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## OLIVENITE-ADAMITE SOLID SOLUTION FROM OXIDATION ZONE IN RĘDZINY (WEST SUDETES, POLAND)

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**Abstract.** An extensive hydrothermal polymetallic mineralization with a well developed oxidation zone rich in secondary minerals occurs in dolostones several hundred meters from the Karkonosze granite at Rędziny. Using XRD and FTIR methods, mineral phases representing transitional members of the olivenite-adamite solid solution have been identified. Electron microprobe analyses reveal the most common varieties to be zincian olivenite and cuprous adamite with compositions ranging from  $(\text{Cu}_{1.17}\text{Zn}_{0.83})(\text{AsO}_4)(\text{OH})$  to  $(\text{Zn}_{1.38}\text{Cu}_{0.62})(\text{AsO}_4)(\text{OH})$ . The two minerals are subordinate in the weathering zone which can be characterized as having been a zone of low  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$  activity and with mineralizing solutions of increased pH. A high  $\text{Ca}^{2+}$  concentration due to the ubiquitous presence of carbonate rocks resulted in the expansion of the stability field of another arsenate, conichalcite (or Zn-bearing conichalcite), which is a common mineral there.

*Key-words:* olivenite-adamite, X-ray data, IR spectrum, chemical composition, Rędziny, Poland

### INTRODUCTION

Olivenite  $\text{Cu}_2(\text{AsO}_4)(\text{OH})$  and adamite  $\text{Zn}_2(\text{AsO}_4)(\text{OH})$  occur mainly in the oxidation zone of polymetallic deposits enriched in copper and zinc. Together with libethenite  $\text{Cu}_2(\text{PO}_4)(\text{OH})$  and eveite  $\text{Mn}_2(\text{AsO}_4)(\text{OH})$ , they belong to the adamite group (Strunz, Nickel 2001).

Olivenite and adamite form a solid solution as was proved by investigations of both natural and synthetic samples (Guillemin 1956; Minčeva-Stefanova 1964; Toman 1977; Williams et al. 2002). Other important elements in the olivenite-adamite solid solution include Fe, Co and Mn in cation sites and P replacing As. X-ray investigations of the  $(\text{Cu}_x\text{Zn}_{1-x})_2(\text{AsO}_4)(\text{OH})$  series show that zincian olivenite in which the content of the

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adamite end-member exceeds 20 mol.% is monoclinic and, below this value, orthorhombic (Toman 1977; Burns, Hawthorne 1995). Braithwaite (1983) discovered that the positions of some absorption bands in infrared spectra depend on the chemical composition of the olivenite-adamite series minerals and that, in the absence of cationic admixtures, an exact composition can be determined with IR analyses.

In Poland, Zn-olivenite has been described from Miedzianka near Kielce (Wieser, Żabiński 1986). In Lower Silesia, the mineral has been found on dumps at the old Frisch Glückstollen mine in Miedzianka (*vide* Lis, Sylwestrzak 1986) and, more recently, by Holeczek and Janeczek (1991). The presence of Zn-olivenite in Rędziny was noted in unpublished reports by Franus (1996) and Gołębiowska (2003).

#### GEOLOGICAL SETTING AND MATERIAL

The Rędziny dolostones, located within the schist series of the Kowary-Czarnów unit (Kozdrój 2003; Mazur 2003), occur within the eastern metamorphic cover of the Variscan Karkonosze granite. As a result of Hercynian movements, a dolostone lens was fragmented into several parts separated by NNW-SSE trending schist zones. These zones acted as conduits for hydrothermal solutions derived from the nearby Karkonosze granite intrusion and from which various ore minerals crystallized. In addition to the predominant arsenopyrite and cassiterite, the list of minerals includes chalcopyrite, pyrite, pyrrhotite, sphalerite, galena, Cu(Ag)-Pb-Bi(Sb) sulphosalts, and others. Weathering of the primary sulphides resulted in a wealth of secondary arsenates, vanadates, phosphates, carbonates and oxides (*e.g.* Franus 1996; Gołębiowska, Pieczka 1998; Gołębiowska 1999; Parafiniuk, Domańska 2002; Gołębiowska et al. 2002; Gołębiowska 2003; Pieczka et al. 2004, 2005).

