4O_{15}$ was established in Cs–Sr–U–O system. Pseudo ternary phase diagrams of Rb_2O–SrO–UO_3 and Cs_2O–SrO–UO_3 were drawn using new quaternary compounds and various phase fields characterized by X-ray powder diffraction were reported. The preparation and structure analysis of $K_8A_2U_6O_{24}$ compounds (A = Ca, Sr and Ba) by X-ray powder diffraction method have been reported earlier [12].

In continuation with earlier work, present study is carried out on Na–Sr–U–O quaternary system to establish the phase relationship in the system as a prelude to investigations of a series of alkali metal–alkaline earth metal–uranium–oxygen system. The investigations were carried out by preparing various phases of different compositions in the Na–Sr–U–O system by solid state reaction route and characterizing them by the X-ray powder diffraction (XRD) method.

2. Experimental

2.1. Preparation of samples

$NaNO_3$ and $SrCO_3$ (both analar grade, Merck, 99.99%) and UO_3 were used as starting materials for the preparation of different phase mixtures in Na–Sr–U–O system. UO_3 was prepared by decomposing ammonium diuranate (ADU) at 350 °C. Ammonium diuranate was precipitated by reacting uranyl nitrate (nuclear pure) solution with ammonia. The formation of β-UO_3 was confirmed by comparing the XRD data with those reported in the literature [13]. As $NaNO_3$ and $SrCO_3$ are hygroscopic, the compounds were dried at 200 °C for 12–14 h and weighed immediately. The three compounds were then mixed in different molar proportions of UO_3, Na_2O and SrO for making fifteen equilibrium mixtures, listed in

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Table 1
Phase identification of various compounds in the Na_2O–SrO–UO_3

Mixture number	Mole fraction of the oxides				Phase identified by XRD	
	Na_2O	SrO	UO_3			
A	0.33	0.17	0.50	$Na_8Sr_2U_6O_{24}$[a]		
M1	0.20	0.70	0.10	$SrCO_3$	Sr_3UO_6	Na_2O
M2	0.15	0.60	0.25	A	Sr_3UO_6	Na_2O
M3	0.10	0.50	0.40	A	Sr_2UO_5	$Sr_3U_2O_9$
M4	0.05	0.50	0.45	A	$Sr_3U_2O_9$	$SrUO_4$
M5	0.10	0.30	0.60	A	SrU_4O_{13}	$SrUO_4$
M6	0.10	0.10	0.80	A	SrU_4O_{13}	UO_3
M7	0.25	0.05	0.70	A	$Na_6U_7O_{24}$	UO_3
M8	0.31	0.04	0.65	A	$Na_2U_2O_7$	$Na_6U_7O_{24}$
M9	0.40	0.05	0.55	A	$Na_2U_2O_7$	Na_2UO_4
M10	0.50	0.05	0.45	A	Na_2UO_4	Na_4UO_5
M11	0.65	0.05	0.30	A	Na_4UO_5	Na_2O
M12	0.40	0.30	0.30	A	Sr_3UO_6	Na_2O
M13	0.03	0.67	0.30	A	Sr_3UO_6	Sr_2UO_5
M14	0.10	0.02	0.88	A	$Na_6U_7O_{24}$	UO_3
M15	0.33	0.12	0.55	A	$Na_2U_2O_7$	$Na_6U_7O_{24}$

[a] The new phase = A.

