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## NEW DATA ON MINERALOGY, PETROLOGY AND THE ORIGIN OF CALC–SILICATE ROCKS FROM NOVO SELO – KRIVA LAKAVICA, EASTERN MACEDONIA

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## Abstract

The paper presents the results of the latest research carried out on calc– silicate rocks from Novo Selo – Kriva Lakavica, Eastern Macedonia. One hundred samples were collected for research. Chemical, optical and X-ray analyses were performed. The results show that the dominant material in these rocks is calcite. The following minerals were also present: diopside-hedenbergite, augite, actinolite, pargasite, K-feldspar, plagioclase, quartz, titanite, vesuvianite, clinozoisite, andradite–grossular, scapolite and prehnite. These mineral associations and the regional distribution of calc–silicate rocks from Novo Selo – Kriva Lakavica indicate regional metamorphic conditions of origin. Such mineral associations have been described in many regional metamorphic complexes.

Key words: calc-silicate rocks, calcite, regional metamorphism

Introduction. The rocks investigated in this study are located in Eastern Macedonia, along the road that connects Stip and Radovis. Their colour varies from light gray to dark gray, depending on the presence of coloured minerals. Initial observations give an impression that these rocks are granitic. They are covered with Neogene and Paleogene sediments to the south, with Cretaceous, Paleogene and Neogene sediments to the east, and with alluvium from the Kriva Lakavica River to the west. North of Madenska River they are sparsely found, because they are covered with Neogene sediments or hacked with Jurassic granitoids. The main mass is almost continuous over a length of 4 km and is developed mainly on the right bank of the rivers of Kriva Lakavica and Madenska. The width of its largest part ranges from 50 to 100 m to the south and reaches more than 1 km in the northern part of the map. On the basis of regional and detailed research

performed by several different authors, the following geological formations have been established in the region between Novo Selo and Kriva Lakavica (Fig. 1).

The Mesozoic rocks comprise Jurassic ultrabasites, Jurassic and Cretaceous granitoids and Cretaceous sediments. Ultrabasites are present on both sides of Kriva Lakavica River. The mineralogical and petrological examinations of these rocks show that they are mostly dunites and harzburgites. Gabbro is present on the right side of Madenska River. Granitoid rocks are prevalent in this region and represent a direct continuation of the Stip granitoid massif. These granitoids have been dated at  $155 \pm 5$  million years [<sup>1</sup>] and  $143 \pm 7$  million years [<sup>2</sup>] before present time.

Unlike biotite granites, granosyenite occurs in thin dykes and veins that penetrate the metamorphic rocks and granodiorites. Cretaceous sediments are represented by conglomerate facies with transition to sand- and marlstone. The Paleogene period is represented by facies of conglomerates and facies of tuff-flysch sediments. The Neogene vulcanites – trachy–andesites form an independent magmatic massif, which has its own specific metalogeny and metamorphism. Neogene sediments occupy large areas on the left side of Kriva Lakavica. On the left bank of the river, they cover the complex of calc silicate rocks. Neogene sediments consist of gravel and sandy sediments. On the right bank of Kriva Lakavica, Neogene sediments are in the form of towels. Therefore, the majority of them are eroded and the underlying calc silicate rocks are visible over a large area.

Quaternary sediments are presented by delluvium and alluvium. The delluvium is relatively scarce and forms a thin arable layer. The alluvium is found along Kriva Lakavica and Madenska Rivers and its width ranges from 20 to 200 m.

Materials and methods. One hundred samples were collected for our investigation. The collection sites are shown on the geological map (Fig. 1). The chemical characteristics of these rocks were determined using the method AES–ICP at the Faculty of Natural and Technical Sciences of Stip, Macedonia. For more accurate detection and quantification of trace elements and rare elements, several control analyses were performed with ICP–MS at IGEM – Moscow, Russia. The optical and X-ray research was carried out at EOOD – Sofia, Bulgaria, at the Department of Geology of the Faculty of Science at the University of Zagreb, Croatia and at IGEM – Moscow, Russia.

**Results.** The data obtained for the chemical composition of the investigated rocks, presented in Table 1, show that the presence of oxides in all samples is almost identical, regardless of the sampling location, which indicates homogeneity of the rock.

