

## CONGOLITE AND TREMBATHITE FROM THE KŁODAWA SALT MINE, CENTRAL POLAND: RECORDS OF THE THERMAL HISTORY OF THE PARENTAL SALT DOME

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### ABSTRACT

Pseudocubic crystals of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate,  $\leq 1.2$  mm in size and yellowish through pale-violet to pale-violet-brown and brownish in color, were found in the Kłodawa salt dome, located within the Mid-Polish Trough (central Poland), an axial part of the Polish basin belonging to a system of Permian-Mesozoic epicontinental basins of Western and Central Europe. The crystals occur in adjacent parts of the Underlying Halite and Youngest Halite units bordering the Anhydrite Pegmatite unit around the PZ-3/PZ-4 (Leine/Aller) boundary. The internal texture of the crystals reflects phase-transitions from rhombohedral congolite (space-group symmetry  $R3c$ ) through orthorhombic ericaite ( $Pca2_1$ ) to a cubic, high-temperature, mainly Fe-dominant analogue of boracite ( $F43c$ ), indicating formation of the phases under increasing temperature. Currently, the parts of the crystals showing orthorhombic and cubic morphology generally represent congolite and, much less frequently, trembathite paramorphs after higher-temperature structural varieties of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  and  $(\text{Mg,Fe,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$ , which are unstable at room-temperature. The phase-transitions  $R3c \rightarrow Pca2_1$  and  $Pca2_1 \rightarrow F43c$  recorded in the morphology of the growing crystals took place at temperatures of 230–250 °C and 310–315 °C, respectively, whereas the total range of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate crystallization was determined to be ca. 100 °C to 350–360 °C. The primary congolite crystallized as a result of the metasomatic reaction of Fe and Mn carriers present within the host rock-salts of the Underlying Halite and Youngest Halite with  $(\text{BO}_3)^{3-}$ -bearing brines and fluids released from primary evaporate sediments (probably mainly from Upper Younger Halite with potash impurities of the PZ-3 cyclothem) in diagenetic and metamorphic processes induced by disturbances in halokinetic stability, intensive faulting of the pre-Zechstein basement and decompression, and, connected with movements of evaporates, their upward flow in the crust and the formation of a diapiric structure. The phase-transitions  $R3c \rightarrow Pca2_1$  and  $Pca2_1 \rightarrow F43c$  have been forced by inflows of Mg-bearing fluids sourced from relatively thick loads of potash salts (Younger Potash) of the PZ-3 cyclothem, containing inclusions of carnallite, kainite, and kieserite. The brines and fluids have utilized cracks in Pegmatite Anhydrite as convenient pathways for migration in the dome, in contrast to the host rock-salts, which are too solid to be penetrated by the fluids for distances greater than a few meters.

**Keywords:** congolite, trembathite, phase-transitions, thermal history, Kłodawa dome, Permian–Mesozoic Polish Basin

### INTRODUCTION

The boracite group contains five minerals: boracite,  $\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$ , ericaite,  $\text{Fe}_3\text{B}_7\text{O}_{13}\text{Cl}$ , and chambersite,  $\text{Mn}_3\text{B}_7\text{O}_{13}\text{Cl}$ , crystallizing with space-group symmetry  $Pca2_1$ , and congolite,  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$ , and trembathite,  $(\text{Mg,Fe})_3\text{B}_7\text{O}_{13}\text{Cl}$ , crystallizing with space-group symmetry  $R3c$ . Boracite is known as an accessory mineral in marine evaporates, but occurrences of the other minerals are much less frequent in

nature. Ericaite is known mainly from the Zechstein salt deposits of Germany (Kühn & Schaacke 1955, Heide & Völksch 1979, Heide *et al.* 1980, Horst 1989); it also has been found in the Venice salt dome, Plaquemines Parish, Louisiana, USA (Smith 1994). Chambersite was first found in the Barber Hill salt dome, Chambers County, Texas, USA (Honea & Beck 1962), and next in the Darrow salt dome, Ascension Parish, Louisiana, USA; in the Lake Hermitage and Venice salt domes, Plaquemines Parish, Louisiana, USA (Smith 1994); in

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two chemical-sedimentary B-Mn-Fe deposits in China: the Zhongxincun deposit, Pinggu District, Beijing, and the Dongshuichang deposit, Jixian, Tijian (Fan *et al.* 1999); and in the Penobscquis potash deposit, Canada (Grice *et al.* 2005). Congolite was first described from Mesozoic salts of the Kouilou Department in the Republic of Congo, (Brazaville) (Wendling *et al.* 1972), and then found only in the Penobscquis and Millstream evaporite deposits of southern New Brunswick, Canada (Roulston & Waugh 1981, Burns & Carpenter 1996, Grice *et al.* 2005), and in the Boulby potash mine, Loftus, Cleveland, England (<http://www.mindat.org/>). Trembathite was first found in the Salt Springs deposit, and then in the Penobscquis and Millstream deposits, all located in New Brunswick, Canada (Burns *et al.* 1992, Burns & Carpenter 1996, Grice *et al.* 2005), and in the Boulby potash mine, England (Schindler & Hawthorne 1998).

Burns & Carpenter (1996, 1997) explained in detail the phase relations of the boracite-group minerals in terms of Fe/Mg ratio and temperature. At room temperature, boracite exists as an orthorhombic phase up to an Fe/(Fe + Mg) ratio close to 0.36, when a phase-transition related to the change of the orthorhombic structure to rhombohedral takes place. Rhombohedral trembathite,  $(\text{Mg,Fe})_3\text{B}_7\text{O}_{13}\text{Cl}$  (always with  $\text{Mg} > \text{Fe}$ ), is stable from  $\sim 0.36$  to 0.50; above 0.50 rhombohedral congolite,  $(\text{Fe,Mg})_3\text{B}_7\text{O}_{13}\text{Cl}$  (always with  $\text{Mg} < \text{Fe}$ ) is the stable phase. With increasing temperature,

trembathite and congolite change their rhombohedral structure to orthorhombic, typical of boracite, and transform to high-temperature boracite with  $\text{Mg} > \text{Fe}$  and ericaite with  $\text{Fe} > \text{Mg}$ . At still higher temperatures, all the minerals undergo a phase transition to the cubic boracite structure (*F43c*). Boundaries between the phases with rhombohedral, orthorhombic, and cubic structure are linear and depend on the Fe/Mg ratio (Burns & Carpenter 1996). In temperature – composition space, the boundary for the *F43c*  $\leftrightarrow$  *Pca2<sub>1</sub>* transition is determined by 330 °C, as the highest temperature of the transition in pure  $\text{Fe}_3\text{B}_7\text{O}_{13}\text{Cl}$ , and has a slope of  $-7.1$  °C per 10 mol.%  $\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$ . For the *Pca2<sub>1</sub>*  $\leftrightarrow$  *R3c* transition the boundary is determined by 25 °C for the phase with 36 mol.%  $\text{Fe}_3\text{B}_7\text{O}_{13}\text{Cl}$  and a slope of about 45 °C per 10 mol.%  $\text{Fe}_3\text{B}_7\text{O}_{13}\text{Cl}$  (Burns & Carpenter 1996).