Table 2
X-ray diffraction data of $Na_8Sr_2U_6O_{24}$ (λ = 0.15418 nm) a = 0.8326 (10) nm

h	k	l	$d_{(obs)}$ (nm)	$d_{(cal)}$ (nm)	I/I_0
1	1	0	0.5950	0.5910	45
1	1	1	0.4818	0.4824	08
2	0	0	0.4182	0.4179	100
2	1	1	0.3419	0.3412	16
2	2	0	0.2953	0.2954	93
3	1	0	0.2641	0.2642	08
2	2	2	0.2424	0.2413	06
3	2	1	0.2233	0.2233	05
4	0	0	0.2084	0.2089	26
4	2	0	0.1866	0.1868	17
4	2	2	0.1709	0.1706	14
5	1	0	0.1639	0.1639	05
4	4	0	0.1477	0.1477	09
6	0	0	0.1394	0.1393	10
6	2	0	0.1319	0.1320	08

Table 1. The reaction mixtures were heated in a furnace in alumina boat initially at 600 °C for 4 h, reground intermittently and then further heated at 850 °C for 30 h. followed by air quenching.

2.2. Instrumental analysis

XRD data of all the heated products were recorded on DIANO X-ray diffractometer using Cu Kα radiation (λ = 0.15418 nm) at the rate of 1° (2θ) per minute to analyse the formation of various phases. The phases identified in the samples are listed in Table 1 along with their compositions. For the structure derivation of the pure phase, intensities for the diffraction peaks were recorded in the range of 13–100° (2θ) with step size of 0.02° (2θ) and a counting time of 5 s for each step.

3. Results and discussion

3.1. Phase diagram studies

The three pseudo-binary systems, Na_2O–UO_3, SrO–UO_3 and Na_2O–SrO were used for establishing the pseudo-ternary phase diagram of the Na_2O–SrO–UO_3 system. The Na_2O–UO_3 and SrO–UO_3 systems have been already investigated.

Na_2O–SrO: No ternary compound is reported in this boundary system.

Na_2O–UO_3: This pseudo-binary system has six reported compounds [1,7,14], $Na_2U_{13}O_{40}$, $Na_2U_7O_{22}$, $Na_6U_7O_{24}$, $Na_2U_2O_7$, Na_2UO_4 and Na_4UO_5 which are made up of Na_2O and UO_3 in the ratios, $(Na_2O)(UO_3)_{13}$, $(Na_2O)(UO_3)_7$, $(Na_2O)_3(UO_3)_7$, $(Na_2O)(UO_3)_2$, $(Na_2O)(UO_3)$ and $(Na_2O)_2(UO_3)$, respectively. In an extensive review of the literature of the high temperature reactions of alkali metal salts with uranium oxides in molten and in the solid state to form alkali metal uranates, Griffiths and Volkovich [9] have stated that formation of polyuranates, with higher uranium content than diuranate was limited to the formation of mixtures of $U_3O_8 + Na_6U_7O_{24}$. Carnall et al. have [15] investigated sodium polyuranates and reported that polyuranates $Na_2O \cdot xUO_3$ with x ranging between 2.1 and 2.5 have a structure close to that of $Na_2U_2O_7$ whereas x ranging between 6 and 13 have not been well characterized and their structure were said to be related to UO_3 or U_3O_8. Cordfunke and Loopstra [7] have reported that though sodium polyuranates with atomic ratio Na/U < 1 have been claimed to exist, in most cases these are ill identified and when heated the products were identical to $Na_2U_2O_7$. In their investigation, they found only one distinct phase $Na_6U_7O_{24}$ (Na/U = 0.857) whose XRD is almost identical to that $Na_2U_2O_7$. When $Na_6U_7O_{24}$ was heated in air above 950 °C, it decomposed into a mixture of $Na_2U_2O_7$ and U_3O_8 [7]. In order to confirm this observation, $Na_6U_7O_{24}$ was prepared by heating the mixture of UO_3 and $NaNO_3$ in 6:7 molar proportion at 850 °C for 30 h in an air. The formation of the compound was confirmed by XRD data which was identical to that of the reported one [7]. On heating this compound in air at 950 °C for about 10–12 h, it decomposed to give $Na_2U_2O_7$ and U_3O_8. However, at 850 °C, it was found to be stable in air revealed from the XRD. To confirm the formation of other stable phases in Na_2O–UO_3 pseudo binary system, the compounds of $Na_2U_2O_7$, Na_2UO_4 and Na_4UO_5 were also prepared by mixing UO_3 and $NaNO_3$ in 1:1, 2:1 and 4:1 molar proportions, respectively and heating these mixtures at 850 °C in air for 30 h. The XRD patterns confirm the formation of the desired phases. As discussed above $Na_2U_{13}O_{40}$ and $Na_2U_7O_{22}$ with Na/U = 0.154 and

