

Droninoite, $\text{Ni}_3\text{Fe}^{3+}\text{Cl}(\text{OH})_8 \cdot 2\text{H}_2\text{O}$, a New Hydrotalcite-Group Mineral Species from the Weathered Dronino Meteorite¹

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Abstract—A new mineral, droninoite, was found in a fragment of a weathered Dronino iron meteorite (which fell near the village of Dronino, Kasimov district, Ryazan oblast, Russia) as dark green to brown fine-grained (the size of single grains is not larger than 1 μm) segregations up to 0.15 \times 1 \times 1 mm in size associated with taenite, violarite, troilite, chromite, goethite, lepidocrocite, nickelbischofite, and amorphous Fe^{3+} hydroxides. The mineral was named after its type locality. Aggregates of droninoite are earthy and soft; the Mohs hardness is 1–1.5. The calculated density is 2.857 g/cm³. Under a microscope, droninoite is dark gray-green and nonpleochroic. The mean (cooperative for fine-grained aggregate) refractive index is 1.72(1). The IR spectrum indicates the absence of SO_4^{2-} and CO_3^{2-} anions. Chemical composition (electron microprobe, partition of total iron into Fe^{2+} and Fe^{3+} made on the basis of the ratio $(\text{Ni} + \text{Fe}^{2+}) : \text{Fe}^{3+} = 3 : 1$; water is calculated from the difference) is as follows, wt %: 36.45 NiO, 12.15 FeO, 17.55 Fe_2O_3 , 23.78 H_2O , 13.01 Cl, $-\text{O}=\text{Cl}_2$ –2.94, total is 100.00. The empirical formula ($Z = 6$) is $\text{Ni}_{2.16}\text{Fe}_{0.75}^{2+}\text{Fe}_{0.97}^{3+}\text{Cl}_{1.62}(\text{OH})_{7.10} \cdot 2.28\text{H}_2\text{O}$. The simplified formula is

$\text{Ni}_3\text{Fe}^{3+}\text{Cl}(\text{OH})_8 \cdot 2\text{H}_2\text{O}$. Droninoite is trigonal, space group $R\bar{3}m$, $R3m$, or $R32$; $a = 6.206(2)$, $c = 46.184(18)$ Å; $V = 1540.4(8)$ Å³. The strong reflections in the X-ray powder diffraction pattern [d , Å (I , %) (hkl)] are 7.76(100)(006), 3.88(40)(0.0.12), 2.64(25)(202, 024), 2.32(20)(0.2.10), 1.965(0.2.16). The holotype specimen is deposited at the Fersman Mineralogical Museum, Russian Academy of Sciences, Moscow, registration number 3676/1.

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INTRODUCTION

Meteorites, primarily those containing a significant amount of metallic and sulfide phases, strongly differ from terrestrial objects in geochemistry and mineralogy. This causes a specific character of weathered products at the Earth's surface. The mineral assemblages produced at the early stage of terrestrial weathering of high-metal meteorites are particularly specific. Those dealing with weathered products of such meteorites are at the interface between supergene mineralogy; the geochemistry of extraterrestrial matter; and the fields of chemistry and metallurgy concerning corrosion of ferrous metals. This still undeveloped line of research can not only expand our knowledge on mineral matter, but

make a new contribution to geochemistry and genetic mineralogy.

The Dronino iron meteorite found in 2000 in the vicinity of the village of Dronino, Kasimov district, Ryazan oblast, Russia, turned out very promising with regard to supergene minerals. This meteorite is attributed to ataxites and composed of low-Ni kamacite with minor taenite, chromite, and Fe and Ni sulfides (Russell et al., 2004; Grokhovsky et al., 2005). Locally, it is highly enriched in sulfides (up to tens of vol %), among which troilite and violarite are predominant.

