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OCCURRENCE AND MINERAL CHEMISTRY OF ISHIKAWAITE IN THE MINERALIZED PEGMATITES OF GABAL SAMMA, SOUTHEASTERN SINAI, EGYPT

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ABSTRACT

The occurrence and mineralogical characteristics of ishkawaite in unzoned pegmatite hosted in the monzogranite of Gabal Samma area, Southeastern Sinai, Egypt are investigated. Ishkawaite occurs in highly radioactive pegmatitic bodies in which the averages of uranium and thorium contents are (211&1480 ppm respectively). The mineral presents in association with uranophane, thorite, uranothorite, columbite, betafite, metamict zircon and fluorite. The pegmatites occur as pockets (5x10m) and dykes (1x20m) hosted in the monzogranites. The investigated mineral is generally black to dark brown in color, forming anhedral grains with a black to brown streak and opaque with vitreous luster.

The mineralogy and geochemistry of the studied ishkawaite were determined using microscopic examination as well as quantitative analysis by ICP-MS. The ICP-MS shows the average oxides composition: 47.61% Nb₂O₅, 22.83% UO₂, 6.06% Fe₂O₃, 5.44% Ta₂O₅, 4.69% REE₂O₃, 4.34% ThO₂, 3.23% CaO and 2.99% Y₂O₃. The minor to trace elements include Mn, Ti, Al and Zr. The calculated empirical formula is (U_{0.231} Th_{0.045} Fe⁺³_{0.207} Ca_{0.157} Y_{0.071} REE_{0.06} Mn_{0.048} Al_{0.021} Na_{0.015} K_{0.007} Zr_{0.007} Mg_{0.002} Hf_{0.001})_{Σ0.883} (Nb_{0.976} Ta_{0.067} Ti_{0.028})_{Σ1.071} O₄. The simplified formula is (U, Th, Fe⁺³, Ca, Y, REE) (Nb, Ta, Ti) O₄. The formula of the study ishkawaite is non stoichiometric and show some deficiency in the A-site relative to the B-site. Metamictization, a common feature of ishkawaite is suspected to be the main reason for this deviation from ideal stoichiometry.

The deviations of Y/Ho and Zr/Hf ratios of the investigated ishkawaite from chondritic trace-element ratios are characteristic for strong magmatic with hydrothermal alteration.

INTRODUCTION

Samarskite-group minerals having general formula (A³⁺B⁵⁺O₄) where A represents Fe, Ca, REE, Y, U and Th, while B represents Nb, Ta and Ti can be classified into: samarskite-Y, calciosamarskite and ishkawaite. The only significant difference between samarskite-Y, ishkawaite and calciosamarskite lies in the occupancy of the A-site. The A-site of samarskite-Y is dominated by Y+REE whereas the

A-site of ishkawaite is dominantly U+Th and calciosamarskite is dominantly Ca. Also, ishkawaite is depleted in light rare earth elements (LREE) and enriched with heavy rare earth elements (HREE) (Hanson et al. 1999). Due to its very complex chemical composition, occurrence in metamict state and frequent pervasive alteration, the definition of samarskite and description of its crystal structure has always been a challenge (Tomasic et al., 2010).

Kimura (1922) was the first one who described ishikawaite (U,Fe,Y,Ca) (Nb,Ta) O₄ as U-rich, REE-poor mineral. After two years, Ohashi (1924) stated that, there is a crystallographic relationship exists between ishikawaite and samarskite. Cerny and Ercit (1989) considered ishikawaite as a probable U-rich samarskite. Warner and Ewing (1993) proposed that samarskite is an A³⁺ B⁵⁺ O₄ mineral with an atomic arrangement related to α -PbO₂, where B-site contains Nb, Ta and Ti and the A-site contains all other cations.

Hanson et al. (1999) stated that samarskite –group minerals should include only those that have Nb > Ta and Ti in the B-site. The X-ray diffraction analysis of the recrystallized type specimen of ishikawaite reveal that they have the same structure as samarskite-Y recrystallized at high temperature.

In Egypt, Raslan (2008) reported the presence of ishikawaite in the mineralized Abu Rushied Gneiss, South Eastern Desert. Ishikawaite mineral is associated with columbite, Hf-rich zircon and dark Li-mica mineral.

This study presents genuine data on the mineral chemistry of the ishikawaite occurring in the pegmatitic bodies hosted in the monzogranite of Gabal Samma area. The available data may contribute significantly to the genesis of the mineral.

