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Rameauite and agrinierite, two new hydrated complex uranyl oxides from Margnac, France

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SUMMARY. Two new hydrated uranyl oxides from Margnac, France, are described. Rameauite, $K_2O \cdot CaO \cdot 6UO_3 \cdot 9H_2O$, occurs with uranophane and calcite on pitchblende, agrinierite, $2(K_2O, CaO, SrO) \cdot 6UO_3 \cdot 8H_2O$, with uranophane in small cavities in 'gummitte'.

Rameauite is monoclinic with a 13.97, b 14.26, c 14.22 Å, β $121^\circ 1' \pm 5'$; the space group is $C2/c$. The principal d -spacings, with hkl values and relative intensities, are: 7.12, 020, vvs; 3.566, 040, vs; 3.495, 402, vvs; 3.473, 202, vs; 3.185, 224, vs; 3.139, 422, vvs; 2.993, 400, vvs; 2.495, 442, ms. Measured density 5.6 g.cm⁻³. The orange crystals are twinned on {100}, flattened on {010} and elongate parallel to [001]; there is a good {010} cleavage. $\alpha \parallel [010]$, γ : [001] 4 to 6° in the obtuse angle β , $2V_\alpha$ 32°; the two principal reflectivities for yellow light on {010} are 10.6 and 10.4%. The main endothermic peaks on the d.t.a. diagram are at 95, 130, and 170 °C (loss of water), and at 1050 °C (loss of oxygen).

Agrinierite is orthorhombic with a 14.04, b 24.07, c 14.13 Å; space group $Cmmm$ on the assumption of holohedral symmetry. Measured density 5.7 g.cm⁻³. The principal d -spacings are 7.08, 002, vvs; 6.05, 220, ms; 3.516, 400 & 332, s; 3.485, 062 & 260, vs; 3.153, 204 & 134, vs; 3.128, 262, vvs; 2.023, 660, s. The orange crystals are tabular on {001} with pseudo-hexagonal section bounded by the trace of {010}, and show sector twinning on {110} as in billietite; there is a good {001} cleavage. The two reflectivities for yellow light on the face {001} are 12.0 and 11.2%, corresponding to a refractive index of about 2. $\alpha \parallel [001]$, $\gamma \parallel [010]$, $2V_\alpha$ 55°. The principal endothermic d.t.a. peaks are at 163 °C (loss of water) and 1060 °C (loss of oxygen).

Rameauite is named after J. Rameau and agrinierite after H. Agrinier. The original specimens are deposited in the Mineralogical Collection of the University of Paris VI.

RÉSUMÉ. Deux nouveaux uranates hydratés provenant de Margnac, France, sont décrits. La rameauite, de composition idéale $K_2O \cdot CaO \cdot 6UO_3 \cdot 9H_2O$, est associée à l' α -uranotile et à la calcite sur de la pechblende, l'agrinierite, $2(K_2O, CaO, SrO) \cdot 6UO_3 \cdot 8H_2O$, a été observée avec l' α -uranotile dans des petites cavités dans de la 'gummitte'.

La rameauite est monoclinique avec a 13,97, b 14,26, c 14,22 Å, β $121^\circ 1' \pm 5'$; le groupe spatial est $C2/c$. Les raies principales du diagramme de poudre sont: 7,12, 020, FFF; 3,566, 040, FF; 3,495, 402, FFF; 3,473, 202, FF; 3,185, 224, FF; 3,139, 422, FFF; 2,993, 400, FFF; 2,495, 442, mF. La densité mesurée est égale à 5,6 g.cm⁻³. Les cristaux, oranges, sont maclés sur {100}, aplatis sur {010}, et allongés parallèlement à [001]. Le clivage {010} est bon. $\alpha \parallel [010]$, γ : [001] 4 à 6° dans l'intérieur de l'angle β ; $2V_\alpha = 32^\circ$; les deux pouvoirs reflecteurs, pour la lumière jaune, de la face {010} sont égaux à 10,6 et 10,4%. L'analyse thermique différentielle donne des pics à 95, 130 et 170 °C (départ d'eau) et à 1050 °C (départ d'oxygène).