Based on optical, chemical and X-ray examinations, the presence of the following minerals was established: calcite, diopside, augite, hornblende, actinolite, pargasite, andradite, grosular, K-feldspar, plagioclase, quartz, clinozoisite, titanite, vezuvianite, scapolite and prehnite. The chemistry of these minerals is given in Tables 2 and 3.



Fig. 1. Geological map of the Novo Selo – Kriva Lakavica (according to the geological map of the Republic of Macedonia, 1:100 000) with location of the calc silicate rocks. 1. Alluvial deposits; 2. Roughly clastic lake sediments; 3. Gravely sandy lake sediments; 4. Latites-trachy andesites; 5. Andesite, latite; 6. Andesite; 7. Volcanic tuffs; 8. Volcanic sediments; 9. Limestone; 10. Sandy, mudstone; 11. Granite and granodiorite; 12. Ultrabasite-serpentinite; 13. Calc silicate rocks; 14. Banding muscovite gneiss; 15. Silification; 16. Samples taken from wells NS-1, NS-2; 17. Sampling locations, marked in the text with V1, V2, V3, ...; 18. Sampling locations, marked in the text with T1, 2, 3, ...; 19. Sampling locations, marked in the text with V1, 2, 3, ...;

20. Sampling locations, marked in the text with Pr1, 2, 3, ...; 21. Samples R1, 2, 3, ...

	Ns-105	Pr-2	T-9	B-8	R-5	V-6	B-3	B-4
$SiO_2$	8.764	15.030	13.378	8.543	8.534	9.113	7.464	12.250
$\mathrm{TiO}_2$	0.247	0.226	0.283	0.084	0.126	0.179	0.151	0.265
$Al_2O_3$	3.847	5.750	3.680	2.699	2.564	3.366	1.790	4.450
FeO	1.397	1.866	1.860	1.140	1.066	1.408	1.312	2.287
MnO	0.047	0.072	0.055	0.053	0.084	0.048	0.076	0.064
MgO	1.317	1.746	1.665	0.767	1.143	0.951	1.019	1.059
CaO	47.030	45.017	44.580	49.220	44.570	46.940	49.99	45.890
Na <sub>2</sub> O	0.012	0.038	0.032	0.022	0.012	0.035	0.035	0.007
$K_2O$	1.611	0.293	0.969	0.546	0.442	1.106	0.739	0.272
$P_2O_5$	0.049	0.040	0.135	0.056	0.029	0.036	0.057	0.241
Loss of ignition	35.205	29.495	32.790	36.56	36.86	36.318	37.235	32.42
$H_2O$	0.089	0.202	0.148	0.056	0.188	0.104	0.027	0.105
Total	99.919	100.07	100.03	100.15	99.942	99.965	100.215	100.053
Sr	2531	481	1474	3420	2745	3039	2664	2466
Ba	127	31	81	69	101	126	80	39
Y	0.4	4	4	4	2	2	1	1
Cr	45	10	22	20	7	26	18	24
Cu	17	0.5	10	14	< 1	6	12	26
Zn	60	27	25	44	6	25	65	30
Pb	18	46	10	41	6	7	4	1
Ni	15	7	8	12	7	2	29	18
Co	28	< 1	1	16	< 0.1	24	23	23
In	17	3	3	12	8	11	18	18
Ga	15	32	128	14	26	18	16	28
Cd	1	< 1	2	< 1	< 1	< 1	<1	< 1
Bi	12	5	6	3	2	3	4	5
Zr	69	67						
Rb	36	9						
Nb	2	1						