GEOLOGIC SETTING

Gabal Samma area is delineated by Latitudes 28° 9' and 28° 11' N. Longitudes 34° 8' and 34° 15' E. (Fig. 1). Gabal Samma is an oval shaped pluton consisting of monzogranites trending E-W direction. Generally, these monzogranites intrude gneisses and migmatites and older granitoids with sharp contacts. The pegmatites present in the monzogranite and concentrated along the pluton periphery (Abd Allah, 1995 & Nasr, 2006). The monzogranites are hard, massive, medium-, to coarse-grained, grey to pink in color and are rich in black mafic minerals. They are characterized

by cavernous weathering and intensive joints taking four trends. The most dominant trends are NW-SE and NE-SW, while N-S and E-W are subordinate. The monzogranites are traversed by dykes varying in composition from acidic to basic and trending mainly to NE-SW direction beside subordinate NW-SE direction (Ahmed, 1985). Petrographically, monzogranite is composed mainly of plagioclases, quartz, K-feldspars and biotite. Plagioclases occur as euhedral crystals twinned according to albite law. They are present in two sizes, medium-sized crystals of oligoclase and fine-sized albite crystals. Some of the phenocrysts exhibit discontinuous lamellar twinning and dissected by cracks filled with iron oxides. Quartz occurs as fine crystals accompanying albite in the ground mass or included in the recrystallized oligoclase and as medium crystals associating the other constituents. Potash feldspars occur as subhedral crystals of string perthite. Mica minerals represent about 4% of the rock occurring as flakes of biotite occasionally enriched by iron oxides (ferribiotite) and characterized by pleochroic halos around minute crystals of zircon. Accessory minerals are mainly sphene and zircon. The sphene crystals are either primary characterized by sphenoidal shape or secondary with undefined forms and associating opaque minerals. Zircon is partially metamictized with obvious clouded colors, mostly yellow to grey, due to wide spreading of the radioelements. They are also zoned and fractured exhibiting radial fractures.

The pegmatites are unzoned and occurring as pockets and dykes hosted in monzogranite of Gabal Samma. They are very coarse-grained and have pink color. The pockets vary in size from 0.3 to 10m in length and from 0.2 to 5m in width in addition to several small pockets of size varying from 25 to 75 cm in length and 10 to 25 cm in width. The pegmatitic dykes have thickness vary from 25 to 75 cm and extend for about 20m. Three pegmatitic anomalies are recorded in the pegmatitic bodies hosted in the monzogranite of Gabal Samma. The radiometric measurements