In this paper, we describe the first occurrence of congolite and trembathite in the Permian-Mesozoic basins of Central Europe, from the Kłodawa salt dome in central Poland. We focus attention on aspects of the compositional and morphological evolution of the minerals during growth that reflect the thermal history of the parental salt dome.

#### GEOLOGICAL SETTING

The Upper Permian sedimentary basin in central Poland constitutes the eastern part of the Permian-

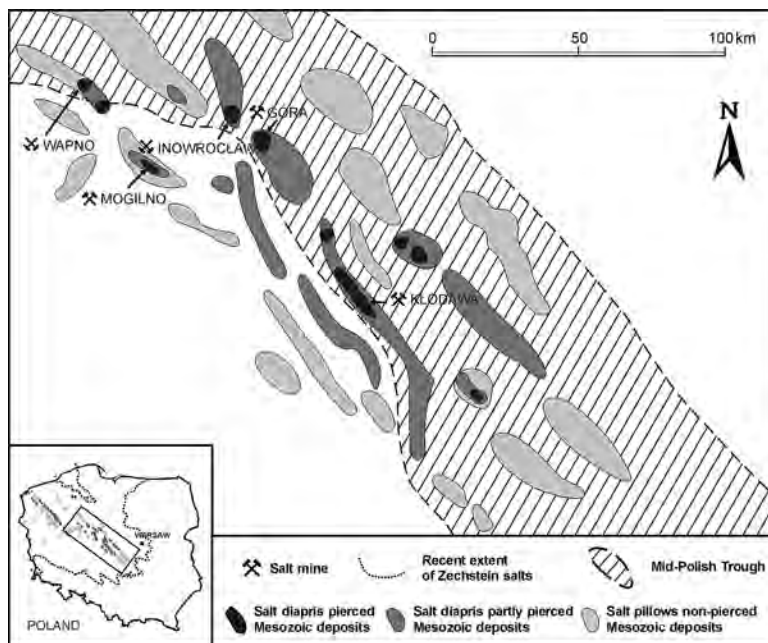


FIG. 1. Salt structures in the central part of the Mid-Polish Trough.

Mesozoic epicontinental basins of Western and Central Europe. In the basin, carbonate–evaporite sequences with potash salts form the Werra (PZ-1), Stassfurt (PZ-2), and the lower part of the Leine (PZ-3) cyclothem, whereas the upper part of the PZ-3 and Aller (PZ-4) cyclothem are composed of terrigenous–evaporate sequences. The total thickness of the Zechstein evaporate sediments in central Poland varies from 180 to 2800 m (Garlicki & Szybist 1986). The Mid-Polish Trough represents the axial part of the

Polish basin, evolving from Permian to Cretaceous times along the NW-SE trending Tornquist-Teisseyre Zone, with evaporate deposits reaching a thickness of ca. 1500 m (Ziegler 1990, Wagner 1994, Dadlez 1997, Kutek 2001) and creating locally numerous salt pillows and domes as a result of halokinetic and tectonic processes (Trusheim 1960, Krzywiec 2004) (Fig. 1). Some of them, especially those that penetrated the Mesozoic cover and form shallow occurrences, have been exploited in the past or are mined currently. They

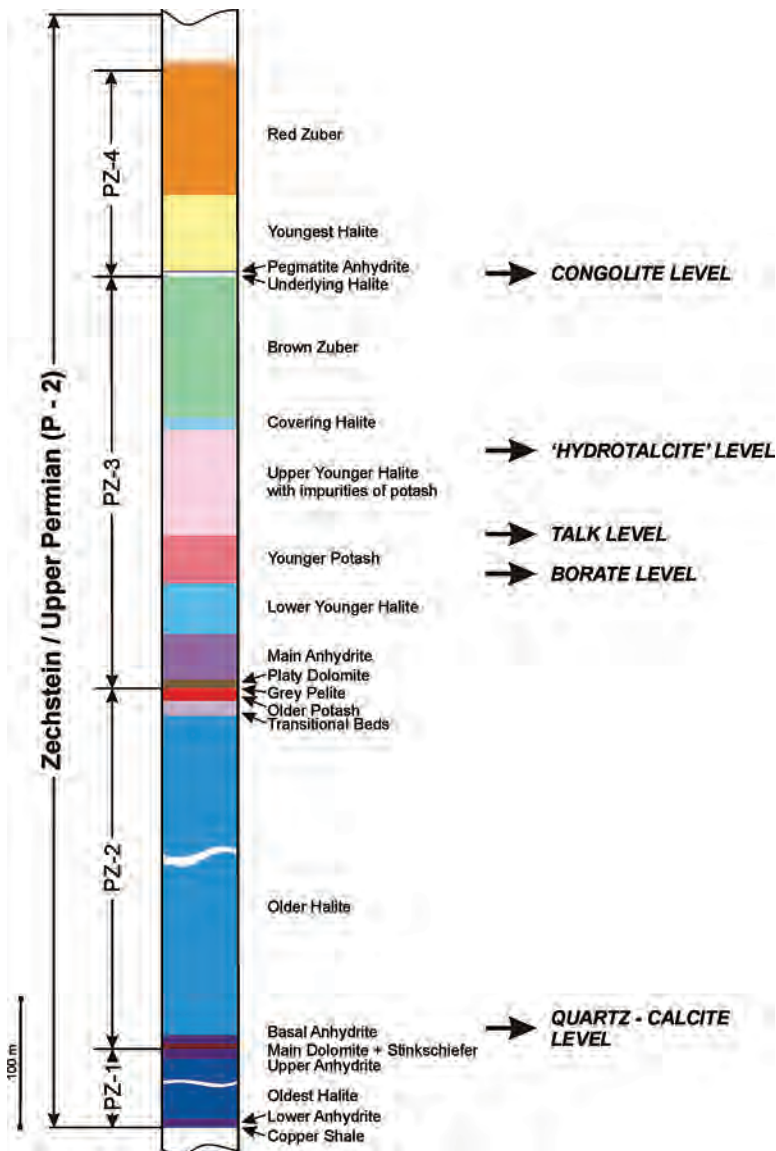


FIG. 2. A profile of the Kłodawa salt dome.

include the following domes: Góra and Mogilno with borehole exploitation of salts, Inowrocław and Wapno with abandoned underground salt mines, and Kłodawa with an active underground salt mine, reaching 750 m in depth. The Kłodawa Salt Mine is located within the Kłodawa dome in the central part of the Mid-Polish Trough. The dome constitutes one of the largest salt structures within the Polish basin, almost 30 km long and 6 km high. All the domes are formed by thick loads of rock-salts intercalated with layers of poor potash-salts and carbonate-sulfate-clay rocks (Fig. 2).