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L'agrinierite est orthorhombique avec a 14,04, b 24,07, c 14,13 Å; le groupe spatial est *Cmmm* en supposant le minéral holoèdre. Les raies principales du diagramme de poudre sont: 7,08, 002, FFF; 6,05, 220, mF; 3,516, 400 & 332, F; 3,485, 062 & 260, FF; 3,153, 204 & 134, FF; 3,128, 262, FFF; 2,023, 660, F. La densité mesurée est égale à 5,7 g.cm⁻³. Les cristaux, de couleur orange, sont aplatis sur {001} avec un contour pseudo-hexagonal et montrent des macles en secteur selon (110) comme dans la billiéite. Il y a un bon clivage suivant {001}. Les indices de refraction correspondant aux deux pouvoirs reflecteurs de la face (001) sont de l'ordre de 2 pour la lumière jaune; $\alpha \parallel [001]$, $\gamma \parallel [010]$; $2V_\alpha = 55^\circ$. Les principaux pics endothermiques sont à 163 °C (départ d'eau) et à 1060 °C (perte d'oxygène).

Le nom de rameauite est donné en l'honneur de J. Rameau et celui d'agrinierite en l'honneur de H. Agrinier. Les échantillons originaux sont déposés dans la Collection de minéralogie de l'Université de Paris VI.

THERE are several hydrated uranyl oxides containing barium, calcium, and potassium, including the end-members billietite, BaO.6UO₃.11H₂O, becquerelite, CaO.6UO₃.11H₂O, and compreignacite, K₂O.6UO₃.11H₂O. Two new species having compositions K₂O.CaO.6UO₃.9H₂O and 2(K₂O,CaO,SrO).6UO₃.8H₂O, between becquerelite and compreignacite, with different lattice parameters and space groups, have been discovered at Margnac, in the Massif Central, France.

Rameauite, monoclinic, is named after Jacques Rameau (†1960), prospector of the Commissariat à l'Énergie Atomique, who discovered several uranium deposits including that of Margnac 2. Agrinierite, orthorhombic, is named in memory of Henri Agrinier (1928–71), engineer in the mineralogy laboratory at the Commissariat à l'Énergie Atomique since 1950. He contributed to the identification by microchemistry of most of the uranium-bearing minerals studied by the C.E.A. in France and abroad. He specialized in paper chromatography and applied it to problems of geochemistry (e.g. Se and Te), and especially to the development of rapid field methods for V, Mo, P, Au, etc. In addition to his innate temperament for research, his professional conscience and his talent for teaching, he had a great loyalty that was appreciated by all who knew him.

The descriptions of these two new minerals were submitted to the I.M.A. before publication. The original specimens are deposited in the Mineralogical Collection, Université de Paris VI.

Rameauite occurs as small (up to 1 mm long) orange crystals with calcite and α -uranophane on a block of pitchblende. They are all twinned on {100}, slightly flattened parallel to {010}, elongated parallel to [001], with a pseudo-hexagonal section (fig. 1). The observed forms are {010}, {100}, {001}, and {110}; measured (100):(001) = 58° 40', (010):(110) = 49° 50'. There is a good cleavage parallel to {010}.