Table 3
X-ray powder diffraction data of the reported compounds in Na_2O–UO_3 and SrO–UO_3 systems, used in this work

Compound	d-values in (nm)										Reference
Na_4UO_5	0.536_{10}	0.396_9	0.274_7	0.240_4	0.379_4	0.167_2	0.232_2	0.191_2	0.171_2	0.213_1	19
α-Na_2UO_4	0.495_{10}	0.286_8	0.489_4	0.284_4	0.283_4	0.287_3	0.202_3	0.350_2	0.247_2	0.220_2	7
β-Na_2UO_4	0.393_{10}	0.285_7	0.587_6	0.299_3	0.208_3	0.291_2	0.266_2	0.260_2	0.204_2	0.186_2	7
$Na_2U_2O_7$	0.597_{10}	0.318_5	0.334_4	0.198_3	0.270_2	0.298_2	0.339_2	0.321_2	0.187_2	0.247_2	20
Sr_3UO_6	0.303_{10}	0.298_3	0.502_2	0.428_2	0.309_2	0.214_2	0.177_2	0.173_2	0.256_2	0.214_2	6
Sr_2UO_5	0.329_{10}	0.313_{10}	0.564_8	0.505_8	0.283_8	0.202_8	0.198_8	0.196_8	0.383_8	0.379_8	6
$Sr_3U_2O_9$	0.322_{10}	0.197_4	0.280_4	0.169_4	0.552_3	0.396_3	0.207_3	0.643_2	0.276_2	0.168_2	6
$SrUO_4$	0.327_{10}	0.323_{10}	0.285_8	0.198_7	0.550_6	0.399_5	0.270_5	0.407_4	0.253_4	0.203_3	6
SrU_4O_{13}	0.349_{10}	0.420_9	0.268_7	0.337_4	0.180_4	0.178_4	0.263_4	0.210_3	0.197_2	0.161_1	16
Na_2O	0.197_{10}	0.278_4	0.321_3	0.114_1	0.139_1	0.161_1	0.168_1	0.128_1	0.098_1	0.088_1	21
$SrCO_3$	0.354_{10}	0.345_7	0.205_5	0.246_4	0.191_4	0.248_3	0.245_3	0.183_3	0.199_3	0.255_2	22
UO_3	0.342_{10}	0.416_7	0.264_6	0.178_3	0.198_2	0.197_2	0.208_3	0.158_3	0.148_3	0.593_1	23

Ten strongest lines in descending order of intensity (given as subscripts).

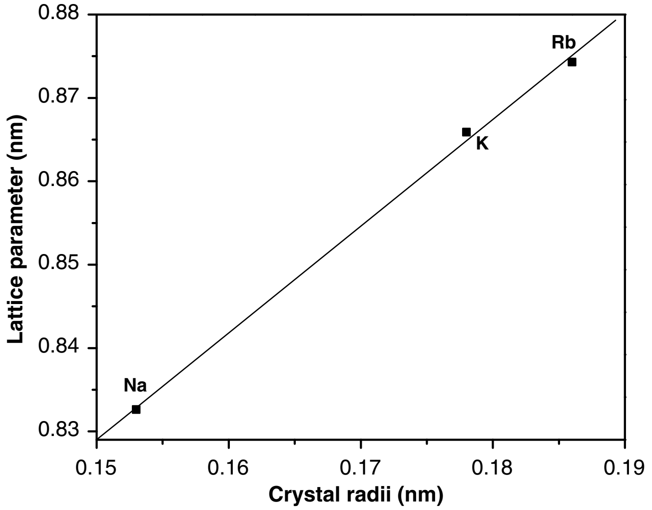

Fig. 1. The plot of ionic radii of Na, K and Rb vs. lattice parameters for $Na_8Sr_2U_6O_{24}$, $K_8Sr_2U_6O_{24}$ and $Rb_8Sr_2U_6O_{24}$.