#### BORATE MINERALS IN THE POLISH SALT DOMES

Generally, two borate minerals, boracite ( $\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$ ) and szaibélyite ( $\text{MgBO}_2\text{OH}$ ), have been recognized in the domes to date. Boracite has been found in the Inowrocław, Kłodawa, and Mogilno domes. In the Inowrocław dome, the mineral occurs as spherical microcrystalline aggregates of orthorhombic form, with sizes ranging from some millimeters to almost 30 cm, disseminated within the kieserite–carnallite salts of the PZ-2 cyclothem (Poborski *et al.* 1956, Prochazka 1957, Fijał 1973), and as epigenetic paramorphs of the low-temperature orthorhombic form after the higher-temperature cubic form (Cybulski 1974), found in strongly tectonically disturbed anhydrite of the PZ-3 and PZ-4 cyclothem. The boracite crystals are translucent to transparent, greenish, rarely bluish and colorless, with sizes up to 10 mm. In the Kłodawa dome, boracite was found within the Younger Salt unit of the PZ-3 cyclothem (Fig. 2), in the floor of the kieserite–carnallite layer (Hanczke 1969, Fijał 1970, 1973, Wachowiak 1998). The boracite forms colorless, translucent to transparent crystals that reach up to 3 mm in size, representing paramorphs of the orthorhombic form after the cubic form. In the Mogilno dome, boracite was found in kieserite–sylvinite lamellae within pure rock-salts of the PZ-3 cyclothem, neighboring thin and tectonically disturbed layers of anhydrite (Wachowiak *et al.* 2012). In the dome, colorless to greenish boracite crystals, with sizes reaching to 5 mm, represent exclusively the low-temperature orthorhombic paramorphs after the high-temperature cubic form. Szaibélyite has been recognized only in the Kłodawa dome as a finely crystalline admixture disseminated in the floor of the kieserite–carnallite layer of the PZ-3 cyclothem (Fijał 1970, 1973, Wachowiak 1998).

Beside the above-mentioned minerals, a subordinate borate phase forming “cubic” crystals, with a general composition  $(\text{Fe},\text{Mg},\text{Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  that initially was classified as ericaite (Wachowiak 1998) was also recognized in the Kłodawa salt deposit. However, more detailed investigations showed that it is congolite. The congolite was found in the Underlying Halite and Youngest Halite, two stratigraphic units in the Zechstein profile separated by the Pegmatite Anhy-

drite, located around the border of the PZ-3 and PZ-4 cyclothem (Fig. 2). In the Kłodawa dome, the Underlying Halite forming the roof of the PZ-3 cyclothem is represented by a 3 m thick layer of grey-orange rock-salt containing 93.4–96.1 wt.% NaCl. In the floor of the layer, there is Brown Zuber; in the roof, Pegmatite Anhydrite occurs. The Youngest Halite forms a lode, about 70 m thick, of almost pure halite, containing 98.5–99.0 wt.% NaCl, pale orange to pale pink in color, but intensively pink in close proximity to anhydrite lamellae. Congolite occurs in the floor of the layer, at a distance of 3–4 m from the contact with Pegmatite Anhydrite, in an interval of a further 4–5 m. All the units are easy to find because they occur almost vertically on the dome and are crossed by underground passages at all exploitation levels of the Kłodawa mine.

Samples of rock-salt, each about 10 kg, were collected from the Underlying Halite and Youngest Halite units. Halite was dissolved in water, then the residue was weighed. The content of insoluble minerals ranged between 3.9–6.6 wt.% in the Underlying Halite and 0.6–1.4 wt.% in the Youngest Halite. Grains with sizes ranging from 0.05 to 0.6 mm constituted about 95% of the insoluble residue; in all samples, anhydrite was present as the predominant mineral with a content *ca.* 99.0–99.5 vol.%, in association with traces of autogenic quartz, calcite, hematite, fine-crystalline chlorite, and hydromica aggregates (Wachowiak 1998). Congolite is present, usually in the amount of some tens of discrete crystals or intergrowths with a total mass of 0.1–0.5 g in a single rock-salt sample (10–50 ppm). In all samples the congolite crystals are similar; they differ one from another, at most, in size, *i.e.*, the crystals occurring in the the Underlying Halite unit are smaller and rarer than those occurring in the Youngest Halite.

The Kłodawa congolite forms euhedral, pseudocubic crystals with dominant  $\{100\}_{\text{cubic}}$  and less prominent  $\{111\}_{\text{cubic}}$  forms, or occurs as aggregates of intergrown crystals. The sizes of the crystals commonly range from 0.3 to 0.6 mm. Only a dozen crystals out of some hundred from the rock-salt samples of the Underlying Halite and Youngest Halite units had sizes greater than 1 mm, and not more than 1.2 mm. The crystals have a vitreous luster, and are commonly translucent to transparent, with color varying from yellowish through pale-violet to pale-violet-brown and brownish, with a zonal texture often visible in hand specimens (Fig. 3).

#### METHODS

A dozen crystals of  $(\text{Fe},\text{Mg},\text{Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate with sizes about 0.5–0.6 mm were mounted in epoxy on a glass plate in varying positions to present different orientations in thin section. The plate was ground to about 30  $\mu\text{m}$  thickness and polished. Optical observations in transmitted light were carried out using an Olympus BX-12 microscope.

TABLE 1. X-RAY DIFFRACTION PATTERNS OF "CONGOLITE" FROM THE KŁODAWA SALT MINE

1		2		3		4		hkl
$d_{hkl}$	$l/l_0$	$d_{hkl}$	$l/l_0$	$d_{hkl}$	$l/l_0$	$d_{hkl}$	$l/l_0$	
6.086	80	6.072	50	6.100	200	6.087	130	012
4.299	130	4.297	80	4.310	450	4.302	190	110, 104
3.5123	350	3.5086	290	3.520	450	3.511	490	202, 006
3.0421	1000	3.0406	1000	3.050	800	3.043	1000	024
2.7219	530	2.7218	330	2.725	1000	2.723	540	122, 116
2.4875	200	2.4869	75	2.491	25	2.486	150	300
2.4825	60	2.4824	60			2.486	150	018
		2.3417	70	2.346	100			125
		2.1538	25	2.158	600	2.151	380	220
2.1512	450	2.1509	710	2.153	600	2.151	380	208
				2.061	750	2.057	360	131, 119
2.0568	450	2.0568	835	2.058	750	2.057	360	217
2.0287	130	2.0298	150	2.032	40	2.029	110	312, 036
1.9230	50	1.9231	140	1.925	200	1.924	60	134, 128
		1.8373	15	1.838	700			042
1.8356	85					1.834	290	226
1.8331	460	1.8339	350	1.834	700	1.834	290	02.10
1.7586	100	1.7569	340	1.761	600	1.758	180	404
1.7543	60	1.7530	110	1.755	400			00.12
1.6892	90	1.6896	85	1.692	450	1.689	130	232
1.6872	70	1.6872	230	1.688	450	1.689	130	21.10
1.6282	70	1.6258	120	1.629	500	1.627	100	324, 318
1.6245	40	1.6230	110	1.625	100			11.12
1.5861	30			1.587	140			235
		1.5843	60	1.585	140	1.584	40	229
1.5829	30	1.5826	55			1.584	40	12.11
1.5214	35	1.5217	70	1.523	200	1.522	40	048
1.4767	75	1.4778	50	1.479	450	1.477	110	502, 416, 13.10
1.4745	45	1.4731	85	1.474	140			01.14
1.4353	30	1.4351	85	1.437	350	1.435	70	330, 054
				1.435	300			238
1.4335	50	1.4333	90			1.435	70	30.12
1.4080	15			1.408	140			241
1.4058	20	1.4056	30	1.406	140			505, 31.11
1.4041	20	1.4037	50			1.405	20	21.13
1.3973	20	1.3969	65	1.400	80			422
1.3950	60	1.3951	120	1.397	200	1.395	50	40.10
		1.3932	120	1.396	350	1.395	50	20.14
		1.3606	110	1.364	600	1.361	90	244
1.3607	100	1.3588	25	1.362	600	1.361	90	22.12
		1.3355	25	1.338	100	1.336	10	425, 419
				1.335	200			11.15
1.3277	20	1.3264	30	1.326	100	1.329	20	336, 32.10, 12.14
1.2987	15	1.2981	35	1.298	350	1.298	30	514
1.2972	25	1.2970	30	1.298	350	1.298	30	508

Notes: 1, 2 – "congolite" from the Kłodawa salt dome; 3 – data of Wendling *et al.* (1972); 4 – data of Dawty & Clark (1973).