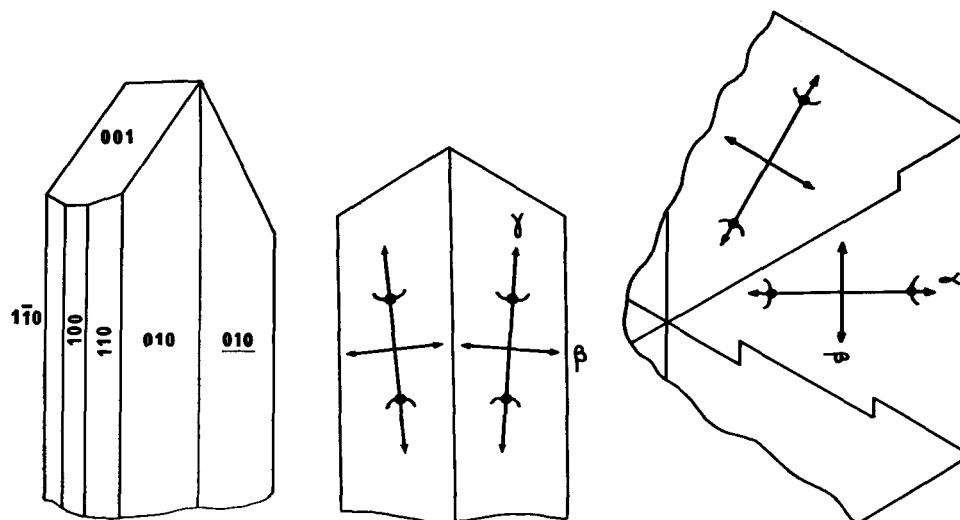
Rameauite is monoclinic pseudo-hexagonal with a 13.97, b 14.26, c 14.22 Å, β 121° 1' ± 5'; the cell-volume is 2426 Å³; space group *C2/c* or *Cc*. The relation between the morphology and the unit-cell axes was determined with a Buerger precession camera. The d -values and relative intensities (visual estimates) of the powder diffraction lines are given in Table I. The extinction angle γ : [001] on (010) is 4 to 6° in the obtuse angle β ; $\alpha \parallel [010]$, and $2V_\alpha$ is 32° (fig. 2). The two reflectivities were measured on the face (010) between 400 and 700 nm, and the corresponding refractive indices calculated (table II).

TABLE I. X-ray powder data for rameauite and agrinierite.
Guinier-de Wolff focusing camera; d_{\max} 11 Å.