The Dronino meteorite shower fell on the Russian Platform approximately 5000–8000 years ago after the last glaciation. Numerous fragments of the meteorite from a few grams to 300 kg in weight (total weight is not less than 3 t) were scattered over a few square kilometers. The overwhelming majority of the fragments were recovered from a depth of 0.5–1 m below the surface from glacial and postglacial sandshale sediments. The degree of supergene alteration of meteoritic matter depends on the host rock. For example, the fragments

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Table 1. Chemical composition of droninoite (average of four point analyses)

Component	Wt %	Range	Standard
NiO	36.45	35.13–37.62	Ni
FeO*	12.15	27.31–29.21**	Fe
Fe ₂ O ₃	17.55		
Cl	13.01	12.50–13.40	NaCl
H ₂ O _{calc.} ***	23.78		
–O=Cl ₂	–2.94		
Total	100.00		

Notes: F, Na, Mg, P, S, K, Ca, Al, Si, Mn, Co, and Cr are below detection limits (<0.05–0.2 wt % for various elements). * Fe was partitioned into Fe²⁺ and Fe³⁺ according to (Ni + Fe²⁺): Fe³⁺ = 3 : 1. ** Total iron in the form of FeO. ***Calculated from the difference.

that fell into clay beds are only slightly altered or unaltered altogether except for rusty films or thin crusts. At the same time, fragments of the meteorite recovered from waterlogged sandy interlayers and lenses are highly weathered; they are significantly and often completely replaced with crystalline goethite, hematite, akaganeite, lepidocrocite, and amorphous Fe³⁺ oxides and hydroxides. Partially altered fragments exhibit zoning: the outer rusty oxide–hydroxide shell envelops relics of meteorite material combined with newly formed minerals in the core. Caverns filled with sulfates, chlorides, carbonates, nickel, ferrous and ferric oxides as products of early supergene alteration are typical of the inner zone. A new mineral chukanovite Fe²⁺(CO₃)(OH)₂, an iron analogue of malachite, members of rosasite group (Pekov et al., 2007), reevesite, honessite, nickelhexahydrite and nickelbischofite were discovered from this assemblage of the Dronino meteorite. Note that kamacite was subjected to weathering to the greatest degree, whereas taenite and sulfide minerals are more stable and frequently observed as relics in the samples, where kamacite is completely converted into aggregates of oxygen-bearing phases.

The new mineral species described in this paper, a hydrous Ni and Fe chloride-hydroxide belonging to the hydrotalcite group, was found in a fragment of the Dronino meteorite, partially altered under terrestrial conditions. It was named *droninoite* after type locality. The type material is deposited at the Fersman Mineralogical Museum, Russian Academy of Sciences, Moscow, Russia, registration no. 3676/1.

OCCURRENCE, GENERAL CHARACTERISTICS, PROPERTIES, AND CHEMICAL COMPOSITION

Droninoite was found in the central part of a meteorite fragment 12 × 17 × 19 cm in size. From the outside, the fragment is coated by monomineralic fine-grained goethite crust. The intermediate zone, mainly composed of iron oxides and hydroxides with relics of

taenite and sulfides, is compact and contains pockets of orange-brown lepidocrocite, brown veinlets of the insufficiently studied sulfate–hydroxide of hydrotalcite group, and grains of magnetite, which also occurs as octahedral crystals up to 0.5 mm in size encrusting the walls of small caverns. The core is composed of a polymineralic aggregate consisting of both weakly altered meteoritic minerals (taenite, violarite, troilite, and chromite) and products of alteration of primary minerals, mainly kamacite, under terrestrial conditions: goethite, lepidocrocite, nickelbischofite, and amorphous Fe³⁺ hydroxides. Taenite is retained as numerous lamellae up to a few mm in size. Relics of violarite and troilite reaching 1 cm in size are irregularly shaped or close to ellipsoid. Chromite occurs as isolated sporadic grains a few millimeters in size. Nickelbischofite and amorphous Fe³⁺ hydroxides were identified as veinlets, pockets, and spherulitic crusts coating the walls of cavities.