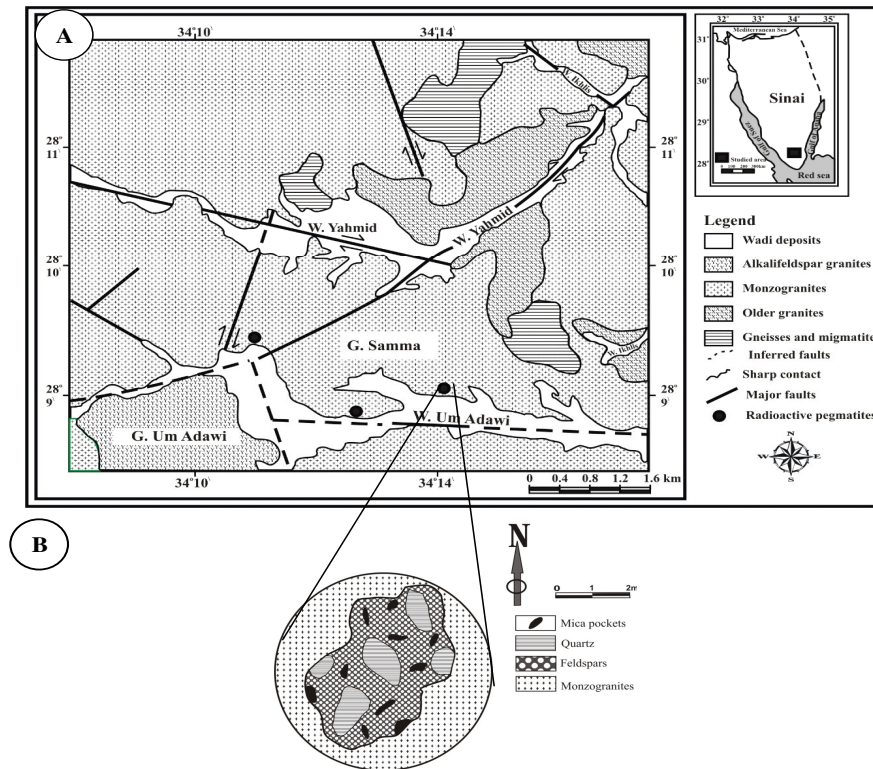


Fig.1: A) Geological map of Gabal Samma (after Nasr, 2006)
 B) Sketch showing the mode of occurrence of pegmatite pocket

of these pegmatitic anomalies using Gamma spectrometer technique show that the average of the uranium and thorium contents in these anomalies are 211 & 1480ppm respectively. Samarskite, columbite, thorite, uranothorite, betafite, metamict zircon and fluorite are the accessory minerals (Nasr, 2006).

METHODOLOGY

Representative 5 mineralized samples were collected from the radioactive pegmatites of Gabal Samma. The samples were crushed, ground, sieved and then were subjected to heavy liquid separation process using bromoform (sp. gr = 2.85gm/cm³). The

pure individual ishikawaite grains were hand-picked and investigated under the binocular microscope. The identification of the picked mineral was carried out by X-ray diffraction technique (XRD) using unit Philips PW-3710 with generator PW-1830, Cu target tube and Ni filter at 40 Kv and 30 mA at the Egyptian Nuclear Materials Authority labs.

Semi-quantitative analysis was carried out by the Environmental Scanning Electron Microscope (ESEM) supported by Energy Dispersive Spectrometer unit (EDS) model Philips XL at the Egyptian Nuclear Materials Authority Labs. The analytical conditions were 25-30 Kv accelerating voltages, 1-2 micron beam diameter and 60-120 second counting

times. Minimum detectable weight concentration of elements ranges from 0.1 to 1 wt%. The quantitative analysis of ishkawaite was further carried out by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at the Acme Labs, Vancouver, Canada. About 5 gm of the picked mineral was ground in an agate mortar to 200 mesh size. The samples were fused with LiBO₂ digested and analyzed using the ICP-MS. The analytical precision monitored by international rock standards is found to be better than $\pm 7\%$ for rare earth elements (REE) and with 5-10% for trace elements and $\pm 2\%$ for major oxides.

MICROSCOPIC INVESTIGATION

The mineral occurs as black to dark brown anhedral grains with a black to brown streak (Fig.2). Ishikawaite is opaque with vitreous luster. Cleavage and parting are absent because it is completely metamict. The resulting metamict state is due to the recoiling destruction of U and Th for the crystal structure (Tomasic et al., 2008). The destruction of a mineral crystal lattice in a metamict mineral is reflected in its physical properties. Usually, this

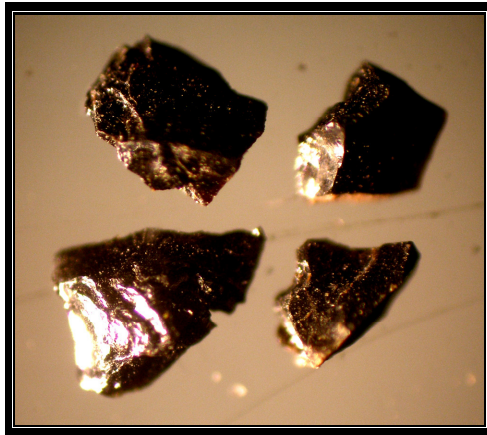


Fig.2: Anhedral ishkawaite grains, binocular microscope

means a decrease in the density, the hardness, the birefringence and the refraction indices for metamict minerals.

The hardness of the study ishkawaite ranges from 5 to 6 and the specific gravity is 6.3 gm/cm³. Due to pervasive alteration and advanced metamictization of the mineral, the sample shows no diffraction peaks in the X-ray diffraction study. So, the sample was recrystallized by heating to a temperature of 1100 °C for 12 hours in reducing conditions. Figure (3) shows the XRD pattern of the recrystallized mineral.

CHEMICAL COMPOSITION

Several crystals of ishkawaite were subjected to semi-quantitative analyses using an Environmental Scanning Electron Microscope (ESEM). The EDX revealed that the ishkawaite crystals are characteristically enriched in Nb₂O₅, UO₂, Fe₂O₃, Ta₂O₅, TiO₂, ThO₂, CaO, Y₂O₃ and traces of MnO (Table,1 and Figs. 4 &5).