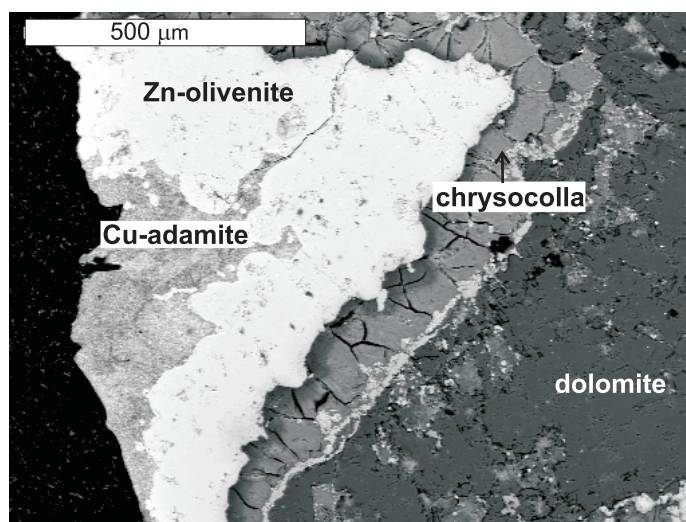


Fig. 1. Druse within dolostone filled with needles of Cu-adamite, homogeneous Zn-olivenite and chrysocolla. BSE image

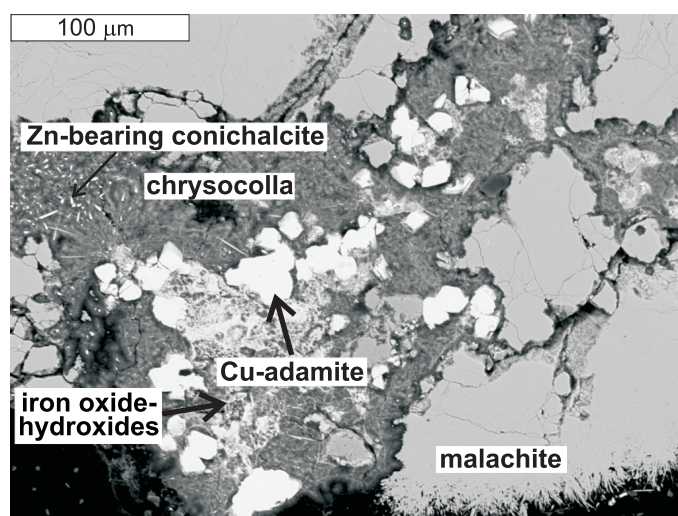


Fig. 2. Homogeneous crystals of Cu-adamite filling, with chrysocolla, iron oxide-hydroxides and fine needles of Zn-bearing conichalcite, a cavity within malachite. BSE image

Olivenite-adamite is a rare mineral at Rędziny. It occurs on the IIIrd exploitation level of the quarry in hydrothermally altered dolostones intersected by quartz veins with iron oxide-hydroxides and malachite. The olivenite-adamite is associated with other copper arsenates, i.e. strashimirite, cornwallite and clinotyrolite, Zn-bearing conichalcite and chrysocolla and hemimorphite. Traces of primary mineralization coexisting with olivenite-adamite commonly include relics of tennantite. The colour of the olivenite-adamite members depends on the ratio between Zn and Cu; Zn-rich varieties are white to pale green whereas those rich in Cu are apple green. They are developed as spherical accumulations of intergrowing crystals with a vitreous-silky lustre or as spherulitic enclaves in dolomite-quartz-hematite aggregates (Fig. 1). In some instances, they form micro-aggregates in fine fractures (Fig. 2) or occur as thin encrustations covering other minerals.