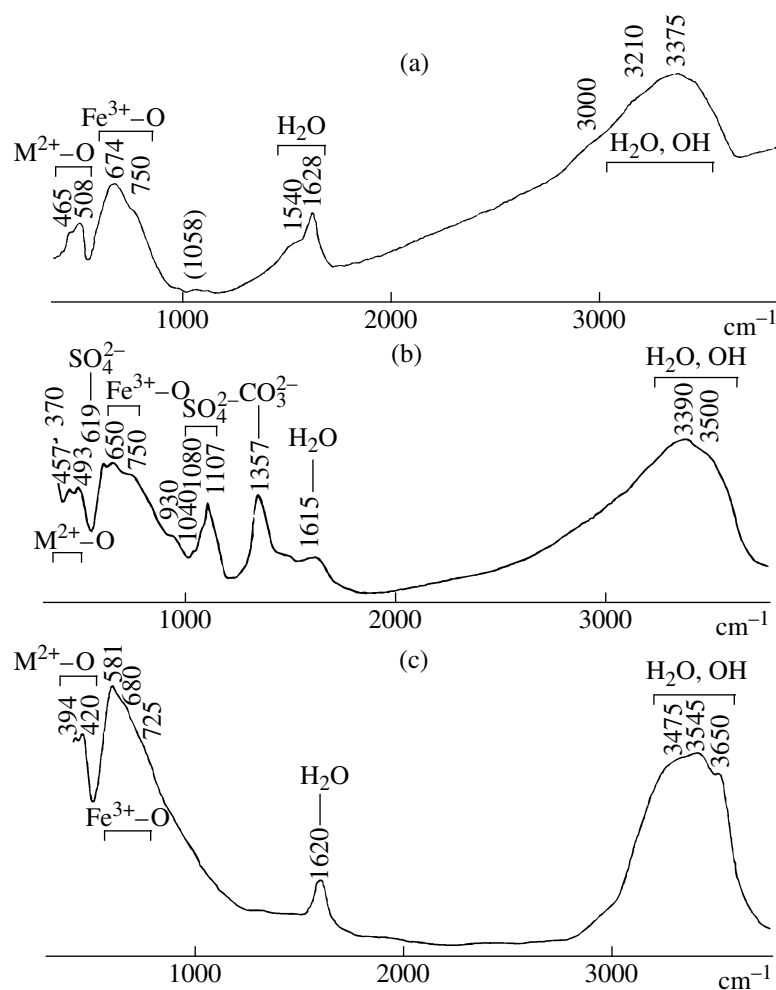
Fine-grained (the size of single grain is no more than 1 μm) segregations of dark green to brown droninoite fill interstices between aggregates of taenite, violarite, and troilite, locally directly contacting with nickelbischofite. As follows from their relationships, the replacement of nickelbischofite with droninoite cannot be ruled out. Monomineralic droninoite segregations between taenite lamellae are about 0.15 × 1 × 1 mm in size. Droninoite is partially and then completely replaced with lepidocrocite toward the margin of the meteorite fragment.

Aggregates of the new mineral are earthy and soft (wrinkled and polished by nail); the Mohs hardness is about 1–1.5. Single droninoite grains are too small for cleavage to be observed, even under a microscope. By analogy with other minerals of the hydrotalcite group, it should be eminent cleavage parallel to (001). The fracture of aggregates of the new mineral is earthy, with dull luster. The calculated density is 2.857 g/cm³.

Under a microscope, droninoite is gray-green and nonpleochroic. Due to the small size of single crystals, only the average refractive index of the mineral was measured: $n = 1.72(1)$.

The wavenumbers in the IR spectrum are (cm⁻¹); sh is shoulder; s is strong band; w is weak band; wavenumbers of the strongest bands are underlined; see figure): 3375s, 3210sh, 3000sh (O–H stretching vibrations), 1628, 1540sh (bending vibrations of H₂O molecules); 1058w (bending vibrations of Fe³⁺...O–H? groups), 750sh, 674s (Fe³⁺–O stretching vibrations), 508, 565 (Ni–O and Fe²⁺–O stretching vibrations). The IR spectra of structurally related iowaite and reevesite are shown in the figure for the purpose of comparison. The characteristic bands assigned to CO₃ ions (range 1350–1500 cm⁻¹) are absent in the IR spectrum of droninoite in contrast to reevesite as its carbonate analogue.