Rameauite; Cu-K α_1 λ 1.5405 Å				Agrinierite; Cu-K $\alpha_1+\alpha_2$ λ 1.5418 Å			
d_{obs}	I	hkl	d_{calc}	d_{obs}	I	hkl	d_{calc}
7.12 Å	vvs	020	7.13 Å	9.22 Å	vwv	111	9.20 Å
6.12	mw	002	6.09	7.08	vvs	002	7.07
5.99	mw	200	5.99	6.05	ms	{ 220	6.06
4.59	vw	220	4.58			{ 040	6.02
4.41	vwv	{ 130	4.42	4.98	vwv	202	4.98
		{ 11 $\bar{3}$	4.41	4.61	vw	222	4.60
4.36	vwv	{ 31 $\bar{2}$	4.37	4.59	vwv	310	4.59
		{ 112	4.35	4.39	vw	113	4.39
3.566	vs	040	3.565	4.02	vwv	{ 330	4.04
3.495	vvs	40 $\bar{2}$	3.491			{ 060	4.01
3.473	vs	202	3.469	3.540	mw	004	3.533
3.309	vwv	{ 11 $\bar{4}$	3.307	3.516	s	{ 400	3.510
		{ 332	3.304			{ 332	3.509
3.185	vs	22 $\bar{4}$	3.180	3.485	vs	{ 062	3.489
3.139	vvs	422	3.136			{ 260	3.483
3.124	vvs	222	3.120	3.296	vwv	313	3.289
2.993	vwv	400	2.994	3.284	vwv	243	3.279
2.818	vwv	42 $\bar{4}$	2.816	3.153	vs	{ 204	3.156
		{ 15 $\bar{1}$	2.778			{ 134	3.151
2.776	vw	{ 150	2.774	3.128	vvs	262	3.124
		{ 33 $\bar{4}$	2.773	3.037	w	{ 422	3.041
		{ 13 $\bar{4}$	2.765			{ 440	3.032
2.761	w	{ 420	2.760	3.016	vwv	{ 172	3.019
2.521	m	24 $\bar{4}$	2.517			{ 080	3.009
2.495	ms	44 $\bar{2}$	2.494	2.793	m	{ 244	2.795
		{ 332	2.381			{ 154	2.791
2.380	w	{ 060	2.377	2.495	vw	404	2.490
		{ 025	2.306	2.483	m	{ 532	2.482
2.301	w	{ 60 $\bar{4}$	2.298			{ 264	2.480
2.287	w	313	2.283	2.407	vwv b	{ 315	2.407
2.268	vwv	402	2.265			{ 0.10.0	2.407
		{ 35 $\bar{4}$	2.189	2.395	vw	513	2.400
2.188	vw	{ 62 $\bar{4}$	2.187			551	2.391
		{ 15 $\bar{4}$	2.185	2.357	vw	006	2.355
		{ 153	2.173	2.301	m	444	2.301
2.176	vw	{ 224	2.173	2.288	vw	{ 084	2.291
		{ 351	2.164			{ 480	2.284
2.161	vw	{ 422	2.158	2.280	mw	{ 0.10.2	2.278
2.046	s	60 $\bar{6}$	2.044			{ 2.10.0	2.277
2.036	s	006	2.031	2.187	w	{ 514	2.189
		{ 31 $\bar{7}$	2.002			{ 622	2.184
1.999	m	{ 600	1.996	2.176	vw	{ 284	2.178
		{ 352	1.980			{ 570	2.175
1.979	vw	{ 264	1.976	2.171	vw	{ 482	2.174
		{ 71 $\bar{3}$	1.965			{ 2.10.2	2.167
1.966	ms	{ 626	1.965	2.140	vwv	{ 0.10.3	2.143
		{ 462	1.965			{ 1.11.1	2.137
		{ 531	1.959	2.084	vwv b	{ 642	2.084
1.957	ms	{ 172	1.958			{ 393	2.083
		{ 171	1.955	2.023	s	660	2.021

TABLE I (cont.)

Rameauite; Cu-K α_1 , λ 1.5405 Å				Agrinierite; Cu-K $\alpha_{1+\alpha_2}$, λ 1.5418			
d_{obs}	I	hkl	d_{calc}	d_{obs}	I	hkl	d_{calc}
1.932	vw	71 $\bar{5}$	1.932	2.007	m	0.12.0	2.006
		644	1.931	1.955	vw b	406	1.956
1.925	m	620	1.922	1.945	ms	730	1.946
		244	1.922	1.922	mw	176	1.925
						195	1.924
						712	1.923
				1.861	vw b	2.12.2	1.861
						446	1.860



FIGS. 1 to 3: FIG. 1 (left). Twinned crystal of rameauite. FIG. 2 (centre). Optical orientation of rameauite, in projection on (010). FIG. 3 (right). Twinned crystal of agrinierite, in projection on (001), showing the optical orientation.

Rameauite was analysed with a microprobe and the corrections carried out taking into account the quantity of water found by thermogravimetric analysis; strontium and barium were looked for but not found. The results, with the molecular ratios in parentheses, are: K₂O 5.0 (1.08), CaO 2.4 (0.87), UO₃ 84 (6), H₂O 8.2 (9.3), total 99.6%. The ideal formula is K₂O.CaO.UO₃.9H₂O; the measured density is 5.60 g.cm⁻³ and the calculated density 5.55 g.cm⁻³ for Z = 4. The thermogravimetric analysis was carried out on a sample of 8.04 mg, and the differential thermal analysis on a sample of 8 mg (fig. 4); the first three endothermic peaks corresponding to the loss of water occur at 95, 130, and 170 °C, and the small peak at 1050 °C corresponds to loss of oxygen with formation of U₃O₈. The thermogravimetric curve shows a rapid

loss of water up to about 130 °C and a slight, gradual loss up to about 500 °C, but no breaks or plateaux.

