Ixiolite and niobian ixiolite: A new occurrence from Metapal pegmatite, Bastar District, Madhya Pradesh, India

We report here the occurrence of ixiolite associated with pegmatite within the pegmatitic belt of Metapal (lat. 18°52′, long. 81°33′). These are hosted by granites and basic intrusives like metagabbro and amphibolites exposed in Madhya Pradesh and adjoining areas of Orissa. They are emplaced into rocks of Bengpal group, belonging to lower Proterozoic age. The rocks comprise andalusite-sericite-biotite schist, gneisses, sericite quartzite and banded iron formation[1].

The pegmatite occurring in Metapal area is zoned, and found to be trending N 55°W–S 55°E with quartz as a core and perthite occurring in the intermediate and wall zone. The nature of border zone remained obscure due to intensive weathering of pegmatite. Quartz, feldspar, muscovite, beryl and spessartite garnet are the minerals found in the wall zone (Figure 1). Intermediate zone comprising of perthite composition is not continuously found all over the pegmatite. Massive quartz core is prominent in the south-western part. It is grey to smoky in colour. Rich mineralization of Nb–Ta minerals is confined at the margin of quartz core. Main Nb–Ta minerals found in the area are columbite–tantalite, ixiolite, euxenite, aeschynite, microlite, fersmite, occurring in association with cassiterite, beryl, monazite, zircon, ilmenite and magnetite.

During the evaluation stage of the pegmatite for columbite–tantalite resources, samples of the heavy minerals were analysed to know the concentration of Nb–Ta and Sn by wavelength dispersive X-ray fluorescence technique (WDXRF). This study indicated the presence of Nb, Ta and Sn in the sample. Few such individual Nb–Ta-bearing crystal samples, which were found to contain higher amount of Sn, were also examined by X-ray powder diffraction technique. On study, the mineral was identified as ixiolite. Pure fraction of mineral sample was obtained by crushing it to 100 mesh and subjecting it to heavy liquid (methylene iodide, sp. gr. 3.31) separation. Few mineral samples, which mineralogically characterized to be pure ixiolite, were sent for detailed chemical analysis. Comparison of the chemical analyses with published values (ref. 2, table no. 313, p. 466) and data from X-ray diffraction studies confirmed the presence of ixiolite. The *d* values and chemical results on the two pure ixiolite investigated are given in Tables 1–3.

The X-ray powder diffraction studies were carried out on microprocessor-controlled Siemens D-500 diffraction sys-