The chemical composition of droninoite (Table 1) was determined with a CamScan MV2300 scanning electron microscope equipped with a YAG detector of



IR spectra of (a) droninoite, (b) SO₄-bearing reevesite, and (c) iowaite.

secondary and back-scattered electrons and EDS with a Link INCA Energy semiconductor (Si–Li) detector operating at 15.7 kV, 0.5 nA, and a scanned area of 16 × 16 μm. Such conditions allowed us to avoid damage to the mineral under the beam. The water content was not measured due to the lack of pure matter. In addition, the H₂O determination with routine procedures (Pienfeld method, TGA) is hampered due to uncertain composition of volatile products of pyrolysis: H₂O, Cl₂, HCl, or H₂ (see Frost et al., 2006) and probable autooxidation of Fe²⁺ by water during thermal decomposition of droninoite. Therefore, the water content was calculated from a deficient total from the microprobe analysis.

The empirical formula calculated on the basis of [(OH) + Cl + H₂O] = 11 (Z = 6) is Ni_{2.16}Fe_{0.75}Fe_{0.97}Cl_{1.62}(OH)_{7.10} · 2.28H₂O; the OH/H₂O ratio is calculated on the basis of charge balance. Taking into account crystallochemical features of the hydrotalcite group minerals, the simplified formula can be written as Ni₃Fe³⁺Cl(OH)₈ · 2H₂O.

The accuracy in determining the chemical composition, average refractive index, and density of droninoite is confirmed by the good compatibility index calculated from the Gladstone–Dale equation: $1 - (K_p/K_c) = 0.025$ (excellent).

CRYSTAL DATA

The submicrometric size of droninoite grains prevents single crystal X-ray study. According to the X-ray powder diffraction pattern and stoichiometry, the new mineral can be confidently attributed to the hydrotalcite group of rhombohedral (ideal symmetry $R\bar{3}m$; unit-cell dimensions are $a \approx 6.1$ – 6.2 , $c \approx 45$ – 47 Å) hydrous hydroxides with additional anions, the structure of which is based on brucite-type stacks.

The X-ray powder pattern of droninoite obtained by the Debye–Sherrer method with an RKU camera 114.6 mm in diameter (Fe_{K α} radiation, Mn filter) is shown in Table 2. Reflections were indexed by analogy with hydrotalcite, Mg₆Al₂(OH)₁₆(CO₃) · 4H₂O.

Table 2. X-ray powder diffraction data on droninoite

I_{meas}	$d_{\text{meas}}, \text{\AA}$	$d_{\text{calc}}, \text{\AA}$	hkl	I_{meas}	$d_{\text{meas}}, \text{\AA}$	$d_{\text{calc}}, \text{\AA}$	hkl
100	7.76	7.697	006	5	1.756	1.751	2.0.20
40	3.88	3.849	0.0.12	5	1.652	1.654	0.2.22
25	2.64	2.669, 2.617	202, 024	10	1.546	1.551	220
5	2.58	2.566	0.0.18	10	1.536	1.539	0.0.30
<5	2.46	2.436	208	5	1.442	1.439	2.2.12
2	2.32	2.323	0.2.10	10b	1.337	1.341, 1.335, 1.328	042, 404, 2.2.18
<5	2.08	2.084	2.0.14	10b	1.289	1.290, 1.283	4.0.10, 0.0.36
15	1.965	1.967	0.2.16	10b	1.216	1.218, 1.212	4.0.16, 0.2.34

Note: broad reflections are labeled by b.