Agrinierite is orthorhombic pseudo-hexagonal, with a 14.04, b 24.07, c 14.13 Å, volume 4775 Å³. The only systematic absences indicate a C -centred cell, so the possible space groups are $Cmmm$, $Cm2m$, $Cmm2$, and $C222$. The d -values and relative intensities of the powder pattern are given in table I.

TABLE II. Reflectivities and corresponding (calculated) refractive indices for rameauite (face (010)) and agrinierite (face (001))

Rameauite					Agrinierite			
λ	R_{\max}	γ	R_{\min}	β	R_{\max}	γ	R_{\min}	β
400 nm	15.2 %	2.28	14.1 %	2.20	—	—	—	—
420	12.4	2.09	11.9	2.05	—	—	—	—
440	11.5	2.03	11.1	2.00	11.3 %	2.01	10.5 %	1.96
460	11.2	2.01	10.9	1.99	11.3	2.01	10.5	1.96
480	10.9	1.99	10.6	1.97	10.8	1.98	10.1	1.93
500	10.7	1.97	10.4	1.95	10.7	1.97	9.9	1.92
520	10.7	1.97	10.5	1.96	11.2	2.01	10.2	1.94
540	10.9	1.99	10.7	1.97	12.4	2.09	10.7	1.97
560	10.8	1.98	10.6	1.97	12.2	2.07	11.5	2.03
580	10.6	1.97	10.4	1.95	12.0	2.06	11.2	2.01
600	10.5	1.96	10.3	1.95	12.0	2.06	11.1	2.00
620	10.4	1.95	10.2	1.94	11.9	2.05	11.1	2.00
640	10.3	1.95	10.2	1.94	11.9	2.05	11.1	2.00
660	10.1	1.93	10.0	1.93	11.8	2.05	11.0	1.99
680	10.1	1.93	10.0	1.93	11.7	2.04	10.9	1.99
700	10.0	1.93	9.9	1.92	11.4	2.02	10.7	1.97

Agrinierite occurs as orange crystals, tabular on {001} with pseudo-hexagonal section bounded by the trace of (010), and showing sector twinning as in *billietite*; there is a good {001} cleavage. The two reflectivities measured on the face (001), between 440 and 700 nm, and the corresponding calculated refractive indices, are given in table II. The optic orientation is $\alpha \parallel [001]$, $\gamma \parallel [010]$; $2V_{\alpha} 55^{\circ}$.

Agrinierite was analysed by the microprobe and also by wet-chemical methods on 21 mg; water was determined thermogravimetrically. Strontium was found by the microprobe; barium was looked for but not found. The wet-chemical analysis gave (molecular ratios in parentheses): SrO 2.05 (0.40), K₂O 3.35 (0.72), CaO 2.20 (0.79), UO₃ 85.15 (6), H₂O 7.45 (8.34), total 100.20 %. The ideal formula is 2(K₂O,CaO, SrO).6UO₃.8H₂O, and with $Z = 8$ gives a calculated density of 5.62 g.cm⁻³, compared with the measured density of 5.7 g.cm⁻³.

The thermogravimetric analysis, carried out on 8.19 mg, showed a rapid loss up to about 130 °C and a more gradual one up to about 500 °C, but no breaks or plateaux. The differential thermal analysis, on 8 mg, showed an important endothermic peak

at 163 °C for the dehydration and another much smaller one at 1060 °C for loss of oxygen (fig. 4).