Six crystals of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate were mounted in epoxy, polished, and coated with carbon for electron microprobe analysis. The analyses were performed at the Inter-Institute Analytical Complex for Minerals and Synthetic Substances of University of Warsaw using a Cameca SX 100 electron microprobe operating in wavelength-dispersive (WDS) mode with the following conditions: accelerating voltage 15 kV, beam current 10 nA, beam diameter 2  $\mu\text{m}$ , peak count-time 20 s, background time 10 s. The following standards, analytical lines, and crystals were used: diopside – Mg ( $K\alpha$ , TAP), hematite – Fe ( $K\alpha$ , LIF), rhodonite – Mn ( $K\alpha$ , LIF), and tugtupite – Cl ( $K\alpha$ , PET). Minimum detection limits for the analyzed elements were: 0.04 wt.% for Mg, 0.16 wt.% for Fe and Mn, and 0.08 wt.% for Cl. Data were corrected with the PAP procedure (Pouchou & Pichoir 1985). The  $\text{B}_2\text{O}_3$  content was not directly analyzed; it was calculated from stoichiometry, assuming normalization of the analyses in relation to 3 (Fe + Mn + Mg) and 14 (O,Cl) atoms per formula unit (*apfu*).

X-ray diffraction patterns were recorded at room temperature with a Philips APD X'Pert PW 2030 diffractometer under the following conditions:  $\text{CuK}\alpha$  radiation with  $\lambda = 1.5418 \text{ \AA}$ , accelerating voltage 35 kV, beam current 30 mA, a graphite monochromator, registration velocity  $0.02^\circ(2\theta)/\text{s}$ , registration range  $2\text{--}75^\circ(2\theta)$ . The data were analyzed using an X'rayan computer program and X-ray standards in the form of ICDD files. Indexing was done using a DHN PDS computer program.

Infrared spectra were recorded in the  $400\text{--}4000 \text{ cm}^{-1}$  region, with resolution  $\sim 4 \text{ cm}^{-1}$  using a BIO-RAD FTS-165 spectrometer. The samples were prepared as standard KBr pellets with sample/KBr mass ratio  $\sim 1:200$ .

## RESULTS

Crystals of the Kłodawa  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate are transparent, colorless in thin section and uniaxial (–), which unequivocally distinguishes the mineral as congolite from the biaxial ericaite.

X-ray diffraction patterns of congolite from Kłodawa are presented in Table 1, in comparison with data for congolite from the type locality in the Republic of Congo (Wendling *et al.* 1972) and a 'trigonal iron-rich analogue of boracite' from Bischofferode, Thuringen, Germany (Dowty & Clark 1973), originally classified as ericaite (Kühn & Schaacke 1955). Infrared spectra of the Kłodawa congolite are presented in Figure 4. Both the X-ray diffraction pattern and the IR spectra, almost analogous with the spectra of congolite from the Penobsquis and Millstream evaporate deposits of southern New Brunswick, Canada (Burns & Carpenter 1997), corroborate the presence of rhombohedral  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate (congolite) in the Kłodawa dome.

Figure 5 presents five back-scattered-electron (BSE) images of the congolite crystals analyzed with WDS, showing their complex texture in detail. The crystals commonly show the presence of numerous rhombohedral Fe-rich cores, sometimes intergrown themselves, with very delicate oscillatory zoning. The cores are overgrown with numerous medium- to dark-grey zones grading to the  $\{100\}_{\text{orthorhombic}}$  form. Such subcrystals sometimes form regular intergrowths composed of 2–4 specimens, which furthermore are overgrown with some distinct concentric zones parallel to faces of the  $\{100\}$  and, less commonly, to  $\{111\}_{\text{cubic}}$  forms. Table 2 provides representative WDS analyses of congolite from morphologically different parts of the crystals.

## EVOLUTION OF CONGOLITE IN THE KŁODAWA SALT DOME

The internal texture of congolite crystals from the Kłodawa salt dome distinctly reflect phase-transitions from rhombohedral congolite through orthorhombic ericaite to a cubic, high-temperature, mainly Fe-dominant, analogue of boracite. This pattern corresponds to crystallization of the borate minerals at increasing temperature. Currently, those parts of the crystals showing orthorhombic and cubic morphology generally represent congolite and, much less frequently, trembathite paramorphs after the aforementioned, higher-temperature structural varieties of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  and  $(\text{Mg,Fe,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$ , which are unstable at room-temperature.

The cores of the crystals, light grey in BSE images, with delicate oscillatory zoning and sometimes with the distinct morphology of the  $\{100\}_{\text{rhombohedral}}$  form (*e.g.*, crystal 23b in Fig. 5), correspond to crystallization of  $(\text{Fe,Mg})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate under conditions when congolite was stable. Compositions in successive overgrowth zones present in the core evolve from almost pure congolite  $(\text{Fe}_{2.86}\text{Mg}_{0.07}\text{Mn}_{0.07})_{\Sigma 3}\text{B}_7\text{O}_{13}\text{Cl}$  to  $(\text{Fe}_{2.59}\text{Mg}_{0.34}\text{Mn}_{0.07})_{\Sigma 3}\text{B}_7\text{O}_{13}\text{Cl}$ , at an almost constant Mn/(Fe + Mn) ratio (Fig. 6a, trend 'R'). The phase-transition temperature of congolite to ericaite was evaluated on the basis of the system temperature –

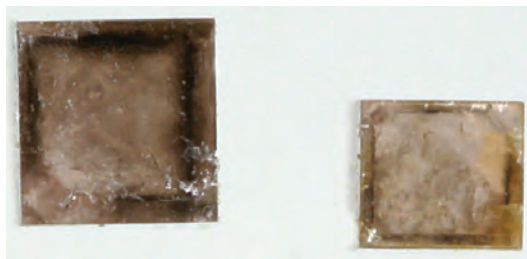


Fig. 3. Example crystals of the Kłodawa congolite. The size of both crystals is about 0.5–0.6 mm.

atomic proportion Fe of Burns & Carpenter (1996) for all the studied crystals. The temperature range, 230 to 250 °C, was determined from the compositions of the outermost zone in the rhombohedral core and the adjacent first zone with orthorhombic morphology by their reference to the path of  $R3c \leftrightarrow Pca2_1$  transition in the system (Fig. 7). Furthermore, compositional oscillation in the cores is relatively slight, whereas the parts of the crystals with orthorhombic morphology show much more variable and simultaneously Mg-rich compositions. All this may indicate that the primary congolite crystallized as a result of the metasomatic reaction of Fe and Mn carriers present within the host rock-salts of the Underlying Halite and Youngest Halite units

with  $(BO_3)^{3-}$ -bearing brines and fluids released from evaporite sediments, which, utilizing convenient pathways for migration in the dome, infiltrated into the host rock-salts at increasing, but relatively low, temperatures of 100–150 °C (Fig. 7). A slight, though progressive, increase of  $Mg^{2+}$  along with temperature may reflect a tendency towards faster crystallization of a congolite variety richer in  $Fe^{2+}$  and the substitution of  $Mg^{2+}$  for  $Fe^{2+}$  at higher temperatures, or may also point to an increasing role for  $Mg^{2+}$  in brines and fluids infiltrating into the rock-salts at higher temperatures.