0.286, respectively are not well characterized, XRD data is not reported in literature and thus were not included in the present work. The four well characterized compounds $Na_6U_7O_{24}$, $Na_2U_2O_7$, Na_2UO_4 and Na_4UO_5 were used for drawing the phase diagram.

SrO–UO₃: This limiting pseudo-binary system has six compounds reported by different workers, Sr_3UO_6, Sr_2UO_5, $Sr_3U_2O_9$, $SrUO_4$, $Sr_3U_{11}O_{36}$, SrU_4O_{13}, which are made up of SrO and UO_3 in the ratios, $(SrO)_3(UO_3)$, $(SrO)_2(UO_3)$, $(SrO)_3(UO_3)_2$, $(SrO)(UO_3)$, $(SrO)_3(UO_3)_{11}$ and $(SrO)(UO_3)_4$, respectively [6,16,17]. Though, some of the compounds such as SrU_2O_7 and $Sr_2U_3O_{11}$ are reported in the literature, but are still not well characterised. Hence these phases are not included in the present work for drawing the phase diagram. The *d*-values of the ten strongest lines in the decreasing order of intensity of the above mentioned ternary compounds in the $Na_2O–UO_3$ and $SrO–UO_3$ systems used for the identification of the phases prepared during the present work, are given in Table 3. The *d*-values of starting compounds UO_3, $SrCO_3$ and Na_2O are also included in Table 3.

There is no reported solubility of Na_2O, SrO and UO_3 in each other or in the compounds formed from them.

XRD data of novel quaternary phase $Na_8Sr_2U_6O_{24}$, was indexed on the cubic system with refined lattice parameter $a = 0.8326\,(10)$ nm, volume = 0.5772 nm^3 and X-ray density $\rho_{(cal)} = 5.89$ g cm^{-3}. X-ray density is in good agreement with the observed density, ($\rho_{(obs)} =$ 5.82 g cm^{-3}) which was obtained pyknometrically, using carbon tetra chloride as solvent. Indexed XRD data of the compound is given in Table 2. The similarity in the cell parameters of $Na_8Sr_2U_6O_{24}$ and $K_8Sr_2U_6O_{24}$ and $Rb_8Sr_2U_6O_{24}$, reported in earlier studies [10,12], suggests that the three compounds are isostructural. It was observed that as the ionic radii of the monovalent alkali metal ion increase, the lattice parameters of the compounds also increase. The least square plot of lattice parameters of $Na_8Sr_2U_6O_{24}$, $K_8Sr_2U_6O_{24}$ and $Rb_8Sr_2U_6O_{24}$ vs. ionic radii values of 6-coordinationed Na, K and Rb (0.153, 0.178 and 0.186 nm, respectively) [18] is shown in Fig. 1.

Many attempts were made to prepare another pseudo ternary compound, $Na_2Sr_2U_4O_{15}$, similar to the one reported in $Rb_2O–SrO–UO_3$ system. But, the compound was found to be a mixture of $Na_2U_2O_7$ and $SrUO_4$.

A pseudo-ternary phase diagram of $Na_2O–SrO–UO_3$ system shown in Fig. 2. was drawn on the basis of the phase analysis of the fifteen samples listed in Table 1 and using the XRD data of above mentioned compounds in the $Na_2O–UO_3$ and $SrO–UO_3$ systems and a new quaternary compound $Na_8Sr_2U_6O_{24}$ [19–23].

