Nigerite from Lixa, near Felgueiras, Douro Litoral province, Portugal

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Summary: Nigerite occurs in pegmatite as hexagonal crystals enclosed in mica or in association with lithiophilite, apatite and vivianite. Absolute hexagonal parameters are a 5·67 and c 13·88 Å. Powder data are given. Forms $\{10\bar{1}3\}$, $\{10\bar{1}1\}$ and $\{10\bar{1}0\}$ present besides the well-developed {0001}. Density 4·25. Refractive index 1·80, birefringence about 0·005, negative optical character. The chemical composition is studied by means of chemical, X-ray fluorescence and optical spectrography methods.

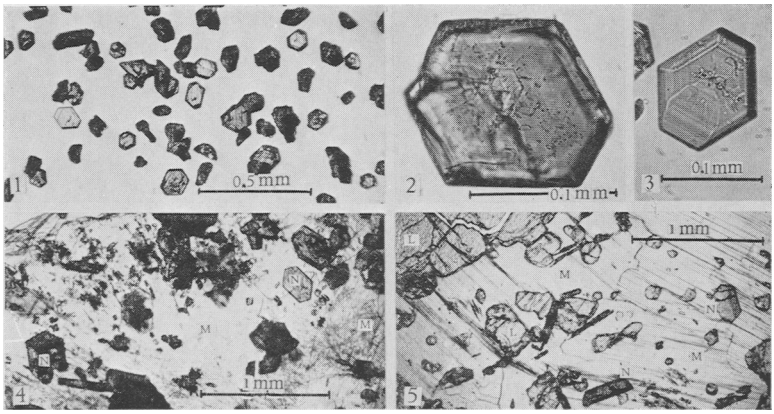
THE new tin–aluminium-bearing mineral, nigerite, was discovered by R. Jacobson and J. S. Webb (1947) in a quartz–sillimanite rock associated with pegmatite in the Kabba province, Nigeria, and described in detail by F. A. Bannister, M. H. Hey, and H. P. Stadler (1947). Another occurrence was reported from a pegmatite vein in eastern Siberia by A. I. Ginzberg, A. S. Nazarova, and L. L. Sukhomazova (1961). No other localities are known to the author's knowledge.

A new occurrence of nigerite is now reported from the village Lixa, conselho de Felgueiras, Douro Litoral province, Portugal, at the spot where the company 'Empresa minera de Caramos' worked a cassiterite-bearing pegmatite in the concession 'Tapada do Salão', 43 km ENE. of Porto. At this locality minute flattened hexagonal crystals of the mineral (figs. 1, 2, and 3) occur in pegmatite and are especially abundant as patches in white mica and in association with blue nests of phosphate minerals composed of lithiophilite, apatite and vivianite (figs. 4–9). The identification of these phosphates is based on X-ray powder photographs.

The hexagonal plates are 80–200 μ, max. 250 μ, in diameter, are 20 to 50 μ thick and macroscopically they show a brown-red colour, while their powder is yellow-brown. They are concentrated, after rough crushing, by electromagnetic and heavy liquid (methylene iodide) separations. A concentrate obtained in this way is contaminated by 12 % black grains almost entirely composed of iron-bearing rutile, as indicated by X-ray powder photographs and chemical tests (fig. 1). Careful and laborious hand-picking under the binocular microscope

provided a small sample (0·4 g) of pure material for the necessary analytical determinations.

The mineral here described shows properties identical, except for the optical character, with those recorded in the literature for nigerite from Nigeria and Siberia. It is felt that this difference does not justify distinguishing the Portuguese mineral from nigerite as a species, but makes it desirable to communicate its characteristics in full detail.



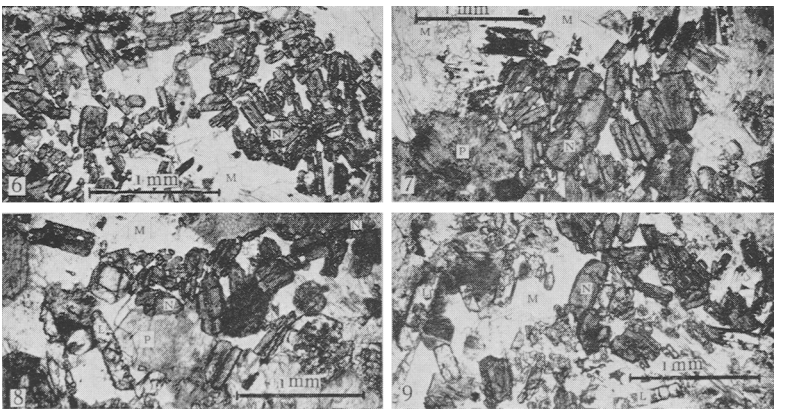
FIGS. 1–5. (1) Concentrate of nigerite and black grains of iron-bearing rutile. (2) Hexagonal crystal of nigerite, showing inclusions. (3) Hexagonal crystal of nigerite, showing zoning. (4) Cleavage fragment of white mica (*M*) with inclusions of nigerite (*N*) and rutile (?) needles. (5) Thin section, showing nigerite (*N*), white mica (*M*), and lithiophilite (*L*).

Very narrow prism and pyramid faces accompany the well-developed hexagonal face. Their good reflections, in spite of the small crystal dimensions, allow approximate readings on the two-circle reflecting goniometer to be made. These demonstrate the presence of a sixfold axis and the ρ values 41°, 69°, and 90° with the hexagonal face (0001) indicate respectively $\{10\bar{1}3\}$, $\{10\bar{1}1\}$, and $\{10\bar{1}0\}$ forms, when using the axial ratio given by F. A. Bannister, M. H. Hey, and H. P. Stadler (1947).