The isostructural character of droninoite and hydroxalcalite is follows from close intensity of most reflections of X-ray powder pattern of the new mineral, primarily series 00 l (Table 2), and theoretical X-ray diffraction pattern of hydroxalcalite calculated from structural data (JCPDS-ICDD database, card 89-0460). At the same time, droninoite differs from the manasseite group minerals, hexagonal analogues ($P6_3/mmc$, $a \approx 6.1$ – 6.2 , $c \approx 15$ – 16 Å) of the talcrite group minerals, in the intensities of major reflections of X-ray diffraction pattern.

All reflections of the recorded X-ray diffraction pattern can be indexed formally in a minor unit cell with the same rhombohedral symmetry $R\bar{3}m$ and dimensions $a = 3.103(1)$, $c = 23.092(9)$ Å. We suggest, however, that it is only a subcell; with it, the fractional value $Z = 3/4$ is deduced even for the simplified formula $\text{Ni}_3\text{Fe}^{3+}\text{Cl}(\text{OH})_8 \cdot 2\text{H}_2\text{O}$. Taking this into account, we chose a unit cell of hydroxalcalite type for droninoite. Its dimensions calculated from the X-ray powder pattern are as follows: $a = 6.206(2)$, $c = 46.184(18)$ Å, $V = 1540.4(8)$ Å³, $Z = 6$. Probable space groups for the new mineral are $R\bar{3}m$, $R3m$, or $R32$.

Note that all synthetic compounds cognate to droninoite (see below) belong to the pyroaurite structural type, i.e., to hydroxalcalite rather than manasseite.

DISCUSSION

The synthetic and technogenic compounds with general formula $(\text{Ni}^{2+}, \text{Fe}^{2+}, \text{Mg})_6\text{Fe}_2^{3+} [\text{SO}_4, \text{CO}_3, \text{Cl}_2(\text{OH})_2](\text{OH})_{16} \cdot n\text{H}_2\text{O}$ structurally cognate to pyroaurite are widely known and repeatedly described in the literature under the generalized name *green rust* (Brindley and Bish, 1976; Refait and Génin, 1992, 1993, 1997; Schwertmann and Fechter, 1994; Chaves, 2005; Chaves et al., 2007). Natural analogues are known for some of these compounds, for example, fougérite $\text{Fe}_6^{3+}\text{Fe}_2^{3+}(\text{OH})_{18} \cdot n\text{H}_2\text{O}$ (Trolard and Bourrie, 2006; Trolard et al., 2007), reevesite $\text{Ni}_6\text{Fe}_2^{3+}(\text{CO}_3)(\text{OH})_{16} \cdot$

$4\text{H}_2\text{O}$ (White et al., 1967; De Waal and Viljoen, 1971; Taylor, 1973), honessite $\text{Ni}_6(\text{SO}_4)(\text{OH})_{16} \cdot 4\text{H}_2\text{O}$ (Heyl et al., 1959; Bish and Livingstone, 1981), iowaite $\text{Mg}_3\text{Fe}^{3+}\text{Cl}(\text{OH})_8 \cdot 2\text{H}_2\text{O}$ (Kohls and Rodda, 1967; Allman and Donnay, 1969; Braithwaite et al., 1994), and other minerals basically attributed to the hydroxalcalite and manasseite groups (Table 3). Technogenic trigonal Fe^{2+} and Fe^{3+} chlorohydroxide having an X-ray diffraction pattern of the pyroaurite type was described under the name chlorsiderite with the idealized formula $4\text{Fe}^{2+}(\text{OH})_2 \cdot \text{Fe}^{3+}\text{OCl} \cdot 4\text{H}_2\text{O}$ from a burnt waste dump near Kopeisk in the Urals (Chesnokov et al., 1996).