## METHODS

Minerals have been identified using X-ray diffraction (PHILIPS X'PERT diffractometer) under the following conditions: graphite-monochromatized  $\text{CuK}\alpha_1$  radiation ( $\lambda = 1.540562 \text{ \AA}$ , operating voltage 34 kV, operating current 30 mA, recording range  $4\text{--}60^\circ(2\theta)$ , scanning speed  $0.02^\circ(2\theta)/1\text{s}$ , quartz as the internal standard; and infrared spectroscopy (BIO-RAD FTS-165 spectrometer). Chemical analyses were carried out at the Inter-Institute Analytical Complex for Minerals and Synthetic Substances at Warsaw University using a Cameca SX-100 electron microprobe operating in the WDS mode (conditions: excitation voltage 15 kV, beam current 20 nA, peak count-time 20s, background time 10s). The following standards and spectral lines were used: As – InAs ( $L_{\alpha}$ , TAP), Mg – diopside ( $K_{\alpha}$ , TAP), Si – diopside ( $K_{\alpha}$ , TAP), Pb – galena

TABLE 1

X-ray powder diffraction data for olivenite-adamite sample from Rędziny

Zn-olivenite from Rędziny		Olivenite ICDD 4-657			Adamite ICDD 39-1353		
$d_{hkl}$	I	hkl	$d_{hkl}$	I (w)	hkl	$d_{hkl}$	I
1	2	3	4	5	6	7	8
5.951	53	$\bar{1}10$	5.94	58	110	5.949	30
					011	4.939	40
4.881	77	011	4.885	78	101	4.893	70
4.761	8	$\bar{1}00; 101$	4.816	63			
		020	4.307				
4.230	70	111	4.199	64	111	4.247	35
4.176					200	4.157	6
3.819	30	$\bar{1}20$	3.814	30			
3.791	20				120	3.793	20
3.736	20	$\bar{2}10$	3.710	9	210	3.733	20
3.483	5	021	3.482	1			
3.214	2	121	3.210	1			
3.196	4	$\bar{1}21$					
3.178	2	211					
3.118	3	$\bar{2}11$					
3.011	33				002	3.029	25
2.978	100	$\bar{2}20$	2.976	100	220	2.975	100
2.707	15	$\bar{1}30$	2.708	26	112	2.700	70
2.683	40				130	2.688	30
2.670		112			221	2.671	17
2.636	30	$\bar{1}12$	2.656	59	310	2.634	30
		$\bar{3}10$	2.612	20			
2.576	12	031	2.582	8	031	2.573	15
2.516	15	301			301	2.519	20
		$\bar{3}01$	2.491	14			
2.460	55	$\bar{1}31; 131$	2.464	52	022	2.469	65
					131	2.458	60
2.434	35	022	2.442	22	202	2.448	5
2.413	35	$\bar{2}02; 311$	2.409	38	311	2.415	65
		$\bar{3}11$	2.393	31			
2.352	20	122	2.340	22	212	2.352	40
2.335	4	212					
2.324		$\bar{1}22$	2.315	7	320	2.323	3
2.289		$\bar{2}12$					
2.191	14	231					
2.148	4	040	2.152	4			
2.116	4	222					

Table 1 cd.