The density observed on pure grains by sinking and floating in Clerici solution, in the presence of test mineral fragments, is 4·2. Pycnometer measurements on 4 g impure material gave the value 4·25 which can only be affected very slightly by the presence (12 %) of iron-bearing rutile.

The hardness is difficult to establish with such small grains, but must lie above $5\frac{1}{2}$ as the crystals during crushing scratch glass slides.

Under the microscope the mineral appears transparent with yellow to brown colour. Empty (?) cavities, of a few μ, and fine zoning are displayed parallel to the hexagon edges (figs. 2 and 3). Irregular fracture cracks are observed. The minute grains do not show interference figures but it can be stated, without doubt, from inclined hexagons and universal stage observations that the mineral is uniaxial negative. Investigation



FIGS. 6–9. Thin sections with nigerite (*N*), white mica (*M*), lithiophilite (*L*), and phosphate (*P*).

of grains on the universal stage and measurement of their thickness, show that the birefringence is about 0·005. The refractive indices are high (about 1·80 according to the immersion method, using melts of piperine and iodides in the presence of standard mineral pieces). The crystals show a weak pleochroism with yellow along ϵ and yellow-brown along ω. The absence of fluorescence is noted.

The crystallography was also studied by means of powder, and two oriented single crystal, X-ray photographs from a 5·4 cm diameter camera using filtered Co-radiation. The absolute parameters found are a 5·67 and c 13·88 Å. The measured spacings by the powder method are given in table I.

The composition has been studied by different methods: chemical, X-ray fluorescence (XRF) and optical spectrographic analyses. Most determinations requiring only small quantities have been made, as indicated in table II, on pure material, while aluminium and the loss on

TABLE I. X-ray powder data for Portuguese nigerite

4·63	m	1·802	vw	1·228	w	1·110	vw	0·951	w
3·98	m	1·641	m	1·215	w	1·076	m	0·948	vw
2·82	s	1·545	m	1·188	vw	1·050	m	0·942	w
2·42	vs	1·430	m	1·160	vw	1·042	vw	0·931	m
2·17	w	1·413	w	1·148	vw	1·005	mw	0·930	w
2·01	w	1·354	vw	1·127	vw	0·982	vw	0·926	w
1·843	w	1·272	mw	1·119	vw	0·973	vw	0·914	vw

(Visual intensities: vs, very strong; s, strong; m, medium; w, weak; vw, very weak, from 5·4 cm diameter camera with filtered Co-radiation.)

TABLE II. Chemical analyses of nigerite

	Portugal		Nigeria (Bannister, *et al.*)	Siberia (Ginzberg, *et al.*)
Al_2O_3	55·0 %	(1)	50·91 %	57·68 %
SnO_2	23·2	(2)	25·33	19·10
Fe_2O_3 }	10·9	(2)	11·90	1·84
FeO }			2·65	4·13
ZnO	5·3	(2)	4·51	8·77
TiO_2	3·3	(3)	0·17	0·51
H_2O^+	1·2	(4)	1·57	1·37
SiO_2	0·9	(3)	0·48	4·19
Ga_2O_3	0·3	(5)	—	present
MgO	0·1	(5)	1·28	0·50
MnO	0·1	(5)	0·09	0·11
CaO	absent	(5)	not det.	1·05
PbO	< 0·1	(5)	0·94	absent
	100·3		99·83	99·25

(1) Chemical analysis (gravimetric and colorimetric) corrected for 12 % rutile.
(2) XRF, confirmed by optical spectrography, on pure sample.
(3) Colorimetry, carefully checked by optical spectrography, on pure sample.
(4) Loss on ignition at 1000° on impure sample.
(5) Optical spectrography on pure sample.

ignition had to be determined on impure material taking into account the contaminating 12 % rutile. Lack of sufficient pure sample and accessible methods suitable for small samples did not allow the determination of ferrous iron. The XRF was performed on beads prepared from a mixture of 10 g ignited borax, 2 g BaO_2 and 0·15 g pure material. A LiF-analyser was used for the analytical lines FeK_αN1, ZnK_αN1, SnK_αN1, and SnK_αN2 combined with discrimination technique. The ultraviolet spectrographic determinations were based on intensity comparison of analytical lines with those of Sn obtained by direct current arcing, on the cathode, of standard powder mixtures of oxides and finely ground isolated nigerite crystals. This method was also applied on a

small sample of nigerite from Egbe Distr., Kabba province, Nigeria, kindly provided by the British Museum (Natural History), and confirmed the analytical data for Fe, Ti, Zn, Mg, Mn, and Si already published for this substance completing them with 0·3 % Ga_2O_3.

The analytical results are given in table II and compared with the known data for Nigerian and Siberian nigerite.

Cd, V, Bi, As, Sb, Nb, and Ta were looked for by optical spectrography but were not found.

It is seen from the above descriptions that the properties of the Portuguese mineral fit satisfactorily, except the optical character, with those of nigerite from the other localities. Special care has been taken to check this behaviour and repeated observations confirmed the negative character of the Portuguese nigerite and the positive sign of the Nigerian mineral.

An explanation for the difference in optical character is lacking. It may be that the relatively high Ti-content of the Portuguese nigerite is responsible for the difference.

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