3.2. Structural studies

The structure of $Na_8Sr_2U_6O_{24}$ was derived from space group Im3m (no. 229) by using the GSAS software [24] with the EXPGUI [25] interface to perform Rietveld analysis of the X-ray powder

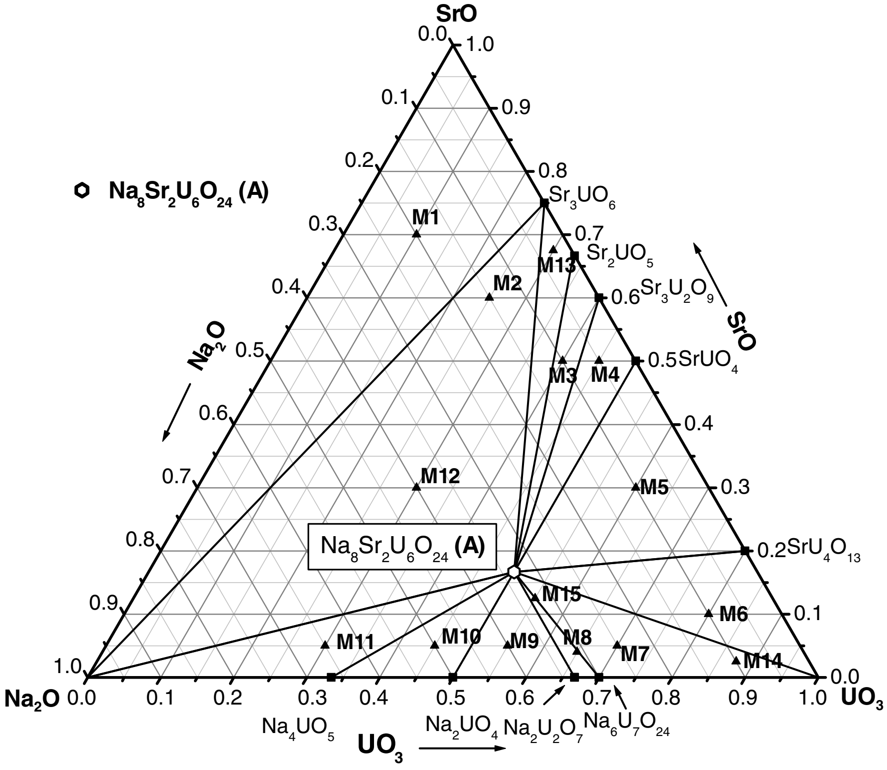

Fig. 2. Isothermal section of the pseudo-ternary phase diagram of the $Na_2O–SrO–UO_3$ system at 850 °C.

Table 4
Rietveld refinement details for $Na_8Sr_2U_6O_{24}$

Radiation	Cu Kα λ 0.1 5418 (nm)
Space group	Im3m
a (nm)	0.8326 (10)
V (nm^3)	0.5772(1)
Dcalc ($g\,cm^{-3}$)	5.89
Z	1
Rp (%)	7.54
Rwp (%)	10.96
$R^{*}F^{2}$	7.75
χ^2	1.03
No. of variables	24

$R_p = 100 \times \Sigma|\, y_{obs} - y_{cal}|/\Sigma\, y_{obs}|$.
$R_{wp} = 100 \times \{[\Sigma w\,(y_{obs} - y_{cal})^2]/\,[\Sigma w\, y_{obs})^2]\}^{1/2}$.