A distinct inflow of  $Mg^{2+}$ -bearing brines and fluids is evident at temperatures of 230–250 °C when crystallization passes from the field of stable rhombohedral

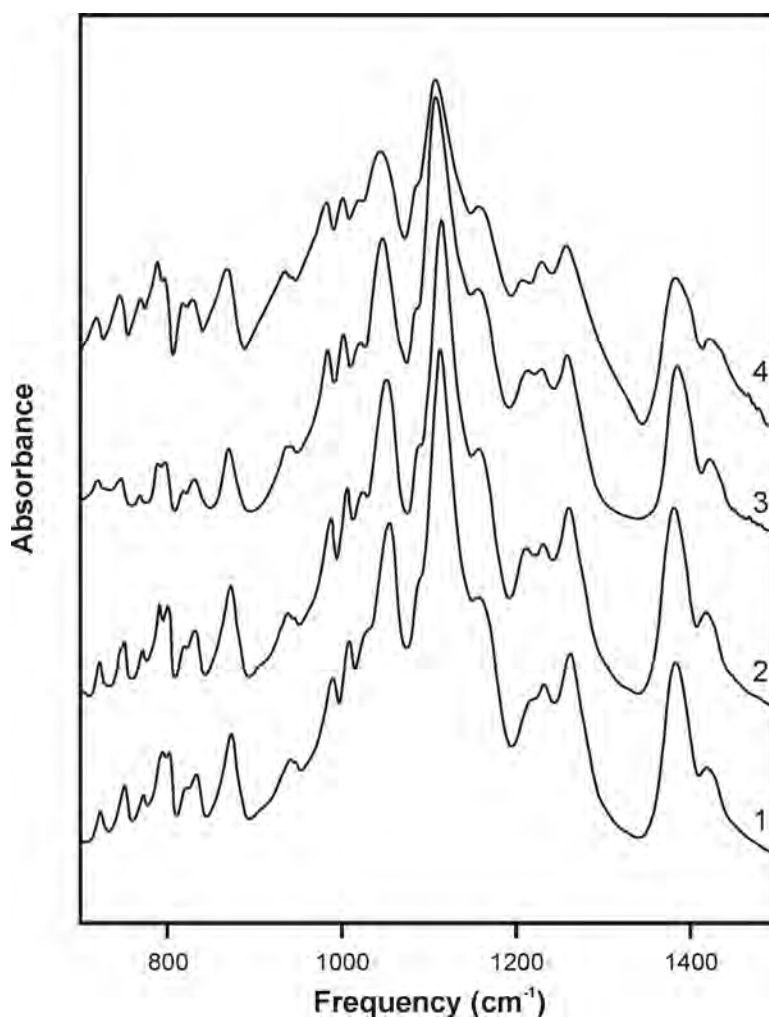


FIG. 4. Infrared spectra of the Kłodawa congolite (3 and 4) compared to the spectra of congolite S8A and S8C (1 and 2, respectively) of Burns & Carpenter (1996) from the Sussex deposits, New Brunswick, Canada, presented by Burns & Carpenter (1997).

TABLE 2. REPRESENTATIVE EMP COMPOSITIONS OF CONGOLITE

Crystal 45c	142	18a	145	36a	11a	32a	116	156	173	Cr. 7b	6	9	6a	39a	10a	15a	60	20a	22a	56	22	
	R			O			C				O	C			C1			C2				
FeO wt. %	41.80	38.44	38.05	28.44	30.24	26.21	34.90	31.41	40.48	24.10	27.74	31.88	37.22	39.21	25.74	33.46	35.21	31.04	27.73	25.42		
MnO	1.07	1.07	1.07	0.79	1.00	1.15	1.56	1.42	2.53	1.04	1.17	1.26	1.45	2.81	2.54	3.22	4.90	6.59	6.71	5.62		
MgO	0.58	2.84	4.07	10.88	9.21	12.36	5.56	8.53	1.15	13.57	10.83	7.65	3.87	1.43	11.35	5.13	2.89	4.86	7.16	9.83		
B <sub>2</sub> O <sub>3</sub>	49.71	50.46	52.50	55.05	53.87	55.90	52.50	54.39	50.98	55.84	54.61	52.88	51.54	50.41	54.98	51.93	51.20	52.35	53.52	55.02		
Cl <sub>2</sub>	7.02	7.19	7.46	7.76	7.91	8.02	7.48	7.74	7.41	7.94	7.66	7.78	7.56	7.39	7.72	7.34	7.57	7.86	7.57	7.84		
O=Cl <sub>2</sub>	-1.58	-1.62	-1.68	-1.75	-1.78	-1.81	-1.69	-1.75	-1.67	-1.79	-1.73	-1.75	-1.71	-1.67	-1.74	-1.66	-1.71	-1.77	-1.71	-1.77		
Total	98.60	98.38	101.46	101.15	100.44	101.83	100.31	101.74	100.87	100.70	100.28	99.69	99.94	99.59	100.59	99.43	100.06	100.93	100.98	101.96		
	Number of ions on the basis of 3 Me <sup>2+</sup> and 14 (O,Cl) pfu																					
Fe <sup>2+</sup> apfu	2.86	2.59	2.46	1.75	1.90	1.59	2.26	1.96	2.69	1.47	1.73	2.04	2.45	2.64	1.59	2.19	2.33	2.01	1.76	1.57		
Mn <sup>2+</sup>	0.07	0.07	0.07	0.05	0.06	0.07	0.10	0.09	0.17	0.06	0.07	0.08	0.10	0.19	0.16	0.21	0.33	0.43	0.43	0.35		
Mg <sup>2+</sup>	0.07	0.34	0.47	1.20	1.03	1.34	0.64	0.95	0.14	1.47	1.20	0.87	0.45	0.17	1.25	0.60	0.34	0.56	0.81	1.08		
B <sup>3+</sup>	7.01	7.01	7.01	7.01	7.00	7.00	7.01	7.01	7.00	7.01	7.01	7.00	7.00	7.00	7.01	7.01	6.99	6.99	7.01	7.01		
Cl <sup>-</sup>	0.97	0.98	0.98	0.97	1.01	0.99	0.98	0.98	1.00	0.98	0.97	1.01	1.01	1.01	0.97	0.97	1.02	1.03	0.97	0.98		
Mn/(Mn + Fe)	0.025	0.027	0.028	0.027	0.032	0.042	0.043	0.044	0.060	0.042	0.041	0.038	0.038	0.068	0.091	0.089	0.123	0.177	0.197	0.183		

Notes: cr. 7b = crystal 7b; R, O, C (C1, C2) – parts of the crystals with rhombohedral, orthorhombic and cubic morphology.