Comparison with some other uranyl oxides. Rameauite and agrinierite were compared with becquerelite and compreignacite, the calcium and potassium end-members, and with billietite, the barium analogue (table III). All five minerals are strongly pseudo-hexagonal with a very good cleavage normal to the pseudo-hexagonal axis. Precession photographs taken with this axis as precession axis are practically identical with

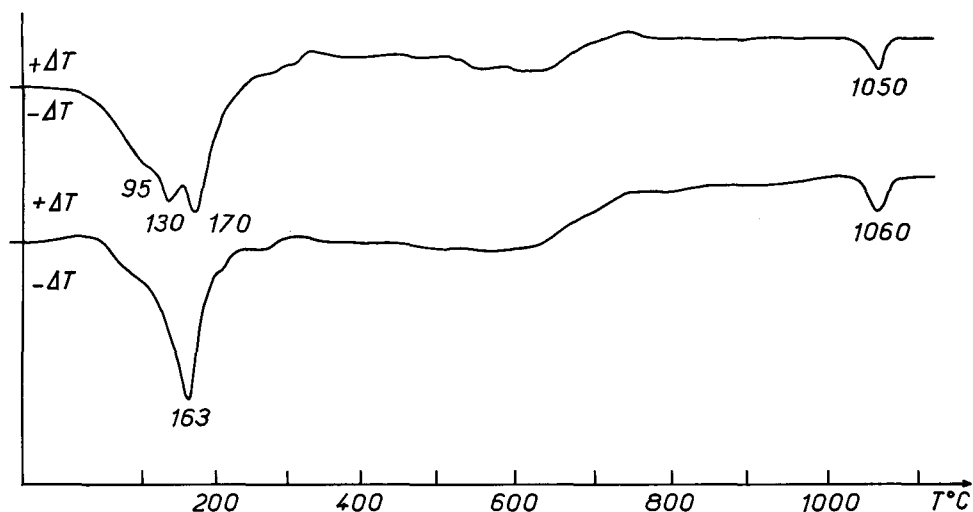


FIG. 4. D.t.a. curves of rameauite (top) and agrinierite (bottom).

regard to the strong reflections, but differ in the presence of weak reflections, which modify the symmetry and the unit cell. The weak extra reflections are shown schematically in fig. 5; the systematic absences are in agreement with those found previously for billietite, compreignacite, and becquerelite (Christ and Clark, 1960; Protas, 1957, 1964). Billietite, compreignacite, becquerelite, and agrinierite are all orthorhombic and differ possibly in the stacking sequence of the layers, which may be controlled by the interlayer cations. The c -axis for all the compounds is approximately the same (14.1–15.1 Å), but the symmetry planes parallel to $\{001\}$ are different: billietite and compreignacite have an n -glide, becquerelite an a -glide, and agrinierite probably a mirror plane. Relative to billietite and compreignacite, which are the most symmetric, becquerelite has a doubled a -axis and cell volume, and agrinierite doubled a and b -axes and a quadrupled cell volume.

Rameauite, on the other hand, is monoclinic and the cell has been oriented in the conventional setting with the unique two-fold (pseudo-hexagonal) axis as $[010]$. The $h0l$ plane of rameauite in reciprocal space is very similar to the hko planes of the orthorhombic uranyl oxides.

Rameauite is identical morphologically with the unnamed orange mineral from Margnac (Protas, 1959), but is monoclinic, not orthorhombic, and contains no strontium; in addition, the strong powder lines of rameauite are doubled and the powder diagram differs in other details.