Table 5
Atomic parameters and interatomic distances in $Na_8Sr_2U_6O_{24}$ values in the parentheses correspond to estimated standard deviation

Atom	Site	*x*	*y*	*z*	B_{iso} (nm^2)
Interatomic distances (nm)					
U	6b	0.0	0.5	0.5	0.004667
Sr	2a	0.0	0.0	0.0	0.078022
Na	8c	0.25	0.25	0.25	0.002064
O1	12e	0.287(17)	0.0	0.0	0.034408
O2	12d	0.25	0.0	0.5	0.001919
U–O1	0.1770 (1)				
U–O2	0.2084 (2)				
Sr–O1	0.2393 (1)				
Na–O1	0.2944 (2)				
Na–O2	0.2960 (1)				

data, based on the atomic parameters of isostructural $Rb_8Sr_2U_6O_{24}$ [10]. The structural parameters of $Na_8Sr_2U_6O_{24}$ (atomic parameter for all the atoms except *x* of O1) and overall thermal parameters of all atoms were refined, modeling individual diffraction profiles on pseudo-Voigt function. The refinement of the appropriate atomic parameters and profile parameters resulted in agreement factors which are given in Table 4. The derived atomic parameters of $Na_8Sr_2U_6O_{24}$ and the relevant bond lengths between different atoms are listed in Table 5. The observed and calculated diffraction patterns of $Na_8Sr_2U_6O_{24}$ and their difference pattern are shown in Fig. 3. The indices of the intense lines are shown in the figure, pointing to their positions.

A structure of $Na_8Sr_2U_6O_{24}$ plotted using the program DRAW-XTL [26] is shown in Fig. 4. In the structure, all the uranium (VI) atoms are present as uranyl. Each uranium atom is surrounded by six oxygen atoms in an octahedral geometry. The four O2 atoms form a square planer arrangement. The two O1 atoms occupy the axial positions. Six O1 oxygen atoms form regular octahedral geometry around the Sr atoms. Each UO6 octahedron is connected to four other uranium octahedra through O2 oxygen atoms and two SrO6 octahedra through O1 oxygen atoms. In this way, the regular SrO6 octahedron is also connected to six uranium octahedra through O1 oxygen atoms. Each sodium atom located in the voids (Fig. 3), is surrounded by six O1 oxygen atoms and six O2 oxygen atoms at the edges within a cube forming a 12-coordination geometry at distances ranging from 0.294 nm to 0.296 nm (Table 6). Thus the structure is made of chains of uranium octahedra on one side and chains of alternating uranium and Sr octahedra on the other side. Chemically, the compound can be considered to be formed by replacement of one third of monovalent sodium ion in Na_2UO_4 with bivalent strontium ions. Structurally, they resembled the perovskite KUO_3 and $RbUO_3$, where 1/4 of the U(V) atom is substituted by Sr^{+2} and thereby forming uranyl octahedral, leading to near doubling of the unit cell of KUO_3 and $RbUO_3$ ($a = 0.429$ and 0.4327 nm, respectively). Structures of KUO_3 and $RbUO_3$ are cubic [27], but in the critical review of Na–U–O system Kleyclamp [1] has reported the structure of $NaUO_3$ as orthorhombic which was confirmed by neutron diffraction by Chippinadale et al. [28].

The collective data of interatomic distances (nm) in the isostructural compounds $Na_8Sr_2U_6O_{24}$ (obtained during the present study) and $K_8Sr_2U_6O_{24}$ and $Rb_8Sr_2U_6O_{24}$ (reported earlier) [10,12] is given in Table 6. From the data it is confirmed that in alkali metal atoms, as atomic radii increases bond lengths also increases.