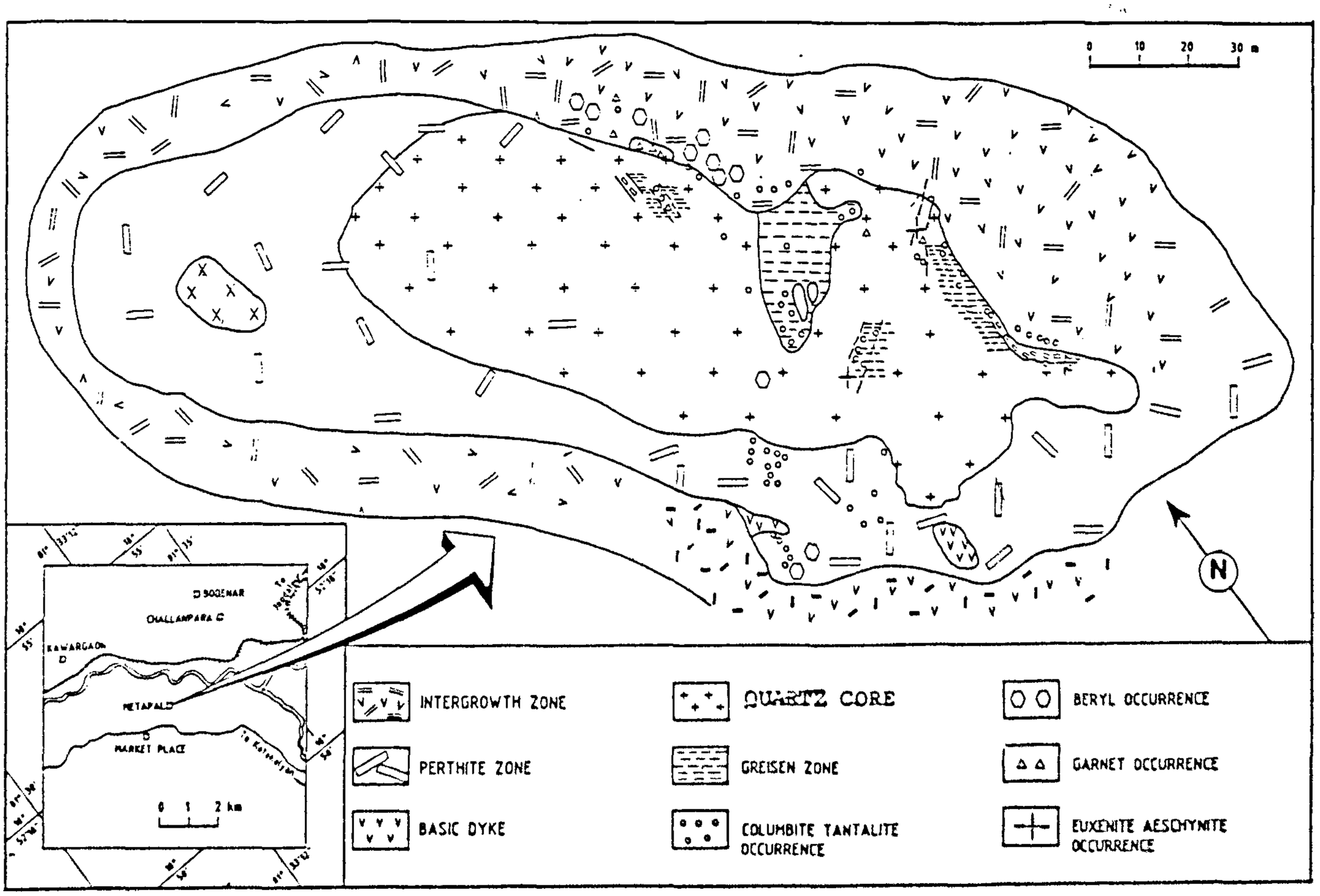

Figure 1. Geological map of Metapal pegmatite, Bastar District, Madhya Pradesh, India.

tem. using graphite crystal monochromated CuK alpha radiation with tube voltage selected at 35 kV and filament current maintained at 20 mA. Diffraction was carried out at a sample movement speed of one degree per minute and detector motion of two degree per minute in the angular range between 4° to 70° two theta. Recording of diffractogram was done on LA-100 digital recorder. The interplanar spacings obtained from both the samples closely match with those of ixiolite[3]. On heating the sample in a muffle furnace at 1000°C and rediffraction, ixiolite transformed to a phase showing interplanar spacings identical to wodginite[4-6], which confirmed the presence of ixiolite. The mineral fraction possessing different physical properties and associated with ixiolite separated at 0.45 A, on Frantz isodynamic separator was characterized to be microlite.

Table 1. *d* values of ixiolite, niobium ixiolite as compared with standard ixiolite given by Nickel *et al.*[3]

d (1)	*d* (2)	*d* (3)
3.6492	3.6545	3.65
2.9761	2.9775	2.98
2.8628	2.8708	2.87
2.5718	2.5592	2.57
2.4999	2.5027	2.51
2.3656	2.3686	2.37
2.2579	–	2.265
2.2077	2.2131	2.213
2.1005	2.0953	2.104
–	–	2.017
1.8214	1.9139	1.915
–	–	1.826
1.7688	1.7709	1.772
1.7403	1.7386	1.746
1.7169	1.5369	1.722
1.5469	1.5369	1.554
–	1.5224	1.521
1.4838	1.4845	1.490
1.4547	1.4558	1.459

1 and 2 are interplanar spacings for ixiolite and niobian ixiolite respectively from Metapal, Bastar District, M.P.
3. Interplanar spacings of ixiolite (Nickel *et al.*[3], table 5, p. 969, PDF card no. 15-733).

Table 2. Chemical composition of ixiolite and niobian ixiolite (all data in weight per cent)

	Sample no. 1	Sample no. 2	Sample no. 3
Nb_2O_5	11.7	58.8	10.5
Ta_2O_5	56.7	14.0	61.47
TiO_2	5.3	4.0	0.38
SnO_2	7.4	0.3	12.27
ZrO_2	1.0	<0.034	0.6
SiO_2	<0.2	<0.05	0.12
WO_3	0.9	1.6	0.3
MnO	6.7	5.4	5.4
FeO	5.4	11.9	8.08
Fe_2O_3	1.3	2.0	–
Al_2O_3	<0.3	0.2	0.16
CaO	0.49	0.5	0.11
MgO	<0.01	nd	–
U_3O_8	nd	0.168	–
Sc_2O_3	–	0.568	–

nd, not determined; Analysis by M. K. Ganguly, Chemical Laboratory, AMD, Hyderabad.
1 and 2, Ixiolites from Metapal pegmatite.
3, Ixiolite from Skogbole, Finland (Nickel *et al.*[3], Table 6, p. 970, PDF card no. 15-733).
Analytical techniques used: Nb_2O_5, Ta_2O_5, TiO_2, SnO_2, WO_3, ZrO_2 by ICP-AES method; Al_2O_3, Fe_2O_3, MnO, CaO, MgO by AAS method; U_3O_8 by fluorimetric method; FeO by titrimetric method; SiO_2 by photometric method.