1	2	3	4	5	6	7	8
2.084	4	$\bar{1}40$	2.082	1	400	2.077	6
2.014	3	041	2.022	1			
2.005	5	410;132			132	2.011	6
1.981	6	330	1.983	2	312	1.987	3
1.957	5	$\bar{1}41$	1.964	6	013;103	1.964	15
1.947	5				141	1.954	11
1.938	5	$\bar{3}12$					
1.930	3	103	1.929	4			
1.912	5	411			411	1.914	11
1.903	5	240					
1.888	5	$\bar{4}11$	1.897	5			
1.884	30	113	1.879	7	331	1.885	12
1.855	5	232	1.856	1			
1.847	7				232	1.855	6
1.838	5	$\bar{2}32$	1.826	3	322	1.844	2
1.804	6	$\bar{3}22;023$	1.798	2	241	1.810	2
1.786	2	421			421	1.785	1
1.762	2	$\bar{4}21$	1.771	1			
		$\bar{1}23;123$	1.756	1			
1.737	7	042	1.742	7	042	1.743	6
1.707	8	402	1.693		402	1.713	19
1.683	4	$\bar{1}50$	1.686	6			
1.664	5				223;150	1.671	15
1.656	10	$\bar{2}23;332$	1.649	10	332	1.660	35
1.639	5				033	1.646	8
1.631	5	033;341	1.628	3	303;510	1.631	19
1.621	7	151	1.621	12	341	1.627	13
1.616	5	$\bar{5}10; \bar{1}51$	1.617	3			
					133	1.615	13
1.607	15	242	1.605	17	151	1.611	13
					242	1.608	65
1.599	10	133; $\bar{2}42$	1.598	10	313;501	1.603	35
1.586	20	$\bar{1}33$			422	1.590	35
1.577	10	$\bar{3}13$	1.580	10	511	1.575	40
		$\bar{4}22$	1.575	23			
1.553	2	$\bar{5}11$	1.561	9			
1.548					520	1.548	2
		$\bar{5}20$	1.538	2	233	1.531	1
1.519	5	323	1.503				
1.506	10				004	1.514	40

(M<sub>α</sub>, PET), Bi – Bi<sub>2</sub>Te<sub>3</sub> (M<sub>α</sub>, PET), P – apatite (K<sub>α</sub>, PET), S – sphalerite (K<sub>α</sub>, PET), Cl – tugtupite (K<sub>α</sub>, PET), Ca – diopside (K<sub>α</sub>, TAP), Zn – sphalerite (K<sub>α</sub>, LIF), Cu – chalcocopyrite (K<sub>α</sub>, LIF), Fe – Fe<sub>2</sub>O<sub>3</sub> (K<sub>α</sub>, LIF), Mn – rhodonite (K<sub>α</sub>, LIF). About 50 points were analysed in sections of the polyminerale aggregates.

## RESULTS AND DISCUSSION

X-ray diffraction data of an olivenite-adamite sample are transitional when compared to the standard data for the Cu<sub>2</sub>(AsO<sub>4</sub>)(OH) and Zn<sub>2</sub>(AsO<sub>4</sub>)(OH) end-members (Table 1); this confirms the solid solution between the two minerals. IR analysis of the same sample of olivenite-adamite (Fig. 3) reveals the stretching vibrations (ν<sub>3</sub>) in the arsenate groups [AsO<sub>4</sub>]<sup>3-</sup> around 858 and 830 cm<sup>-1</sup> that are common to both Zn-olivenite and Cu-adamite. In the range 943–910 cm<sup>-1</sup>, there are visible bands of the same type that are characteristic of olivenite with no more than 20 mol.% of the adamite end-member. This is consistent with the data of Braithwaite (1983) who proved that, in olivenite containing more than 20 mol.% adamite, these bands shift towards lower wave numbers, i.e. to about 850 cm<sup>-1</sup>. Both the shape and positions of the absorption bands in the IR spectrum indicate that the sample from Rędziny is a member of the solid solution with a composition close to Olv<sub>60</sub>-Adm<sub>40</sub>. The absorption bands with frequencies 472 and 538 cm<sup>-1</sup> are the result of stretching vibrations (ν<sub>4</sub>) in the [AsO<sub>4</sub>]<sup>3-</sup>