Synthetic phases similar to droninoite (some of them are probably identical to the new mineral) are regarded in the literature as varieties of green rust. In particular, compounds with formula $\text{Ni}_{3-x}^{2+}\text{Fe}_x^{3+}\text{Fe}^{3+}\text{Cl}(\text{OH})_8 \cdot n\text{H}_2\text{O}$ ($0 \leq x \leq 3$) and structure of the pyroaurite (hydroxalcalite) type were synthesized as a result of oxidation in air of hydroxides Ni(II)–Fe(II) precipitated from aqueous solutions of Ni(II) and Fe(II) chlorides at variable Fe/Ni values (Refait and Génin, 1997): $\text{NiCl}_2 + \text{FeCl}_2 + \text{NaOH} \rightarrow (\text{Fe}, \text{Ni})(\text{OH})_2 \downarrow + \text{NaCl} \rightarrow (+\text{O}_2) \rightarrow \text{Ni}_{3-x}^{2+}\text{Fe}_x^{3+}\text{Fe}^{3+}\text{Cl}(\text{OH})_8 \cdot n\text{H}_2\text{O}$.

As demonstrated by ICP-AES, Mössbauer spectroscopy, and XRD study, pure chloride-hydroxide with a stable stoichiometric ratio of $\text{Fe}^{3+} : (\text{Fe}^{2+} + \text{Ni}) = 1 : 3$ is formed at an Fe/Ni ratio in solution of less than 1/3 (Refait and Génin, 1997). This empirical relationship was used for partition of iron in droninoite into Fe^{2+} and Fe^{3+} .

When there is relatively low Cl activity, corresponding to a total initial concentration of 0.23 mol/l in $\text{NiCl}_2 + \text{FeCl}_2$ solution, the Cl : $(\text{Fe}_{\text{tot}} + \text{Ni})$ ratio in the green rust that forms can reach 1 : 2 (Refait and Génin, 1993).

At an initial stage of oxidation of $(\text{Fe}^{2+}, \text{Ni})$ bearing green rust, a mixture of lepidocrocite and/or amorphous Fe^{2+} and Ni hydroxides is formed (Refait and Génin, 1997). Therefore, it is noteworthy that lepidocrocite that formed after droninoite is closely associated with amorphous iron hydroxide containing Ni admixture.

Table 3. Comparative data on droninoite and closely related hydrotalcite-group minerals (n.d., not determined)

Property	Droninoite	Reevesite	Honessite	Iowaite
Formula	Ni ₃ Fe ³⁺ Cl · (OH) ₈ · 2H ₂ O	Ni ₆ Fe ₂ ³⁺ (CO ₂) · (OH) ₁₆ · 4H ₂ O	Ni ₆ Fe ₂ ³⁺ (SO ₄) · (OH) ₁₆ · 4H ₂ O	Mg ₃ Fe ³⁺ Cl · (OH) ₈ · 2H ₂ O
Symmetry, space group	Trigonal $R\bar{3}m$, $R3m$, or $R32$	Trigonal $R\bar{3}m$	Trigonal, n.d.	Trigonal $R\bar{3}m$
<i>a</i> , Å	6.206	6.15–6.164	3.083	3.118
<i>b</i> , Å	46.184	45.54–45.61	26.71	24.113
<i>Z</i>	6	3	n.d.	3/4 (? – author's note)
Strong reflections in the X-ray powder pattern, <i>d</i> , Å, (<i>I</i> %)	7.76(10), 3.88(40), 2.64(25), 2.32(20), 1.965(15)	7.63(100), 3.80(73), 2.60(81), 2.30(61), 1.946(48), 1.508(34), 1.537(33)	8.84(100), 4.43(40), 2.65(10), 2.62(20), 2.39(10), 1.54(15),	8.109(100), 4.047(40), 2.639(17), 2.363(27), 2.019(23), 1.530(13), 1.560(8)
Wavenumbers of strong bands in the IR spectrum, cm ⁻¹	3375, 1628, 674, 508	3300–3450, 1355–1365, 620–750, 450–510,	3300–3400, 1080–1110, 600–750, 440–510	3300–3450, 1610–1630, 570–600, 390–450
Optical parameters	<i>n</i> = 1.72 (average for aggregate)	Uniaxial (–) ω = 1.72–1.735 ε = 1.63–1.65	<i>n</i> = 1.72 (average for aggregate)	Uniaxial (–) ω = 1.543–1.561 ε = 1.533–1.543
Density, g/cm ³	2.857 (calc)	2.78–2.87 (calc) 2.80–2.88 (meas)	Not measured	2.04 (calc) 2.09 (meas)
Reference	This study	White et al., 1967; De Waal and Viljoen, 1971; Taylor, 1973; data by authors	Heyl et al., 1959; Bish and Livingstone, 1981; data by authors	Kohls and Rodda, 1967; Allmann and Donnay, 1969; Braithwaite et al., 1994; data by authors