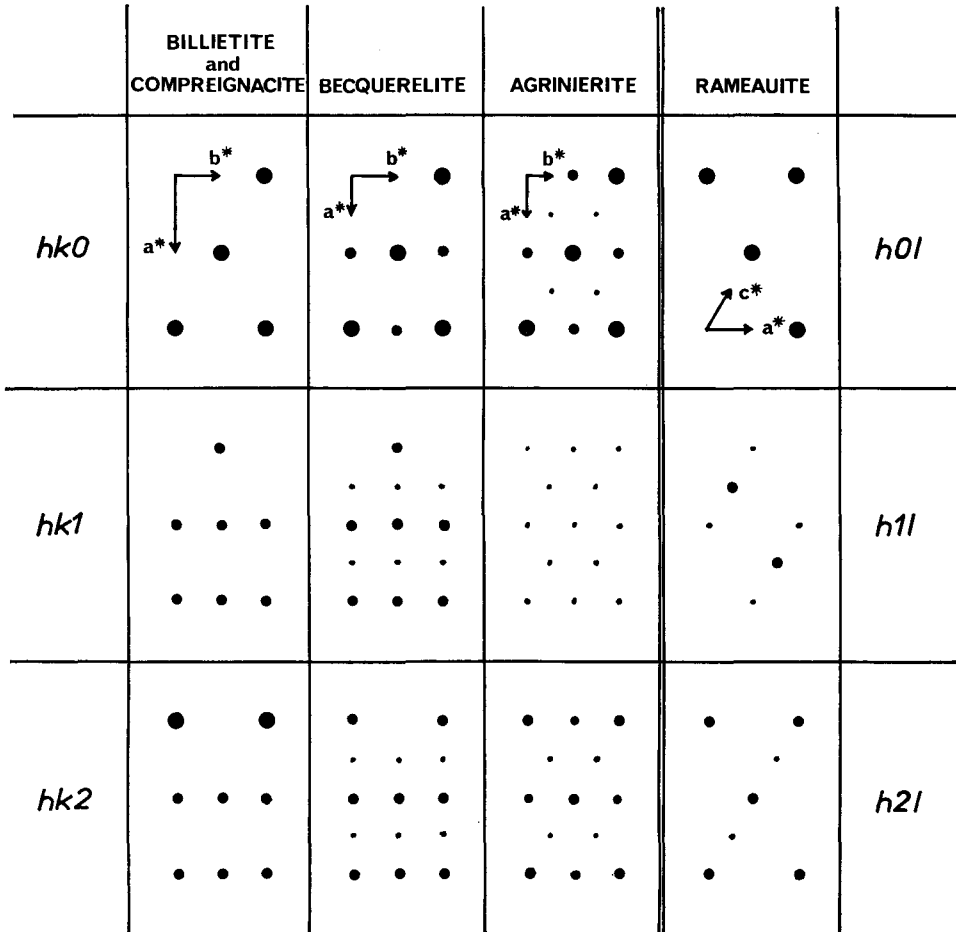


FIG. 5. Schematic representation of the reciprocal lattice planes normal to the pseudo-hexagonal axis for billietite, compreignacite, becquerelite, rameauite, and agrinierite. The sizes of the spots represent approximately the average relative intensities of the reflections for the layer, and not the intensities of the reflections nearest to the origin.

Compared with agrinierite, rameauite contains more potassium relative to calcium, and no strontium.

The X-ray powder photographs of agrinierite and rameauite resemble those of the synthetic triuranates rather than those of the hexauranates billietite, becquerelite, etc. (Peters, 1967); there are, however, important differences. The formulae for these