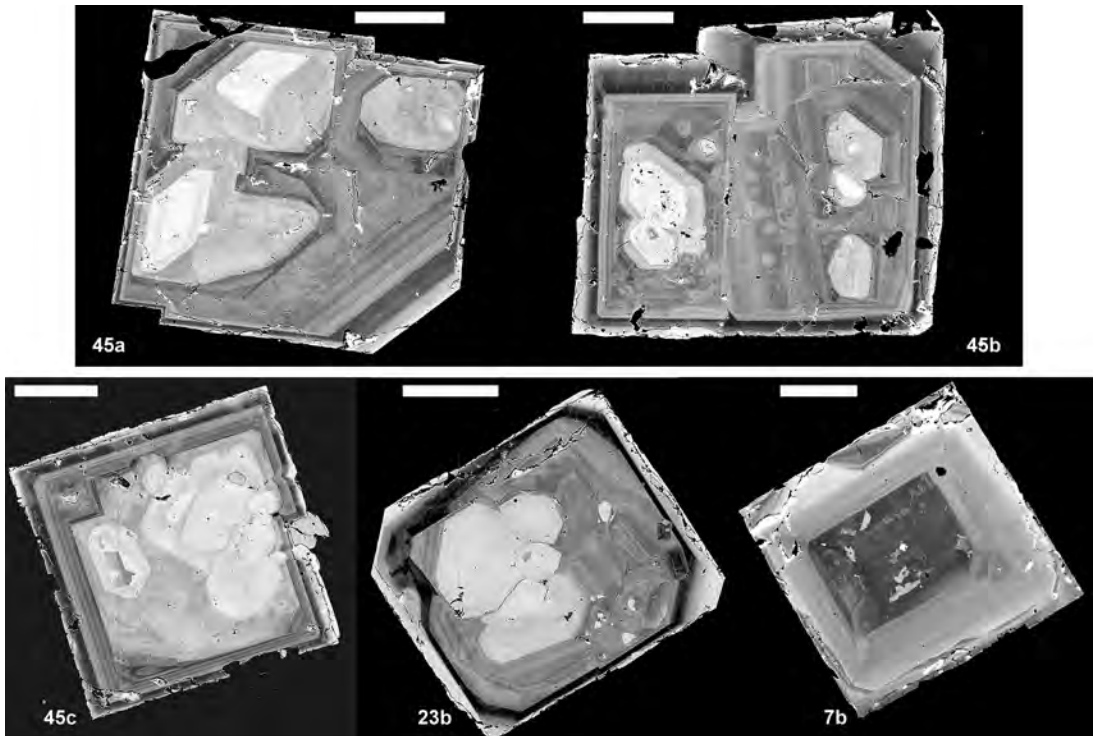


FIG. 5. Example BSE images of the Kłodawa congolite. Scale bar corresponds to 200  $\mu\text{m}$ .



congolite to the field of stable orthorhombic ericaite due to a sudden increase of Mg<sup>2+</sup> in the crystallization environment at almost constant Mn/(Mn + Fe) (Figs. 6, 7). During further crystallization of orthorhombic ericaite, compositions in successive overgrowth zones grading to the {100}<sub>orthorhombic</sub> form (the best visible in crystal 45a in Fig. 5), oscillate between more Mg<sup>2+</sup>- and more Fe<sup>2+</sup>-bearing at progressively increasing Mn/(Mn + Fe) ratios (Fig. 6 a,b; trend 'O'). However, they generally moved towards trembathite, reaching a composition close to (Fe<sub>1.88</sub>Mg<sub>1.06</sub>Mn<sub>0.06</sub>)<sub>Σ3</sub>B<sub>7</sub>O<sub>13</sub>Cl. Such an evolution indicates certain limitations to the inflow of Mg<sup>2+</sup>-bearing fluids and brines, and an increasing role for Mg<sup>2+</sup> and Fe<sup>2+</sup> diffusion to the crystal growing at that stage. The phase-transition temperature of the orthorhombic ericaite to the cubic phase, evaluated as before, varies within the very narrow range of 310–315 °C (Fig. 7). This phase transition could be attained during the progressive compositional evolution of the fluids/brines and growing crystals (e.g., crystals 45a and 45c in Fig. 5) or be forced by a subsequent, higher-temperature inflow of Mg-bearing fluids, marked distinctly in BSE images of the majority of the crystals in the form of the outermost, dark zone with distinct morphology corresponding to {100}<sub>cubic</sub> and, less commonly, {111}<sub>cubic</sub> forms (crystals 45a, 45b, 45c, and 23b in Fig. 5). A Mg-bearing borate that crystallized at this stage reached a trembathite composition, (Mg<sub>1.57</sub>Fe<sub>1.36</sub>Mn<sub>0.07</sub>)<sub>Σ3</sub>B<sub>7</sub>O<sub>13</sub>Cl, which is the richest in Mg found in the Kłodawa crystals (Table 3), with Mn/(Mn + Fe) ratio comparable with values characterizing the crystallization of orthorhombic ericaite (Fig. 6) (the boundary between trends 'O' and 'C'). This higher-temperature inflow of Mg-bearing fluids can be recognized as a short-lived phenomenon because the cubic phase is increasingly Fe-rich outwards, finally reaching a composition close to (Fe<sub>2.74</sub>Mn<sub>0.20</sub>Mg<sub>0.06</sub>)<sub>Σ3</sub>B<sub>7</sub>O<sub>13</sub>Cl, with a relatively high Mn/(Mn + Fe) ratio of 0.05–0.07 (Figs. 6, 7). Considering the ratios between the mean sizes of the rhombohedral core and orthorhombic and cubic zones, the crystallization of the Fe-borate definitely finished at about 350–360 °C, probably due to the diminished availability of (BO<sub>3</sub>)<sup>3-</sup> ions.

Some crystals of the Kłodawa congolite do not show a rhombohedral core, e.g., crystal 7b in Figure 5. The internal part of such crystals, dark in BSE images, in terms of composition commonly corresponds to the late orthorhombic ericaite crystallized under the action of Mg-bearing fluids. This part of the crystals is overgrown by a relatively wide external mantle, showing delicate zoning and typical {100}<sub>cubic</sub> morphology, with compositional characteristics typical of trend 'C' already described as the final member in the 3-phase-crystals of (Fe,Mg,Mn)<sub>3</sub>B<sub>7</sub>O<sub>13</sub>Cl borate. However, in a few crystals of that category, e.g., crystal 7b in Figure 5, the cubic mantle richest in Fe is at least partly overgrown by two additional, rather thin and

incomplete zones, medium grey in BSE images, also with cubic morphology, showing still more advanced geochemical characteristics. In each of the zones, congolite shows compositional differentiation not only across but also lengthways, with the Mn/(Mn + Fe) ratio reaching its highest values of 0.12 and 0.19, respectively (Fig. 6; trends 'C<sub>1</sub>' and 'C<sub>2</sub>'). It can be supposed that they represent a final stage of crystallization of (Fe,Mg,Mn)<sub>3</sub>B<sub>7</sub>O<sub>13</sub>Cl cubic borate in the Kłodawa dome from the most geochemically evolved residual brines and fluids.