However, 4 samples of ishkawaite were subjected to ICP-MS analysis and results are

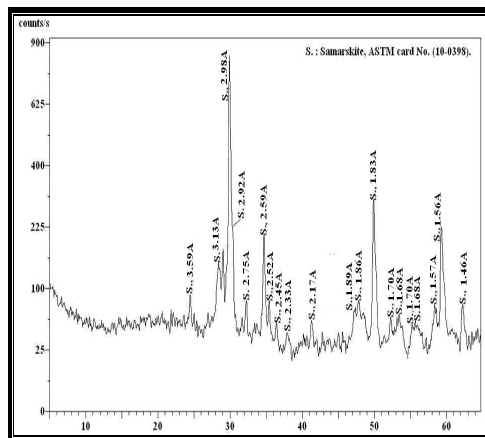


Fig.3: XRD pattern of samarskite grains

Table 1: EDX analysis of ishikawaite

Oxide	Nb ₂ O ₅	Ta ₂ O ₅	TiO ₂	UO ₂	ThO ₂	Y ₂ O ₃	Fe ₂ O ₃	CaO	MnO
Wt%	43.33	5.9	4.13	23.79	3.92	2.14	5.08	7.66	0.97

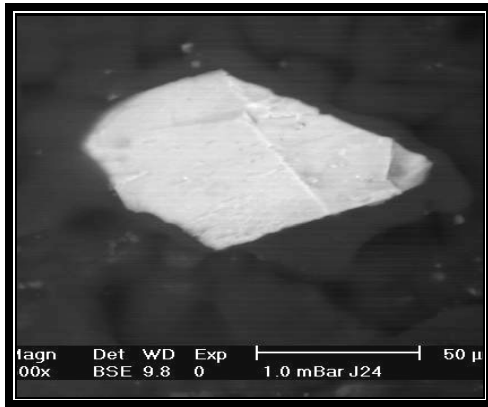


Fig.4: BSE image of ishikawaite

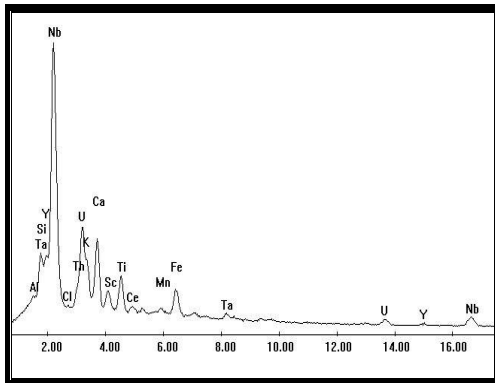


Fig.5: EDX spectrum of ishikawaite

given in Table (2) and Fig. (6). The analyzed data of the studied isikawaite were plotted on the ternary diagram of Hanson et al. (1999), which shows the A-site occupancy of samarskite-group minerals (Fig. 7). The figure shows that all the study samarskite samples lie in the ishikawaite field as they are U+Th dominant.

From the analytical data it is quite clear that the study mineral reflects the chemical composition of ishikawaite as indicated by the following evidences:

1- Both samarskite-Y and ishikawaite have a dominant Nb in the B-site and the distinction between them must be based on the content of B-site occupancy. The obtained ICP-MS data revealed that Nb₂O₅ is the dominant in the study mineral, it ranges from 45.7 to 51.13 wt% with an average of 47.61 wt%. Thus, the study mineral falls actually within the compositional limits of both samarskite-Y and ishikawaite.

2- The samarskite group of minerals must comprise only those that have Nb□ a and Ti in the B-site (Hanson et al., 1999), and the investigated mineral contains an average Ta+Ti=6.26 wt% Nb = 47.61 wt%.

3- The study mineral is rich in both uranium and thorium where U ranges from 20.65 to 25.67 wt% with an average of 22.83 wt %, while Th contents vary from 3.4 to 5.86 wt% with an average of 4.34 wt% (Table 1). According to Hanson et al. (1999), the study mineral is U-rich samarskite (ishikawaite).