Table 1

Chemical composition of the calc silicate rocks from Novo Selo – Kriva Lakavica

		n							n
	Cal-		Vesu-	Preh-		Diop-		Actino-	Parga-
	cite		vianite	nite		side		lite	site
~ ~	В-3	<i>a</i>	Pr-13	NS-105	<i>a</i> .e	Pr-12	~~~	Pr-12	Pr-12
CaO	54.72	$SiO_2$	36.98	43.67	$SiO_2$	48.71	$SiO_2$	39.35	39.00
$Al_2O_3$	1.2	$TiO_2$	1.91	0.13	$TiO_2$	0.74	$TiO_2$	1.84	1.94
FeO	0.267	$Al_2O_3$	15.71	23.64	$Al_2O_3$	5.69	$Al_2O_3$	14.55	14.72
MgO	0.397	FeO	7.20	0.44			$\mathrm{Fe}_{2}\mathrm{O}_{3}$	2.36	
Na <sub>2</sub> O	0.05	MnO	0.09		$\mathrm{Fe}_{2}\mathrm{O}_{3}$		FeO	13.52	16.03
$K_2O$	0.219	MgO	0.22	0.08	FeO	10.18	MnO	0.32	0.26
MnO	0.058	CaO	35.36	27.21	MnO	0.23	MgO	9.97	9.67
$\mathrm{TiO}_2$	0.050	Na <sub>2</sub> O		0.12	NiO		CaO	11.68	11.86
$P_2O_5$	0.002	$K_2O$	0.05		MgO	10.22	Na <sub>2</sub> O	1.91	2.04
Loss of ignition	42.93	Total	97.52	95.29	CaO	23.17	$K_2O$	2.62	2.59
Total	99.90		76(O)	6(O)	Na <sub>2</sub> O	1.03	$H_2O$	1.99	
$\mathbf{Sr}$	2803	Si	17.8		$K_2O$				
Ba	4.34	Al	0.2		Total	99.97	Total	100.11	98.11
Υ	2.89	Al	8.76	3.8	$\mathrm{Si}^{\mathrm{IV}}$	1.83	$\mathrm{Si}^{\mathrm{IV}}$	5.94	5.91
$\mathbf{Cr}$	< 0.1	Ti	0.69	0.02	$\mathrm{Al}^{\mathrm{IV}}$	0.18	$\mathrm{Al}^{\mathrm{IV}}$	2.06	2.09
Cu	< 0.1	Fe	2.9	0.05	T. pos	2.00	T. site	8.00	8.00
Zn	< 0.1	Mg	0.16	0.02	Al <sup>VI</sup>	0.08	$\mathrm{Al}^{\mathrm{VI}}$	0.53	0.54
Pb	16	Mn	0.04		Ti	0.02	$\mathrm{Fe}^{3+}$	0.27	0.18
Ni	1	Ca	18.32	4	$\mathbf{Cr}$		Ti	0.21	0.21
Co	< 0.1	Na		0.02	$\mathrm{Fe}^{3+}$		Mg	2.24	2.19
In	16	Κ	0.04		$\mathrm{Fe}^{2+}$	0.32	$\mathrm{Fe}^{2+}$	1.71	1.86
Ga	6				$\mathrm{Mn}^{2+}$	0.01	Mn	0.04	0.03
Cd	0.2				Ni		M1,2,3	0.04	5.00
Ag	< 0.1				Mg	0.57	Ca	1.89	1.93
					Ca	0.93	Na	0.11	0.07
					Na	0.08	M4site	2.00	2.00
					Κ		Na	0.45	0.53
					$M_1M_2$	2.00	Κ	0.51	0.50
							Ca	0.95	
					Ο	6	Asite		1.03
							Ο	22	23
							OH		2

Table 2	
Chemical composition of calcite, vesuvianite, prehnite, diopside, actinolit	te and pargasite