The elemental analysis of the pure fraction of ixiolite was carried out using ICP-AES, AAS, spectrophotometry, fluorometry and gravimetric techniques. The chemical analyses of the two ixiolite samples are given in Table 2. Cerny and Ercit[7] while describing the ixiolitic group have quoted Nickel *et al.*[3] who stated that ixiolite is a complex oxide with chemical formula (Ta, Nb, Sn, Fe, Mn, Ti_2O_6) and having an orthorhombic unit cell, corresponding to that of disordered columbite–tantalite structure. In contrast to the latter, heating promotes ordering into the wodginite structure rather than ordered columbite–tantalite structure[7]. The present-day ixiolite mineral family represents a good number of compositional varieties, besides the type with Sn and Ta-rich compositions. Ixiolite with substantial Sc^{3+}, Fe^{2+}, Ti^{4+}, W^{6+}, besides Nb and Ta, have been described during last decades.

Nine samples from Metapal pegmatites were analysed by WDXRF method for only Nb_2O_5, Ta_2O_5 and SnO_2 contents (Table 3). These samples were also investigated by X-ray diffraction method and confirmed to be ixiolite (Table 1) which were subjected to chemical analysis (Table 2). Variations observed are Nb_2O_5, 5.81 to 58.8%; Ta_2O_5, 14.0–63.9% and SnO_2, 0.30 to 0.45% (Table 3).

The ixiolite from Metapal has a composition similar to the ixiolite from Skogbole, Finland so far as Ta_2O_5 and SnO_2 contents are concerned[7]. The TiO_2 content in Metapal ixiolite is relatively high. The analyses of ixiolite indicates interesting chemical variations in Nb_2O_5, Ta_2O_5 and SnO_2 contents. The ixiolite rich in Nb_2O_5 has been found in the area besides tantalian varieties. This suggests the presence of both niobian and tantalian varieties of ixiolites occurring in this pegmatite. The samples mineralogically found to be ixiolite get transformed to wodginite[4] on heating, but were chemically found

Table 3. Chemical analysis of ixiolite from Metapal, Bastar District, Madhya Pradesh

Sample no.	Analysis		
	Nb_2O_5	Ta_2O_5	SnO_2
MTP/92-93/28	6.63	59.26	9.39
MTP/92-93/34	5.92	60.79	10.45
MTP/92-93/35	5.81	61.72	10.25
MTP/92-93/49	5.8	63.9	7.9
MTP/92-93/Nb-Ta/68	14.3	52.7	8.1
MTP/92-92/Nb-Ta/72	16.00	52.4	8.8
MTP/92-93/Nb-Ta/73	11.3	58.4	8.0
MTP/92-93/Nb-Ta/75	10.60	60.3	7.9
OPS/MTP/93-94/7	58.8	14.00	0.3

to show different Nb_2O_5 and Ta_2O_5 contents (Table 2).

1. Babu, T. M., *Tin in India, Mineral Resources of India*, Geological Society of India, 1994, Series 7, pp. 96–99.
2. Vlasov, K. A., in *Geochemistry and Mineralogy of Rare Elements and Genetic Types of their Deposits, Mineralogy of Rare Elements*, 1966, vol. 2, pp. 397–567.
3. Nickel, E. H., Rowland, J. E. and Mc Adam, R. C., *Am. Mineral.*, 1963, **48**, 961–979.
4. Grice, J. D., Ferguson, R. B. and Hawthorne, F. C., *Can. Mineral.*, 1976, **14**, 540–549.
5. Ferguson, R. B., Hawthorne, F. C. and Grice, J. D., *Can. Mineral.*, 1976, **14**, 550–560.
6. Thomas Mulja, Anthoney E. Williams-Jones, Robert F. Martin and Scott A. Wood, *Am. Mineral.*, 1996, **81**, 146–157.
7. Cerny, P. and Ercit, T. S., in *Lanthanides, Tantalum and Niobium* (eds Moller, P., Cerny, P., Saupe, F.), Springer, Spl. Pub. No. 7 of the Society for Geology Applied to Mineral Deposits, 1989, pp. 28–79.

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Malachite-bearing shear zone mineralization in the supracrustals of Sadure hills, Sindhudurg District, Maharashtra

We report here the presence of an oxidized zone containing malachite (copper ore) in a Precambrian conglomerate of Saundal sequence exposed in the Sadure hills in the Sindhudurg district of Maharashtra.

The Saundal sequence consists of polymict conglomerate, grit, quartz mica schist, calcareous phylite and green schist, and is intruded by a metabasic rock.

The conglomerate consists of granules and pebbles of quartzite, vein quartz, basic rocks and granite set in a fine-grained matrix. Under the microscope, the matrix reveals the presence of quartz, calcite, biotite, sericite, chlorite and appreciable amounts of pyrite and other opaques. The conglomerate is conformably overlain by grit of similar composition.

An oxidized zone was noticed in the matrix of the conglomerate for a distance of nearly 50 metres on the Vaibhavwadi–Shirale section along southern face of the steep gorge of the Sukh river valley at an elevation of about 280 metres above MSL. Malachite is seen along the schistosity planes in the matrix of the conglomerate and grit. The malachite is dull green in colour and earthy in appearance. Occasionally tiny cavities with euhedral bright green malachite crystals were noticed. In view of the extent of oxidized zone with malachite encrustation, this occurrence needs further detailed examination for its economic significance.

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