The study ishikawaite likes other samarskite-group minerals is enriched in HREE relative to LREE (Hanson et al. 1999), where the average of ΣHREE is 3.73% compared with 0.96% for the ΣLREE. The HREE enrichment of the study ishikawaite may be the result of fluorine preferentially complexing with the HREE relative to the LREE (Simmons et al., 2006). These authors have shown that the equilibrium constant for HREE fluoride complexes is greater than that for LREE fluoride complexes. Thus, it is likely that the HREE are preferentially incorporated into the late-stage fluids, which ultimately produce the mineralogy of the late-stage replacement units. High fluorine fugacity is manifested by the presence of fluorite as an accessory mineral in the pegmatite.

Oxide analyses are recalculated on the

Table 2: Chemical analyses and chemical formulae of the study ishkawaite, Gabal Samma pegmatite

	1	2	3	4	Average	a	b
Nb ₂ O ₅	45.7	51.13	47.12	46.5	47.61	49.49	37.8
Ta ₂ O ₅	8.77	5.67	3.17	4.13	5.44	3.34	5.77
TiO ₂	0.68	0.87	0.95	0.78	0.82	0.35	0.45
ThO ₂	4.38	3.7	5.86	3.4	4.34	3.85	3.27
UO ₂	21.8	20.65	23.18	25.67	22.83	26.37	31.8
La ₂ O ₃	0.13	0.12	0.10	0.15	0.13	-	b.d.l.
Ce ₂ O ₃	0.12	0.11	0.12	0.13	0.12	-	0.1
Pr ₂ O ₃	0.07	0.10	0.11	0.13	0.10	-	0.06
Nd ₂ O ₃	0.36	0.22	0.28	0.24	0.28	-	0.56
Sm ₂ O ₃	0.29	0.25	0.34	0.37	0.31	-	0.29
Eu ₂ O ₃	0.02	0.02	0.02	0.03	0.02	-	b.d.l.
Gd ₂ O ₃	0.97	0.92	0.95	1.06	0.98	-	1.17
Tb ₂ O ₃	0.14	0.12	0.14	0.21	0.15	-	0.14
Dy ₂ O ₃	0.85	0.89	0.95	1.28	0.99	-	0.85
Ho ₂ O ₃	0.20	0.28	0.29	0.39	0.29	-	0.26
Er ₂ O ₃	0.79	0.75	0.64	0.85	0.76	-	0.77
Tm ₂ O ₃	0.13	0.11	0.13	0.19	0.14	-	0.09
Yb ₂ O ₃	0.27	0.24	0.21	0.28	0.25	-	0.17
Lu ₂ O ₃	0.16	0.14	0.16	0.22	0.17	-	0.11
Y ₂ O ₃	3.04	2.87	3.67	2.39	2.99	0.84	3.04
ZrO ₂	0.36	0.27	0.25	0.33	0.30	0.21	0.36
HfO ₂	0.10	0.04	0.08	0.09	0.08	-	-
MnO	1.29	1.23	1.27	1.25	1.26	0.63	2.02
MgO	0.01	0.05	0.04	0.01	0.03	-	-
CaO	2.87	2.97	3.2	3.88	3.23	0.57	0.07
Na ₂ O	0.16	0.14	0.19	0.17	0.17	-	-
K ₂ O	0.13	0.10	0.11	0.11	0.11	-	-
Al ₂ O ₃	0.42	0.41	0.43	0.25	0.38	-	-
Fe ₂ O ₃	6.48	5.45	6.00	6.30	6.06	12.51	8.9
Total	100.69	99.82	99.96	100.79	100.32	100.01	100.3
ΣREE	4.50	4.27	4.44	5.53	4.69	0.15	4.57
ΣLREE	0.99	0.82	0.97	1.05	0.96	-	-
ΣHREE	3.51	3.45	3.47	4.48	3.73	-	-