The experimental data on the synthesis of green rust and relationship of Fe²⁺-bearing nickelbischofite with later droninoite suggest that the latter could be a product of replacement of the former.

The question on the source of chlorine incorporated into supergene minerals of the Dronino meteorite is still open. According to Farrington (1903), Heide (1934), Boddhue (1940), Krinov (1955), Mason (1967), Vdovykin (1973), and Hoffer (1974), lawrencite (Fe,Ni)Cl₂ is the major carrier of Cl in iron meteorites; the Cl isotopic composition indicates extraterrestrial origin of this mineral (Goel, 1964). In the presence of water, lawrencite is unstable and can readily transform into oxygen compounds of Fe and Ni. At the same time, Buchwald (1977) and Buchwald and Koch (1995) cast doubt on the meteoritic origin of lawrencite. Multiple findings of hibbingite, Fe₂(OH)₃Cl, in weathered meteorites (it was also identified in Dronino) are regarded as an indication that Cl was introduced under terrestrial conditions. We leave room for both meteorite and terrestrial sources of Cl for the supergene minerals from Dronino.

Nickel chlorides (nickelbischofite, droninoite) are so far known only in the central part of a single fragment of the Dronino meteorite. In the intermediate zone of the same fragment, where sulfides are oxidized to a greater degree, and in other fragments of this meteorite, nickel and chlorite underwent partition as a result of weathering; Ni is concentrated in low-Cl varieties of reevesite and honessite, whereas Cl, in purely ferrous hibbingite. Miyata (1983) suggested that additional bivalent anions have stronger chemical affinity to the pyroaurite-type phases than monovalent anions. The formation of droninoite appears to be facilitated by local low activity of sulfate ions due to insignificant oxidation of associated sulfides in contrast to the intermediate zone where honessite, a high-sulfate analogue of droninoite, was formed.

In conclusion, we comment on the classification of the hydrotalcite and manasseite groups. They comprise trigonal (rhombohedral) and hexagonal minerals, respectively, with layered structure, identical topology, and general formula A₆²⁺ M₂³⁺ (OH)₁₆X · 4H₂O, where

A = Mg, Ni, Fe, M = Al, Fe, Cr, Mn, Co, and X = CO₃, SO₄, Cl₂, (OH)₂. Minerals of identical composition attributed to different groups are traditionally regarded as polymorphs (for example, hydrotalcite and manasseite having the same composition Mg₆Al₂(OH)₁₆(CO₃) · 4H₂O), although they correspond to the modern definition of polytypes. In spite of the very high OH/X ratio, which is 8 for monovalent X anions and 16 for bivalent anions, and the distinct structural affinity to brucite, Mg(OH)₂, in modern mineralogical classifications, the numerous CO₃-bearing members of these groups are attributed to carbonates; SO₄-bearing members (honessite), to sulfates; Cl-bearing members (iowaite, woodalite), to chlorides; and only fougérite with X = (OH)₂, to hydroxides. The attribution of all these minerals together with structurally cognate coalingite, chlormagaluminite, hydrohonessite, wermlandite, shigaite, and mountkeithite with different proportions A : M : (OH) : X : H₂O to hydroxides with additional X anions is much more valid in terms of both chemistry and crystal chemistry.

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