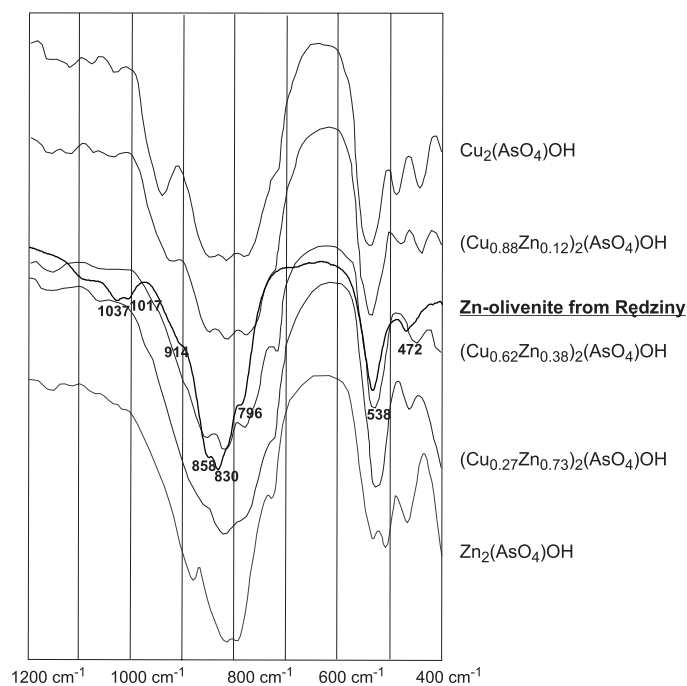


Fig. 3. The IR spectrum of Zn-olivenite from Rędziny compared with standard spectra of olivenite-adamite minerals (after Braithwaite 1983)

group, whereas those with frequencies 1035 and 1017  $\text{cm}^{-1}$  indicate an admixture of phosphate minerals or quartz.

The chemical formulae of all analyses were normalized to a cation (Zn + Cu + Fe + Mn + Ca + Pb + Bi) total of 2.0 in the respective formula units (Table 2).

TABLE 2  
Representative chemical composition of olivenite-adamite from Rędziny

	1	2	3	4	5	6	7	8	9	10	11	12
As <sub>2</sub> O <sub>5</sub>	40.62	40.20	39.85	40.26	40.94	40.50	40.92	39.58	39.22	39.67	38.43	40.48
P <sub>2</sub> O <sub>5</sub>	0.25	0.23	0.14	0.12	0.06	0.26	0.08	0.00	0.10	0.00	0.28	0.13
SiO <sub>2</sub>	0.30	0.11	0.14	0.11	0.04	0.14	0.06	0.00	0.00	0.00	0.00	0.00
SO <sub>3</sub>	0.03	0.00	0.03	0.00	0.08	0.02	0.00	0.00	0.32	0.11	0.27	0.24
ZnO	25.48	24.47	28.15	35.09	38.11	29.60	36.29	23.99	23.68	24.01	23.27	23.19
CuO	28.65	30.14	24.94	19.37	15.93	24.14	17.60	31.11	32.07	31.70	32.14	31.49
CaO	0.00	0.11	0.07	0.17	0.11	0.26	0.09	0.11	0.24	0.25	0.70	0.49
PbO	0.04	0.08	0.13	0.00	0.14	0.07	0.00	0.00	0.00	0.00	0.07	0.10
Bi <sub>2</sub> O <sub>3</sub>	0.19	0.00	0.13	0.21	0.26	0.13	0.07	0.26	0.38	0.00	0.00	0.00
Fe <sub>2</sub> O <sub>3</sub>	1.04	0.75	1.79	0.84	0.69	0.81	0.96	0.12	0.09	0.16	0.02	0.08
MnO	0.18	0.14	0.05	0.10	0.12	0.30	0.30	0.04	0.05	0.07	0.24	0.01
Cl	0.07	0.04	0.00	0.03	0.00	0.05	0.00	0.60	0.00	0.00	0.00	0.00
H <sub>2</sub> O	2.83	3.03	3.08	3.01	2.73	2.87	2.86	3.11	3.33	3.31	3.59	2.85
Total	99.69	99.30	98.49	99.30	99.21	99.16	99.23	98.92	99.48	99.29	99.00	99.06
Zn + Cu + Fe + Mn + Ca + Pb + Bi = 2.0												
(AsO <sub>4</sub> ) <sup>3-</sup>	1.02	1.01	1.01	1.01	1.04	1.02	1.03	0.99	0.97	0.98	0.94	1.02
(PO <sub>4</sub> ) <sup>3-</sup>	0.01	0.01	0.01	0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01
(SiO <sub>4</sub> ) <sup>4-</sup>	0.05	0.01	0.01	0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
(SO <sub>4</sub> ) <sup>2-</sup>	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01
Zn	0.90	0.87	1.01	1.25	1.37	1.05	1.29	0.85	0.83	0.84	0.81	0.85
Cu	1.04	1.09	0.92	0.71	0.59	0.88	0.64	1.13	1.15	1.14	1.14	1.15
Ca	<0.01	0.01	<0.01	0.01	0.01	0.01	<0.01	0.01	0.01	0.01	0.04	0.03
Pb	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Bi	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Fe	0.04	0.03	0.07	0.03	0.03	0.03	0.04	<0.01	0.003	0.01	0.001	0.003
Mn	0.02	0.01	<0.01	0.01	0.01	0.03	0.02	<0.01	<0.01	0.01	0.02	<0.01
Cl	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	0.01	0.01
OH	0.91	0.97	0.99	0.97	0.86	0.92	0.92	1.00	1.05	1.05	1.12	0.95
	<i>Ol</i>	<i>Ol</i>	<i>Ad</i>	<i>Ad</i>	<i>Ad</i>	<i>Ad</i>	<i>Ad</i>	<i>Ol</i>	<i>Ol</i>	<i>Ol</i>	<i>Ol</i>	<i>Ol</i>