ORIGIN OF (BO<sub>3</sub>)<sup>3-</sup>-BEARING AND Mg<sup>2+</sup>-BEARING BRINES AND FLUIDS

In the Kłodawa dome, the crystallization of (Fe,Mg,Mn)<sub>3</sub>B<sub>7</sub>O<sub>13</sub>Cl and (Mg,Fe,Mn)<sub>3</sub>B<sub>7</sub>O<sub>13</sub>Cl borates results from infiltration and metasomatism within the host rock-salts enriched in Fe and Mn, under the action of (BO<sub>3</sub>)<sup>3-</sup>-bearing brines and fluids generated through dehydration of primary Zechstein evaporate sediments in diagenetic and metamorphic processes, inducing lateral and vertical mobility of evaporites and finally dome formation. The relatively low temperature of incipient crystallization, about 100 °C, and the crystallization at the time of almost pure congolite with only a minimal Mg content, suggest that the primary brines and fluids were rather poor in Mg, and the primary congolite, observed currently as rhombohedral cores in the crystals, utilized mainly Fe and Mn from the host rock-salts. A source of the required heat could be (1) the geothermal gradient, probably higher than that at present, (2) heat generated by movement of the salts and their upward flow in the crust, or (3) an additional flux of heat derived from deeper sub-Zechstein basement intensively faulted by tectonic movements of Mesozoic age (e.g., Znosko 1957, Dadlez 1982, Pokorski 1988,

TABLE 3. REPRESENTATIVE EMP COMPOSITIONS OF TREMBATHITE

Cryst. 23b	1	2	3	4	5
	wt.%				
FeO	22.51	22.90	23.50	23.77	24.55
MnO	1.18	1.26	1.14	1.03	1.20
MgO	14.50	14.45	14.01	13.48	13.39
B <sub>2</sub> O <sub>3</sub>	56.04	56.49	56.15	55.24	56.12
Cl <sub>2</sub>	8.10	8.12	8.03	7.97	8.16
-O=Cl <sub>2</sub>	-1.83	-1.83	-1.81	-1.80	-1.84
Total	100.51	101.39	101.01	99.69	101.57
	Number of ions on the basis of 3 Me <sup>2+</sup> pfu and 14 (O,Cl)				
Fe <sup>2+</sup>	1.36	1.38	1.42	1.46	1.48
Mn <sup>2+</sup>	0.07	0.08	0.07	0.06	0.07
Mg <sup>2+</sup>	1.57	1.55	1.51	1.48	1.44
B <sup>3+</sup>	7.00	7.00	7.01	7.00	7.00
Cl <sup>-</sup>	0.99	0.99	0.98	0.99	1.00

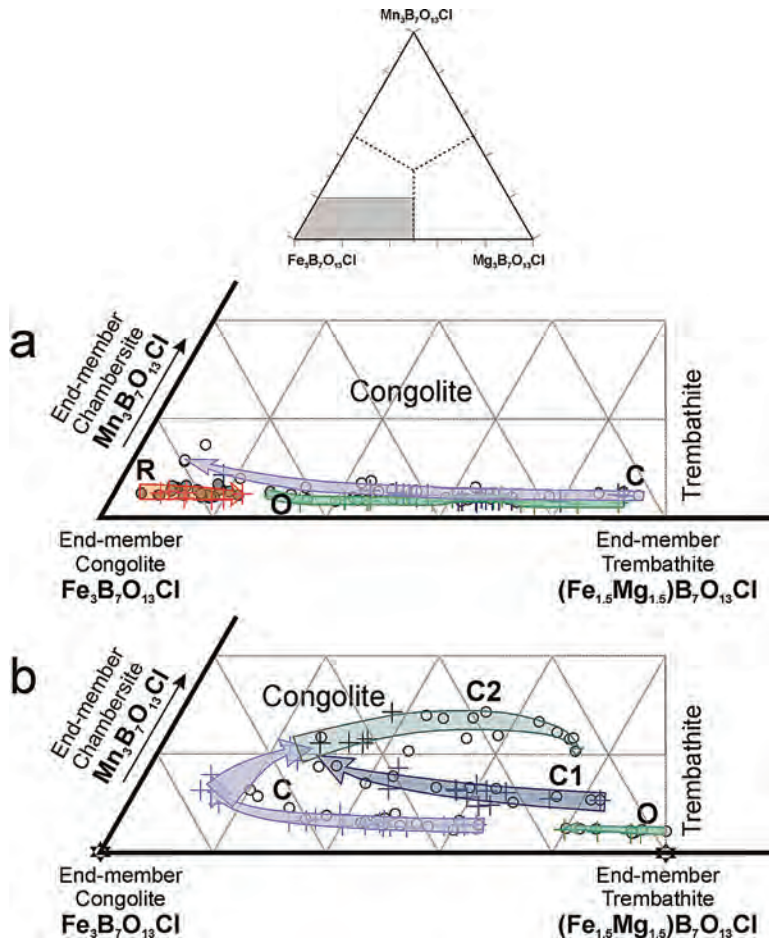


FIG. 6. Example compositional trends in crystals of congolite from the Kłodawa Salt Mine, presented in the system  $\text{Fe}_3\text{B}_7\text{O}_{13}\text{Cl}$ – $\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$ – $\text{Mn}_3\text{B}_7\text{O}_{13}\text{Cl}$ . (a) Crystals with rhombohedral cores (45c). (b) Crystals without rhombohedral cores (7b). Symbols: 'R' and an orange arrow – compositions corresponding to rhombohedral morphology; 'O' and green arrows – compositions corresponding to orthorhombic morphology; 'C' and violet arrows – compositions corresponding to cubic morphology; 'C1' and 'C2', and blue and pale green arrows – compositions from additional zones of cubic morphology. Empty circles (o) and crosses (+) represent data points from two independent microprobe sessions.

Wagner 1994, Krzywiec 2004). Disturbance of halokinetic stability and decompression in rejuvenated fault zones could induce movements of the salts connected with the formation of diapiric structures and the release of brines from primary Zechstein evaporites. Changes in the composition of crystals of  $(\text{Fe},\text{Mg},\text{Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  and  $(\text{Mg},\text{Fe},\text{Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borates during growth indicate that early brines carried  $(\text{BO}_3)^{3-}$  ions, but were rather poor in  $\text{Mg}^{2+}$ . Magnesium appears distinctly only above 200 °C as an inflow of Mg-bearing fluids, transferring the crystallization of  $(\text{Fe},\text{Mg},\text{Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$

borate from the field of stable congolite, first to the field of stable orthorhombic ericaite and then to the field of stable cubic phase that reached higher concentrations of  $\text{Mg}^{2+}$  than  $\text{Fe}^{2+}$ . This might indicate that at higher temperatures, the primary  $(\text{BO}_3)^{3-}$ -containing fluid has been gradually modified by an additional inflow of an  $\text{Mg}^{2+}$ -bearing component. A source of the latter can be seen in the relatively thick deposits of Younger Potash (up to 30–40 m), containing inclusions of carnallite,  $\text{KMgCl}_3 \cdot 6\text{H}_2\text{O}$ , kainite,  $\text{KMgCl}(\text{SO}_4) \cdot 3\text{H}_2\text{O}$ , and kieserite,  $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ , with obliterated primary sedi-