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	K-feld- spar Pr-4	Plagio- clase Pr-7		Clino- zoisite Ns-32		Garnets Pr-13	Tita- nite Pr-7		Scapo- lite Pr-4
$\mathrm{SiO}_2$	64.51	66.63	$SiO_2$	38.70	$SiO_2$	38.26	31.16	$SiO_2$	43.02
$\mathrm{Al}_2\mathrm{O}_3$	18.89	20.87	$\mathrm{TiO}_2$	0.30	$\mathrm{TiO}_2$	0.36	34.85	$\mathrm{TiO}_2$	0.06
$\mathrm{Fe}_2\mathrm{O}_3$			$Al_2O_3$	30.06	$Al_2O_3$	11.5	3.72	$Al_2O_3$	29.63
CaO	0.17	1.31	FeO		$\mathrm{Fe}_{2}\mathrm{O}_{3}$	15.84		FeO	0.28
$Na_2O$	0.92	10.95	MnO	0.06	FeO		0.58	MnO	
$K_2O$	15.12		MgO	0.24	MnO		0.07	MgO	0.24
BaO			CaO	23.33	MgO	0.09		CaO	20.44
Total	99.61	99.76	$Na_2O$	0.13	CaO	35.27	29.30	Na <sub>2</sub> O	1.68
$\mathrm{Si}^{4+}$	11.92	11.70	$K_2O$	0.02	Total	101.32	99.68	$K_2O$	0.26
$Al^{3+}$	4.11	4.32	Cl		$\mathrm{Si}^{\mathrm{IV}}$	6.01	4.07	Cl	0.04
$\mathrm{Fe}^{3+}$			SO		$\mathrm{Al}^{\mathrm{IV}}$	_	0.57	SO	0.13
$Ca^{2+}$	0.03	0.25			T. site	6.01			
Na	0.33	3.73	Total	98.09	$\mathrm{Al}^{\mathrm{VI}}$	2.13		Total	95.65
Κ	3.57		SI	2.98	$\mathrm{Ti}^{\mathrm{VI}}$	0.04	3.42	SI	
$\operatorname{Ba}^{2+}$			$\mathrm{Al}^{\mathrm{IV}}$	0.023	$\mathrm{Fe}^{3+}$	1.87		$\mathrm{Al}^{\mathrm{IV}}$	
Cations	19.96	19.99	T. site	3.00	Osite	4.039		T. site	
Н	16.04	16.02	$\mathrm{Al}^{\mathrm{VI}}$	2.70	Fe		0.06	$\mathrm{Al}^{\mathrm{VI}}$	
Z	3.93	3.98	Ti	0.02	Mn		0.01	Ti	
Ab	8.4	93.8	Mg	0.03	Mg	0.02		Mg	
An	0.9	6.2	Fe	0.31	Ca	5.93	4.10	Fe	
Or	90.7	0.0	Mn		Asite	5.95		Mn	
			Na	0.02	And	46.295		Na	0.52
			Ca	1.92	Gros	53.35		Ca	3.48
			Κ		Pyr	0.35		Κ	
			Sum. cat.	8.00	Spes			Sum. cat.	4
			Ο	13	0	24		0	
								Ma	13
								Me	87

T a b l e -3 Chemical composition of K-feldspar, plagioclase, clinozoisite, garnets, titanite and scapolite

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The matrix is composed of well-crystallized calcites and irregular round grains of other minerals. Calcite is most abundant (40 to 80%) and is present in the form of crystals ranging in size from 0.4 to 1.8 mm. The chemistry of calcite is shown in Table 2. Cell dimensions: a = 4.987 (2) Å; a = 4.987 (2) Å; c = 17.056 (8) Å;  $\gamma = 120^{\circ}$ ; V = 367.3 (2) Å<sup>3</sup>.

Pyroxene is in the Diopside group. Although it contains small inclusions of actinolite and albite, these do not occur in the basic mass. They can be interpreted as relics from earlier metamorphic stages, which progressively disappear during the prograde reaction Act + Cal + Ab  $\rightarrow$  Cpx + Scp + Pl<sub>(cal rich)</sub> + H<sub>2</sub>O. Diopside is formed according to the following reaction:

Ca<sub>2</sub>(Mg, Fe)<sub>5</sub>Si<sub>8</sub>O<sub>22</sub>(OH)<sub>2</sub> + CaCO<sub>3</sub> + SiO<sub>2</sub> = 5Ca(Mg, Fe)Si<sub>2</sub>O<sub>6</sub> + H<sub>2</sub>O + 3CO<sub>2</sub>. In some pyroxene grains the content of Fe and Al increases from the centre to the rim, while the content of Mg decreases. Cell dimensions: a = 9.745 (2) Å; b = 8.893 (2) Å; c = 5.286 (2) Å;  $\alpha = \gamma = 90^{\circ}$ ;  $\beta = 105.89$  (3)°; V = 440.6 (2) Å<sup>3</sup>.

Actinolite and pargasite are far less abundant than diopside. Most probably, they have undergone chemical disintegration. The absence of amphiboles can be interpreted by the fact that in many places, there are grains of epidote and coisite that look like a whole. The chemistry of actinolite and pargasite is shown in Table 2. Pargasite has a high potassium content (2.50-2.61%). From the centre to the rim, the content of Fe and Ca increases, while the content of Ti decreases.