Ol – olivenite; Ad – adamite



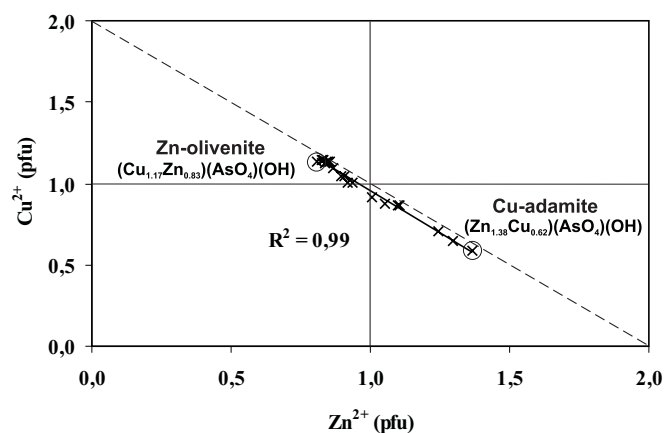


Fig. 4. Cu↔Zn substitution trend in solid solutions of olivenite and adamite

The amounts of H<sub>2</sub>O were calculated by stoichiometry. As the compositions vary between (Cu<sub>1.17</sub>Zn<sub>0.83</sub>)(AsO<sub>4</sub>)(OH) and (Zn<sub>1.38</sub>Cu<sub>0.62</sub>)(AsO<sub>4</sub>)(OH), the sample of olivenite-adamite from Rędziny contains from 59 to 31 mol.% of the olivenite end-member. No pure copper or zinc end-members were found. The trend of Cu↔Zn replacement is plotted in Figure 4. Trace elements detected by EMPA are: Ca (<0.01–0.04 apfu), Mn (<0.01–0.03 apfu), Fe (<0.01–0.07 apfu), Pb (<0.01 apfu), Bi (<0.01 apfu), P (<0.01–0.01 apfu) and S (<0.01–0.01 apfu). The mean OH content of 0.97 pfu is close to its theoretical value of 1.00.

## CONCLUSIONS

The EMPA of the olivenite-adamite solid solution from Rędziny indicate the presence of transitional members with more than 20 mol.% Zn<sub>2</sub>(AsO<sub>4</sub>)(OH) in their composition. Such members crystallize as monoclinic minerals.