Chemical formula based on 4 oxygen atoms							
Nb	0.941	1.029	0.973	0.961	0.976	1.00	0.854
Ta	0.109	0.069	0.039	0.051	0.067	0.03	0.078
Ti	0.023	0.029	0.033	0.027	0.028	0.01	0.017
Sum B	1.073	1.127	1.045	1.039	1.071	1.04	1.048
Th	0.045	0.038	0.061	0.035	0.045	0.04	0.037
U	0.221	0.205	0.236	0.261	0.231	0.28	0.354
La	0.002	0.002	0.002	0.003	0.002	-	0
Ce	0.02	0.002	0.002	0.002	0.002	-	0.002
Pr	0.001	0.002	0.002	0.002	0.002	-	0.001
Nd	0.006	0.004	0.005	0.004	0.005	-	0.010
Sm	0.005	0.004	0.005	0.006	0.005	-	0.005
Eu	0.000	0.000	0.000	0.000	0.000	-	0
Gd	0.015	0.014	0.015	0.016	0.015	-	0.019
Tb	0.002	0.002	0.002	0.003	0.002	-	0.002
Dy	0.013	0.013	0.014	0.019	0.015	-	0.014
Ho	0.003	0.004	0.004	0.006	0.004	-	0.004
Er	0.011	0.011	0.009	0.012	0.011	-	0.012
Tm	0.002	0.002	0.002	0.003	0.002	-	0.001
Yb	0.004	0.003	0.003	0.004	0.004	-	0.003
Lu	0.002	0.002	0.002	0.003	0.002	-	0.002
Y	0.074	0.068	0.089	0.058	0.071	0.02	0.081
Zr	0.008	0.006	0.006	0.007	0.007	0.01	0.009
Hf	0.001	0.001	0.001	0.001	0.001	-	-
Mn	0.050	0.046	0.049	0.048	0.048	0.02	0.086
Mg	0.001	0.003	0.003	0.001	0.002	-	-
Ca	0.140	0.142	0.157	0.190	0.157	0.03	0.004
Na	0.014	0.012	0.017	0.015	0.015	-	0.003
K	0.008	0.006	0.006	0.006	0.007	-	-
Al	0.023	0.022	0.023	0.014	0.021	-	0.013
Fe	0.222	0.183	0.206	0.217	0.207	0.46	0.262
Sum A	0.875	0.798	0.921	0.936	0.883	0.985	0.952
Zr/Hf	8.00	6.00	6.00	7.00	6.75	-	-
Y/Ho	24.67	17.00	22.25	9.67	18.40	-	-

a-Average of 4 microprob analyses of Abu Rushied ishkawaite samples (Raslan, 2008)

b-Average of 8 microprob analyses of Ishikawa, Japan, samples (Hanson et al., 1999)

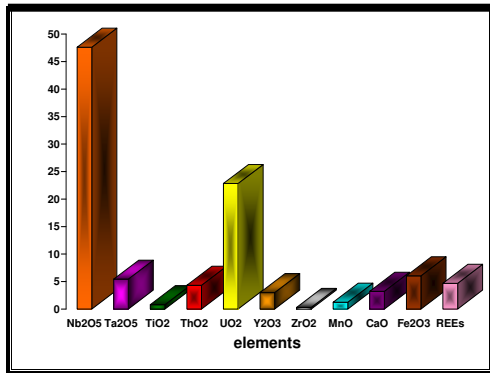


Fig.6: The chemical composition of the investigated ishikawaite as obtained by ICP-MS

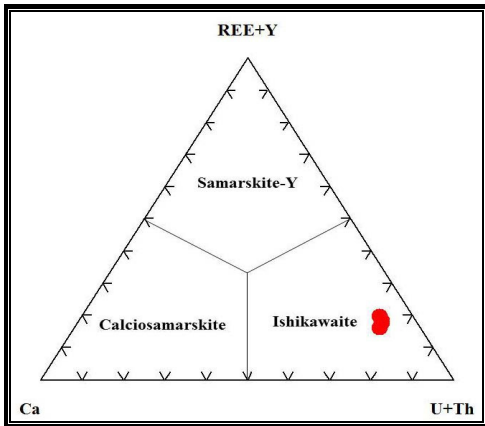


Fig.7: Ternary diagram showing A-site occupancy of samarskite-group minerals, After Hanson et al. (1999)

basis of 4 oxygens where Nb, Ta and Ti are assigned to the B-site and U, Th, Y, Fe, Ca, Mn and REE are occupying the A-site (Table 1). The empirical formula is $(U_{0.231}Th_{0.045}Fe^{+3}_{0.007}Ca_{0.207}Y_{0.157}Mn_{0.071}Al_{0.048}Na_{0.021}K_{0.015}Zr_{0.007}Mg_{0.002}Hf_{0.001}[Gd_{0.015}Dy_{0.015}Er_{0.011}Sm_{0.005}Nd_{0.005}Ho_{0.004}Yb_{0.004}Lu_{0.002}Tm_{0.002}Ce_{0.002}La_{0.002}Pr_{0.002}Tb_{0.002}])_{\Sigma 0.883}(Nb_{0.976}Ta_{0.067}Ti_{0.028})_{\Sigma 1.071}O_4$. The simplified formula is

$(U, Th, Fe^{+3}, Ca, Y, REE)(Nb, Ta, Ti)O_4$.