K-feldspars occur in xenomorphic, relatively large grains with low kaolinization. The size of the grains varies from several millimetres to 2-3 cm. The chemistry of K-feldspar is shown in Table 3. Plagioclases are relatively scarce. They often show two, three or more types of twinning at one and the same location. Table 3 shows the chemistry of plagioclases. Cell dimensions: a = 8.178 (2) Å; b = 12.854 (3) Å; c = 7.110 (2) Å;  $\alpha = 93.58$  (2)°;  $\beta = 116.19(2)^{\circ}$ ;  $\gamma = 89.85$  (2)°; V = 669.1 (2) Å<sup>3</sup>.

Clinozoisites are rich in  $\text{Fe}^{3+}$  (0.31 the 0.38 pfu). Their chemistry is shown in Table 3. Clinozoisites are formed according to the following reaction:

Ca<sub>4</sub>Al<sub>6</sub>Si<sub>6</sub>O<sub>24</sub>CO<sub>3</sub> + H<sub>2</sub>O = 2Ca<sub>2</sub>Al<sub>3</sub>Si<sub>3</sub>O<sub>12</sub>OH + CO<sub>2</sub>. Cell dimensions: a = 8.882 (7) Å; b = 5.649 (7) Å; c = 10.201 (6) Å;  $\beta = 115.47$  (7)°; V = 462.1 (5) Å<sup>3</sup>.

Scapolites contain 77 - 86% Me-component, and are poor in Cl and SO<sub>4</sub>. Their chemistry is shown in Table 3. The values of K<sub>2</sub>O are very low (0.11 - 0.26 wt%). Scapolites are formed according to the following reaction:

 $3CaAl_2Si_2O_8 + CaCO_3 = Ca_4Al_6Si_6O_{24}CO_3.$ 

Cell dimensions: a = 12.183 (2) Å; b = 12.183 (2) Å; c = 7.540 (2) Å;  $\alpha = \beta = \gamma = 90^{\circ}$ ; V = 1118.9 (4) Å<sup>3</sup>.

Garnets are from the grossular–andradite series with an approximately equal content of andradite and grossular. The size of the grains is 0.9 - 1.2 mm. The garnets are characterized by zoning. The content of Si, Fe, Ti and Mg decreases from the centre towards the rim of the grains, while the content of Al and Mn

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increases. And radius is formed according to the following reaction:  $2\text{CaFeSi}_2\text{O}_6 + \text{CaCO}_3 + \text{H}_2\text{O} = \text{CaFe}_2\text{Si}_3\text{O}_{12} + \text{CO}_2 + \text{H}_2\text{O}$ . Grossular is formed according to the following reaction:

 $2\mathrm{Ca}_{2}\mathrm{Al}_{3}\mathrm{Si}_{3}\mathrm{O12OH} + 5\mathrm{Ca}\mathrm{CO}_{3} + 3\mathrm{SiO}_{2} = 3\mathrm{Ca}_{3}\mathrm{Al}_{2}\mathrm{Si}_{3}\mathrm{O}_{12} + \mathrm{H}_{2}\mathrm{O} + 5\mathrm{CO}_{2}.$ 

Titanite is present in typical wedge-shaped crystals. The chemistry of titanite is given in Table 3.

Vesuvianite is present in its iron variety. The content of Fe and Ti decreases from the centre towards the rim. There is a very slight decrease in the content of Mg. The chemical composition is given in Table 2. Vesuvianite is formed according to the following reaction:  $9Ca_3Al_2Si_3O_{12} + 6Ca(Mg, Fe)Si_2O_6 + 33Ca_2Al_3Si_3O_{12}OH = 3Ca_{18}Al_9Si_{18}O_{76} + 42CaAl_2Si_2O_8 + 3H_2O.$ 

Prehnite is present as aggregates in the form of beams which are connected in a nub. The size of the grains is approximately 0.3 mm. Their chemical composition is given in Table 2.

Quartz occurs in small round grains. Its content in the rocks is approximately 3%.

Metallic minerals are very poorly represented in these rocks and appear in very small individual grains of pyrite and limonite. They are not present in some of the samples.

**Discussion.** There are different opinions about the origin of the rocks investigated in the present study. They are considered as carbonatites by  $[^{3, 4}]$ , and as skarns by  $[^{5-7}]$ . Neither of these considerations is in agreement with the results of our investigation.