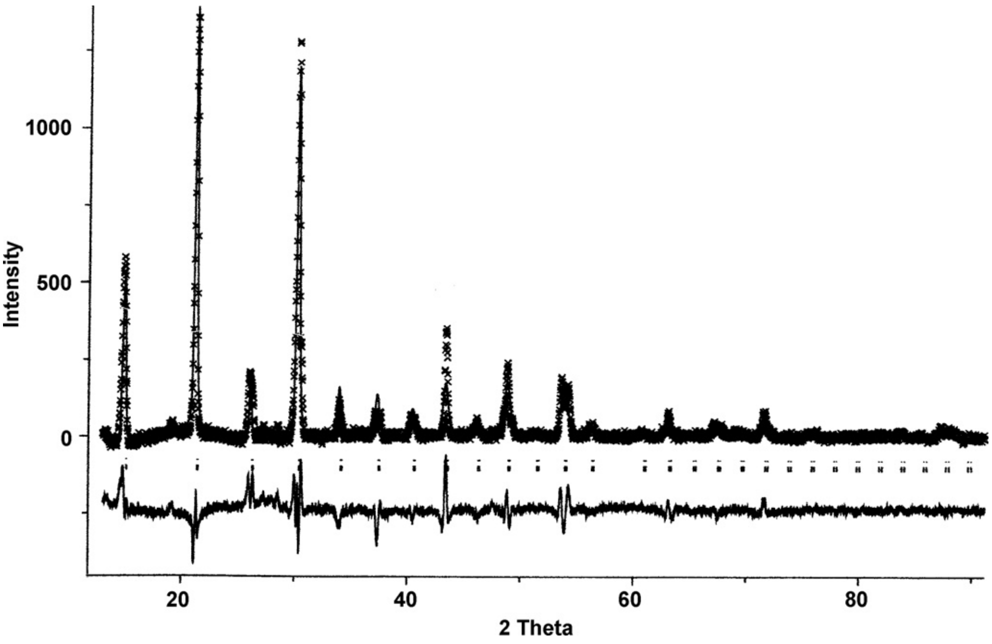

Fig. 3. Rietveld plot of observed and calculated diffraction pattern of $Na_8Sr_2U_6O_{24}$.

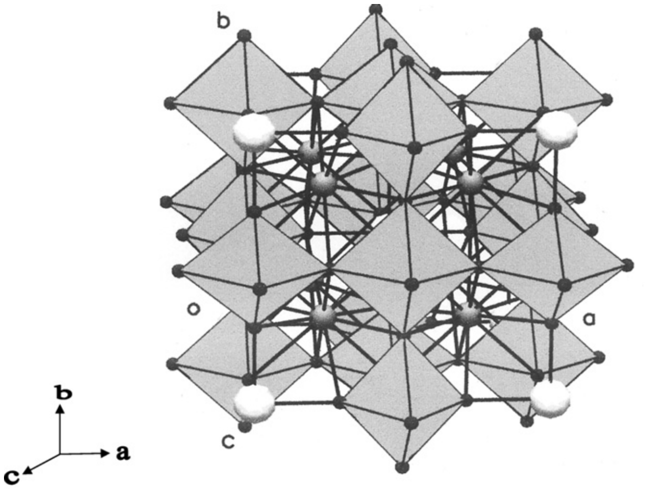

Fig. 4. Structure of $Na_8Sr_2U_6O_{24}$ showing uranium octahedral. Sr-(○), Na-(●), O-(•).

Table 6
Interatomic distances (nm) in $Na_8Sr_2U_6O_{24}$, $K_8Sr_2U_6O_{24}$ and $Rb_8Sr_2U_6O_{24}$

Bonding atoms	Number of bonds	$Na_8Sr_2U_6O_{24}$	$K_8Sr_2U_6O_{24}$	$Rb_8Sr_2U_6O_{24}$
U–O1	2×	0.1770	0.1840	0.1860
U–O2	4×	0.2084	0.2165	0.2185
Sr–O1	6×	0.2393	0.2490	0.2510
Na/K/Rb–O1	6×	0.2960	0.3061	0.3091
Na/K/Rb–O2	6	0.2944	0.3078	0.3108

4. Summary

A novel quaternary phase $Na_8Sr_2U_6O_{24}$ in the Na–Sr–U–O system was prepared and characterised by the powder XRD method. Structure of the compound was derived using Rietveld profile analysis of X-ray powder data. Based on the new phase, well established phases in Na_2O–UO_3 and SrO–UO_3 pseudo-binary systems and 15 phase mixtures synthesized and analysed by XRD in the present work, a quasi ternary phase diagram of Na_2O–SrO–UO_3 system has been drawn.

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