The formula of the study ishikawaite is non stoichiometric and show some deficiency in the A-site (A varies from 0.798 to 0.936 with an average of 0.883) relative to the B-site (B ranges from 1.039 to 1.127 with an average of 1.071). Metamictization, a common feature of ishikawaite is suspected to be the main reason for this deviation from ideal stoichiometry (Uher et al., 2009).

DISCUSSION AND CONCLUSION

The occurrence and physicochemical characteristics of ishikawaite in unzoned pegmatitic bodies hosted in the monzogranite of Gabal Samma area, Southeastern Sinai, Egypt are investigated. The mineral occurs in a radioactive pegmatitic bodies in which the average of uranium and thorium contents are 211 and 1480 ppm respectively. The mineral occurs in association with uranophane, thorite, uranothorite, columbite, betafite, metamict zircon and fluorite.

The investigated ishikawaite is generally black to dark brown in color, anhedral grains with a black to brown streak and opaque with vitreous luster.

The study ishikawaite shows clear decreasing in the Zr/Hf ratio (vary from 6.00 to 8.00 with an average of 6.75) which is lower than the chondritic Zr/Hf ratio =38 Anders and Grevesse (1989), this is characteristic for strong magmatic with hydrothermal alteration (Bau, 1996, Irber, 1999, Geisler et al., 2003 and Rossi et al., 2011). Also, the average Y/Ho ratio of the study mineral is 18.40 lower than the chondritic Y/Ho ratio =28 Anders and Grevesse (1989). Alteration is accompanied by pronounced deviation from the chondritic Y/Ho ratio (Van Dongen et al., 2010).

In conclusion, The study ishikawaite may be subjected to the hydrothermal alteration, after metamictization, because of metamict-

tization leads to the volume expansion and consequent formation of the cracks. These fractures enhanced fluid flux and related interaction processes (Zacek et al., 2009).

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تواجد وكميائيه معدن الأشكاويت فى البجماتيت المتمعدن جبل صما جنوب شرق سيناء - مصر

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تم دراسة تواجد معدن الأشكاويت وخصائصه المعدنية و الكيمائية فى البجماتيت الغير ممنطق الموجود فى صخور المونزوجرانيت بجبل صما جنوب شرق سيناء - مصر. يتواجد البجماتيت فى شكل جيوب وقواطع موجودة فى صخور المنزوجرانيت . يوجد معدن الأشكاويت فى صخور البجماتيت عالية الاشعاعية والتي يبلغ متوسط محتوى اليورانيوم والثوريوم فيه ٢١١ و ١٤٨٠ جزء فى المليون بمصاحبة معادن اليورانوفين، الثورابت، اليورانوثوريت، الكلومبيت، البيتافيت، الزركون المشع والفلوريت. يوجد معدن الأشكاويت فى شكل حبيبات غير منتظمة الشكل يتراوح لونها من الاسود الى البنى الغامق. وقد أوضحت التحاليل الكيمائية الكمية أن معدن الأشكاويت يحتوى على متوسطات لأوكسيد النيوبيوم ٤٧,٦١ %، أكسيد اليورانيوم ٢٢,٨٣ %، أكسيد الحديد ٦,٠٦ %، أكسيد التنتالم ٥,٤٤ %، أكسيد الثوريوم ٤,٣٤ %، أكسيد الكالسيوم ٣,٢٣ %، أكسيد الاثريم ٢,٩٩ % بالإضافة الى أكاسيد العناصر الارضية النادرة ٤,٦٩ % مع وجود بعض العناصر الاخرى بكميات شحيحة مثل الزركونيوم والمنجانيز والالمنيوم. وقد وجد ان عملية metamictization للمعدن قد أدت الى وجود تركيب غير مثالى للمعدن nonstoichiometric وبحساب بعض النسب مثل Y/Ho - Zr/Hf وجد أنها تقل عن مثيلاتها فى الكونديريت وهذا دليل على تكون الأشكاويت من المراحل الاخيرة للمجما وتأثره بالمحاليل المائية الحاره.