TABLE III. Comparison of hydrated uranyl oxides

	Billietite		Compreignacite		Becquerelite		Agrinierite		Rameauite	
	BaO · 6UO ₃ · 11H ₂ O		K ₂ O · 6UO ₃ · 11H ₂ O		CaO · 6UO ₃ · 11H ₂ O		2(K ₂ O, CaO, SrO)6UO ₃ · 8H ₂ O		K ₂ O · CaO · 6UO ₃ · 9H ₂ O	
	*	†	‡	§		†	¶		**	
<i>a</i> (Å)	7·14	7·14	7·15	7·16	13·83	13·86	14·04		13·97	
<i>b</i> (Å)	12·12	12·08	12·16	12·16	12·37	12·38	24·07		14·26	
<i>c</i> (Å)	15·07	15·10	14·82	14·88	14·92	14·96	14·13		14·22	
β	90°	90°	90°	90°	90°	90°	90°		121° 1'	
<i>V</i> (Å ³)	1303	1303	1288	1296	2553	2567	4773		2429	
<i>a'</i>	<i>a</i> 7·14	7·14	<i>a</i> 7·15	7·16	<i>a/2</i> 6·91	6·93	<i>a/2</i> 7·02		<i>c/2</i> 7·11	
<i>b'</i>	<i>b/3</i> 4·04	4·03	<i>b/3</i> 4·05	4·05	<i>b/3</i> 4·13	4·13	<i>b/6</i> 4·01		$\frac{1}{3}$ [201] 4·00	
<i>c'</i>	<i>c/2</i> 7·54	7·55	<i>c/2</i> 7·41	7·44	<i>c/2</i> 7·46	7·48	<i>c/2</i> 7·06		<i>b/2</i> 7·13	
Space groups	<i>Pnmm</i> <i>Pn2n</i>		<i>Pnmm</i> <i>Pn2n</i>		<i>Pnma</i> <i>Pn2a</i>		<i>Cmmm</i> <i>Cm2m</i> <i>Cmm2</i> <i>C222</i>		<i>C2/c</i> <i>Cc</i>	
<i>D</i> _{obs}	—	5·28	—	—	—	5·14	5·7		5·60	
<i>D</i> _{calc}	—	5·27	—	—	—	5·10	5·64		5·55	
<i>Z</i>	2		2		4		8		4	
Composition plane	(110)		(110)		—		(110)		(100)	
Cleavage	{001}		{001}		{001}		{001}		{010}	
2 <i>V</i> _α	35–7°		10–15°		30–2°		55°		32°	

* Katanga B.M. 1954, 173.

† Christ and Clark (1960).

‡ Margnac, France. n° 9105.

§ Protas (1964).

|| Katanga, n° 9100.

¶ Margnac, France. n° 9102.

** Margnac, France. n° 9103.

a', *b'*, and *c'* are the pseudo-orthohexagonal cell constants.

two new minerals are in agreement with the hypothesis that the water is present in the form of H_3O^+ and OH^- ions (Sobry, 1971), but the d.t.a. and t.g.a. curves do not clearly distinguish two episodes for the loss of water.

As can be seen from the following section, the paragenesis of these minerals is often complicated, and they sometimes closely resemble one another. Care was taken to ensure that the material used for morphological, X-ray and chemical studies all came from the same small area of a single hand specimen. In fact, other hand specimens have similar but not identical minerals, which are unlike any uranyl oxides hitherto described. It is our intention to study these probable new species in detail.

Occurrence. The Margnac deposit lies at the south-west end of the uranium province of Monts d'Ambazac (Haute Vienne, France). It consists of a series of NW.-SE. fractures cutting the granites and the leucogranites, the crush zones being more or less silicified, sometimes with a little baryte or fluorite, and impregnated with disseminated pitchblende. Accompanying minerals are rare, generally microscopic, and consist of iron sulphides, galena, sphalerite rare chalcopyrite, and exceptionally bismuthinite.

Locally these fractures cross elongate masses of 'episyenite', an albitized chloritized granite impregnated with crushed calcite and coloured red by hematite. Cavities occur where the quartz has been dissolved out. The mineralization reaches a maximum in such rocks, the cavities being filled by pitchblende and coffinite, which sometimes more or less completely replace albite. In such areas there may be several tons of uranium ore at 20 % or more uranium metal.

Zoned structures occur, consisting from the centre outwards of:

- a. A black nodule of 'gummite', sometimes with relict pitchblende accompanied rarely by ianthinite and schoepite.
- b. Red or orange 'gummite' with becquerelite, billietite, schoepite, compregnacite, agrinierite, and rameaute, the last being the most abundant complex uranyl oxide.
- c. Uranophane, sometimes massive, sometimes powdery and pale yellow, sometimes as remarkable crystals with calcite in fissures in the 'gummite'.
- d. Highly altered granite impregnated with limonite.

This oxidized zone rich in uranium minerals, which was exploited underground from 1953, has now been completely removed by open-cast working.

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