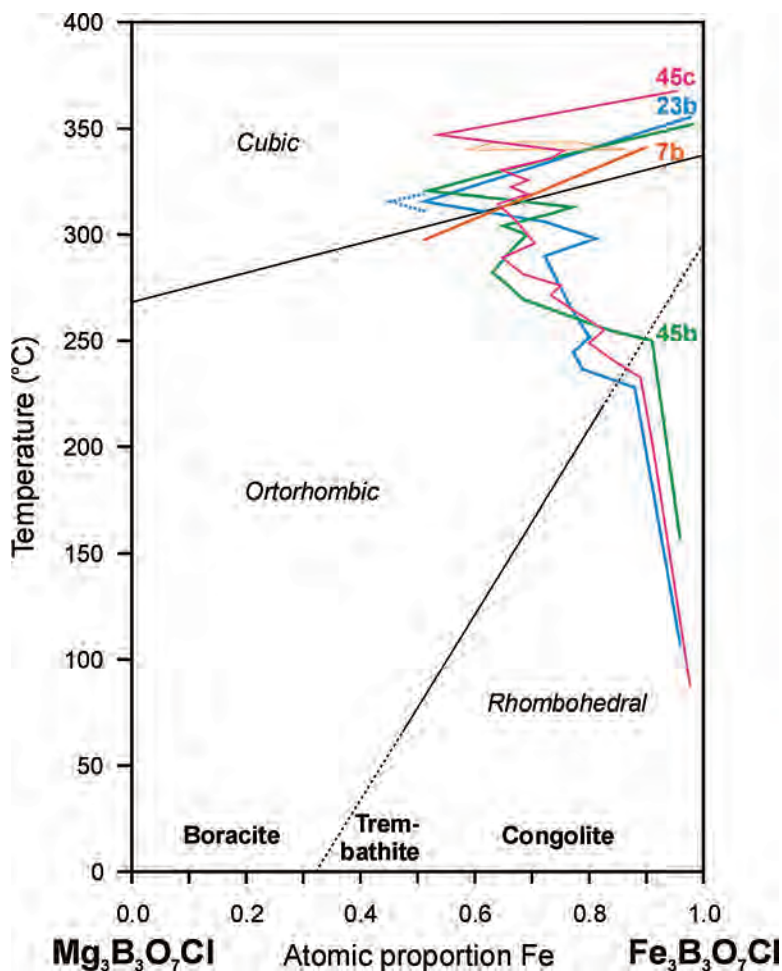


Fig. 7. Hypothetical paths for congolite crystallization in the Kłodawa salt dome in the system atomic proportion Fe versus T (°C).

mentary structures in contrast to the rock-salt, and of Upper Younger Halite with impurities of potash (~150 m) of the PZ-3 cyclothem. The action and a regional role for such a Mg-bearing fluid are marked in the form of common dolomitization of the raurak and argow limestones occurring around the salt domes, *e.g.*, of Inowrocław, Góra, Wapno and the Kłodawa-Łęczysca anticline, even up to 20 km distance. The critical role of K-Mg salts in the generation of Mg-bearing fluids seems unquestionable. For example, in the Inowrocław dome, boracite occurs within the kieserite-carnallite salts of the PZ-2 cyclothem (Poborski *et al.* 1956, Prochazka 1957, Fijał 1973), and in the Kłodawa dome itself, boracite and szaibélyite were found, among others, in the floor of the kieserite-carnallite layer of the PZ-3 cyclothem (Hanczke 1969, Fijał 1970, 1973,

Wachowiak 1998). In addition, in the Kłodawa mine there occurred contemporary leaks of paleobrine from the Main Anhydrite level (A-3), load of cyclothem PZ-3, always saturated with K and Mg.

In the Kłodawa salt dome, congolite was found only in the Underlying Halite and Youngest Halite units around the border of the PZ-3 and PZ-4 cyclothem, at a distance of 3–4 m from their contact with Pegmatite Anhydrite. Such a position may indicate only that Pegmatite Anhydrite, at most only some tens of centimeters thick, was a main pathway conveying  $(\text{BO}_3)^{3-}$  and Mg-bearing brines and fluids, penetrating into the adjacent Underlying Halite and Youngest Halite. The occurrence of congolite at only a short interval from the Pegmatite Anhydrite reflects the similar kinetics of the reactions both in the Underlying Halite and

Youngest Halite rock-salts, leading to the formation of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  and  $(\text{Mg,Fe,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borates. Such a localization of congolite-trembathite crystals in the Kłodawa dome agrees with occurrences of boracite in other Polish salt domes like Mogilno or Inowrocław, where high-temperature forms of boracite have been similarly found in a spatial relationship with cracked anhydrite. This suggests that tectonically disturbed and cracked anhydrite rocks were convenient pathways for the migration of hot brines and fluids within the domes, in contrast to the host rock-salts, which are too solid to be penetrated by the fluids for distances greater than a few meters.

### CONCLUSIONS

The Kłodawa salt dome in central Poland is the first occurrence of congolite and trembathite in Central Europe, and one of the few known worldwide.

The internal texture of congolite crystals from Kłodawa distinctly reflects phase transitions from rhombohedral congolite through orthorhombic ericaite to cubic, high-temperature, mainly Fe-dominant and, less frequently, Mg-dominant phases, during the formation of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  and  $(\text{Mg,Fe,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borates at increasing temperature. Currently, parts of the crystals showing orthorhombic and cubic morphology represent congolite paramorphs after the higher-temperature structural varieties of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$ , which are unstable at room-temperature. Temperature ranges of 230–250 °C and 310–315 °C were determined for  $R3c \rightarrow Pca2_1$  and  $Pca2_1 \rightarrow F43c$  phase-transitions during growth of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate crystals. The thermal range of crystallization of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borates from the Kłodawa dome was in the range ca. 100 °C to 350–360 °C.

The primary congolite crystallized as a result of the metasomatic reaction of Fe and Mn carriers present within the host rock-salts of the Underlying Halite and Youngest Halite units located around the PZ-3/PZ-4 boundary, with  $(\text{BO}_3)^{3-}$ -bearing brines and fluids released from primary evaporate sediments (probably mainly the Upper Younger Halite with potash impurities), in diagenetic and metamorphic processes connected with disturbances in halokinetic stability, and intensive faulting of the pre-Zechstein basement and decompression, inducing upward flow of evaporites and diapir formation. The brines and fluids have utilized cracks in Pegmatite Anhydrite as convenient pathways for migration in the dome, in contrast to the host rock-salts, which are too solid to be penetrated by the fluids for distances more than a few meters.

The  $R3c \rightarrow Pca2_1$  and  $Pca2_1 \rightarrow F43c$  phase-transitions were controlled by inflows of Mg-bearing fluids at increasing temperature. A source of the fluids can be found in the relatively thick loads of potash salts of the PZ-3 cyclothem (Younger Potash), containing

inclusions of carnallite, kainite, and kieserite. The end of the supply of  $(\text{BO}_3)^{3-}$  ions was the main reason for the cessation of  $(\text{Fe,Mg,Mn})_3\text{B}_7\text{O}_{13}\text{Cl}$  borate crystallization in the dome.

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