X-ray diffraction and IR analyses carried out on larger aggregates of the olivenite-adamite have proved the predominance of copper-rich varieties. Only in a few EMPA does Zn prevail over Cu. This means that the solutions in the oxidation zone contained higher concentrations of Cu<sup>2+</sup> compared to Zn<sup>2+</sup>; this assumption is corroborated by the fact that chalcopyrite or tennantite are relatively common at Rędziny, whereas sphalerite has been recorded only in minor amounts. An alternative explanation for the low Zn concentration involves the behaviour of this element in oxidation zones. Zinc is mobile and easily migrates even in neutral solutions; thus, this element became dispersed in the carbonate environment of Rędziny. Experimental conditions for the precipitation and dissolution of arsenates (Magalhães et al. 1988) indicate that the formation of these minerals is accompanied by an increase in pH and a decrease in activity of Me<sup>2+</sup> ions. Elevated Ca<sup>2+</sup> activity, due to the presence of carbonate rocks, would have tended to inhibit the precipitation of simple Cu- or Zn-arsenates. Such conditions would have extended the thermodynamic stability field of conicalcrite,



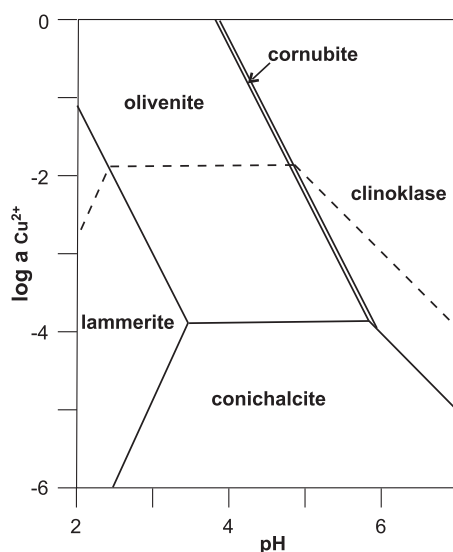


Fig. 5. Stability field diagram for copper (II) arsenate minerals (after Magalhães et al. 1988)

$\text{CaCu}(\text{AsO}_4)\text{OH}$ , or Zn-bearing conichalcite,  $\text{Ca}(\text{Cu,Zn})(\text{AsO}_4)\text{OH}$ , and reduced the likelihood of olivenite, strashimirite or adamite crystallization (Fig. 5). Therefore, these three minerals are rare in the oxidation zone at Rędziny. In comparison, conichalcite is very common there.

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**ROZTWÓR STAŁY POMIĘDZY OLIVENITEM A ADAMITEM  
ZE STREFY UTLENIEŃ W RĘDZINACH  
(SUDETY ZACHODNIE, POLSKA)**

Streszczenie

W dolomitach rędzińskich występuje obszerny, hydrotermalny zespół mineralizacji polimetalicznej oraz równie bogaty zespół mineralizacji wtórnej. Metodami XRD i FTIR zidentyfikowano minerały będące ogniwami pośrednimi pomiędzy olivenitem a adamitem tworzącymi ze sobą roztwór stały. Na podstawie analizy w mikroobszarze

(WDS) określono skład chemiczny poszczególnych członów. Najczęściej spotykanymi odmianami są cynkowy olivenit i miedziowy adamit o następujących skrajnych składach chemicznych:  $(\text{Cu}_{1,17}\text{Zn}_{0,83})(\text{AsO}_4)(\text{OH})$  i  $(\text{Zn}_{1,38}\text{Cu}_{0,62})(\text{AsO}_4)(\text{OH})$ . Niewielki udział olivenitu i adamitu w strefie utlenienia w Rędzinach związany jest z mniejszą aktywnością jonów  $\text{Cu}^{2+}$  i  $\text{Zn}^{2+}$  w roztworach mineralizujących i podwyższeniem pH środowiska. Obecność skał węglanowych w Rędzinach i duże stężenie jonów  $\text{Ca}^{2+}$  wpływa korzystnie na rozszerzanie termodynamicznego pola trwałości innego arsenianu, konichalcytu lub też jego cynkowej odmiany i w konsekwencji powszechną krystalizację tego minerału.