These rocks cannot be considered as skarns, because skarns are associated with narrow contact zones around the granite ranges. Typical skarns are contactreaction metasomatic rocks. They occur only in immediate contact with granite bodies and dykes and in the surrounding area. Skarns exhibit very specific zonality with sharp boundaries between zones. These rocks cannot be considered as carbonatites, because magmatic or volcanic carbonatites never appear as separate, individual massifs. They are always part of the multiple intrusive series of the mantle genesis, which include ultramafic and highly alkaline rocks. It seems that this association is not typical of the studied rocks. Plagioclase and scapolite are never present in carbonatites, while melilites crystallize instead. No carbonatite body contains Pl or Scp. Pl and Scp are the rock-forming characteristic minerals in all of our samples, whereas melilites are fully absent.

Apatite is always present in carbonatites in very large quantities. In our rocks, apatite is practically absent. Some very typical minerals of carbonatites are tetraferriphlogopite, pyrochlore, baddeleyite, REE-carbonates, Ti-magnetite, zirklerite and many other minerals rich in Zr, Nb, Y and REE. None of these mineral indicators of carbonatites were found in the studied rocks. Ti-garnet (schorlomite) is the most typical Grt of carbonatites. The investigated rocks contain only andradite–grossularite, almost Ti-free garnets. Vesuvianite is not stable in carbonatites due to high  $CO_2$  pressure, but it is stable in the researched rocks. Carbonatite rocks are characterized by an increased content of rare elements [<sup>8–11</sup>]. We measured the content of some of these elements which are very characteristic of carbonatites. The content of Sr, Y and Ba in carbonatites in various areas of the world (Y 64–167 ppm, Sr 3775–6200 ppm; Ba 917–2439 ppm) is significantly higher than in the rocks of Novo Selo the Kriva Lakvaica (Y 0.4–4 ppm, Sr 1474–3420 ppm; Ba 31–127 ppm).

The rocks investigated in the present study are located in the geotectonic unit of Vardar zone, which occupies the central part of the Republic of Macedonia. The rocks in the studied part of the Vardar zone have undergone regional metamorphism (Abukuma-type) to the degree of the amphibolite facies, with medium to high temperatures and low pressures  $[1^2]$ .

Abukuma-type metamorphism is associated with granitoid magmatism in the Valandovo–Stip–Bogdanci region [<sup>13</sup>]. This metamorphism has a NW–SE delineation and also stretches to the Trojaci metamorphic series of rocks. These facts are in line with the idea of the presence of paired metamorphic belts associated with zones of subduction, expressed by [<sup>2, 14</sup>].

The existence of paired metamorphic belts in the Vardar zone is also confirmed by [<sup>15</sup>]. The mineral association in Novo Selo – Kriva Lakavica and its regional distribution indicate regional metamorphic conditions. The mineral association present in the calc-silicate rocks from Novo Selo – Kriva Lakavica has been described for many regional metamorphic complexes. Practically all metamorphic associations from Novo Selo – Kriva Lakavica, including Kfs, Pl, Scp carrier associations and their textures, are characteristics of normal regional metamorphic calc–silicate rocks. This metamorphism has occured at greater depths and over a larger area to form metamorphic rocks away from the contact zone and therefore these rocks cannot be considered as skarn. According to the classification of metamorphic rocks given by [<sup>16</sup>], the rocks from Novo Selo – Kriva Lakavica are calc-silicate.

This association has the characteristics of cordierite mica-gneisses and its degree of metamorphism is associated with silimanite muscovite K-feldspar sub-facies, temperature of  $620 \,^{\circ}\text{C} - 650 \,^{\circ}\text{C}$  and pressure of 3.5-3.75 kbar. The characteristic minerals of the rocks from Novo Selo – Kriva Lakvica are typical of calc-silicate rocks in many regions.

**Conclusion.** After summarizing the data collected in this research, we can confirm that the researched rocks are calc–silicate. Based on optical, chemical and X-ray examinations, the presence of the following minerals was established: calcite, diopside, augite, hornblende, actinolite, pargasite, andradite, grosular, K-feldspar, plagioclase, quartz, clinozoisite, titanite, vesuvianite, scapolite and prehnite. This association has been described in many regional metamorphic complexes. The mineral association in Novo Selo – Kriva Lakavica and its regional distribution indicate regional metamorphic conditions of origin.

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