

# Reynoldsite from the Oxidation Zone of the Tochilnogorskoe Deposit (Middle Urals)—The First Find in Russia

V. S. Ponomarev<sup>a,\*</sup>, Yu. V. Erokhin<sup>a</sup>, and S. N. Britvin<sup>b</sup>

<sup>a</sup>Zavaritsky Institute of Geology and Geochemistry, Ural Branch, Russian Academy of Sciences, Yekaterinburg, Russia

<sup>b</sup>St. Petersburg State University, St. Petersburg, Russia

\*e-mail: p123v@yandex.ru

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**Abstract**—Our study of the historical Tochilnogorskoe deposit of refractory stone (Tochilnaya Mountain) in the Rezhevsky district, Middle Urals, has revealed for the first time in Russia a very rare lead chromate oxide, reynoldsite  $\text{Pb}_2\text{Mn}_2^{4+}\text{O}_5(\text{CrO}_4)$ , which was previously discovered in only three locations worldwide. Reynoldsite was found in a muscovite aggregate with secondary Pb–Mn oxides in fractures of veiny quartz, as well as in druse cavities of quartz veins, with crocoite and goethite pseudomorphs after pyrite. The mineral forms elongated, plate-like individuals up to 10  $\mu\text{m}$  in size and monomineralic, tangled fibrous aggregates up to 0.3 mm in size overgrowing cesarolite, muscovite, and quartz. The empirical formula of reynoldsite demonstrates the range  $\text{Pb}_{2.00-2.06}\text{Mn}_{2.01-2.06}^{4+}\text{O}_5(\text{Cr}_{0.96-0.98}\text{O}_4)$ . The strongest reflections of the powder X-ray diffraction pattern [ $d$ , Å( $I$ )] are: 3.060 (100), 3.233 (75), 4.688 (68), 3.004 (64), 3.351 (54), 2.498 (53), 2.912 (44). The mineral is triclinic, space group  $P-1$ ; unit cell parameters are:  $a = 5.013(3)$  Å,  $b = 7.589(9)$  Å,  $c = 10.254(5)$  Å;  $\alpha = 91.86(6)^\circ$ ,  $\beta = 99.65(4)^\circ$ ,  $\gamma = 109.08(6)^\circ$ ;  $V = 361.9(4)$  Å<sup>3</sup>,  $Z = 2$ . The Raman spectrum is presented for the first time for reynoldsite, it can be used to identify the mineral. Reynoldsite is a supergene mineral formed as a result of the weathering of primary minerals (galena in quartz and chromespinelides in host ultramafic rocks).

**Keywords:** reynoldsite, chromate, Raman spectrum, quartz veins, Tochilnogorskoe deposit of refractory stone, Tochilnaya Mountain, Middle Urals

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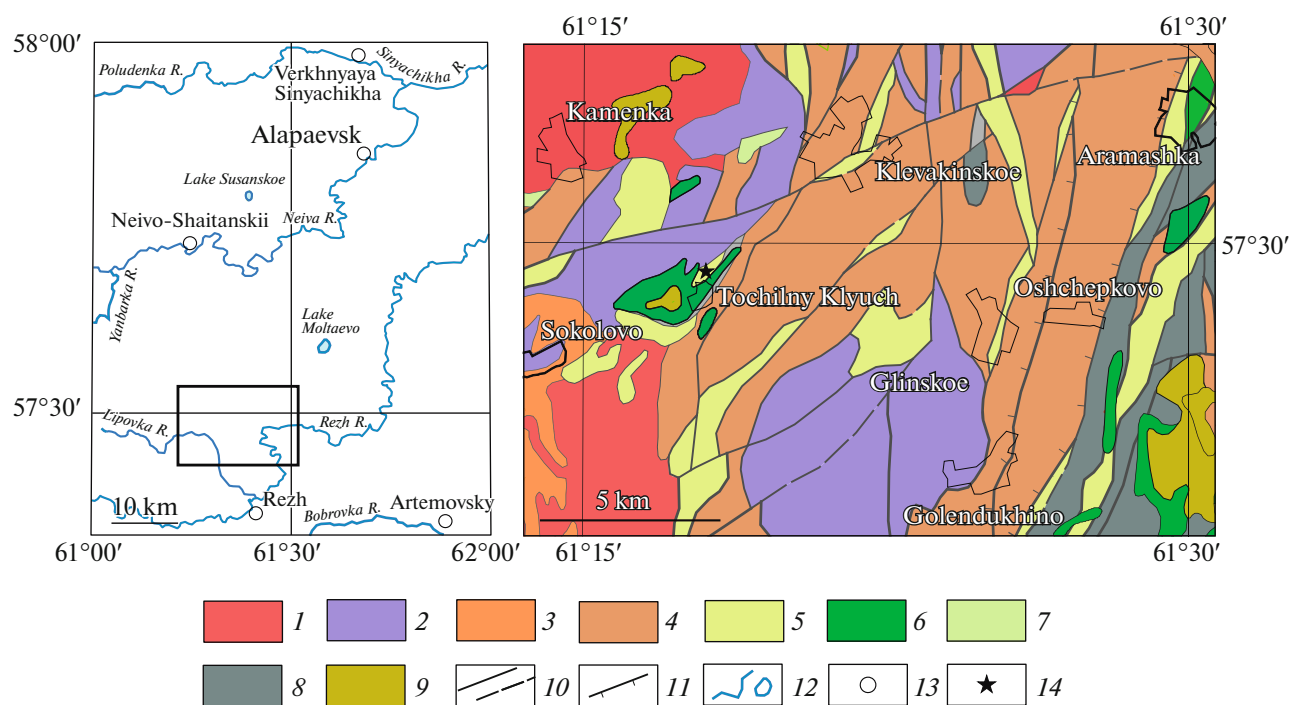
Today, there are sixteen known natural lead chromate minerals: vauquelinite, hemihedrite, georgerobinsonite, iranite, cassedanneite, crocoite, macquartite, molybdo-fornacite, reynoldsite, santanaite, phoenicochroite, fornacite, chromviskontite, chromschieffelinite, evanichite, and embreyite. They all are rare and even exceptionally rare minerals, some of which are found only in one place in the world. In Russia, the finds of seven lead chromate minerals are known: crocoite [1], vauquelinite [2], cassedanneite [3], embreyite [4], fornacite [5], phoenicochroite [6], and chromviskontite [8]. All these minerals, except for fornacite and chromviskontite, were first described at the Berezovskoye gold deposit in the Middle Urals [7]. Our work describes the first find of reynoldsite in Russia, in the historical Tochilnogorskoe deposit of refractory stone in the Middle Urals.

The Tochilnogorskoe deposit, also known as Tochilnaya Mountain or “Baronial Pits” (the Demidov family owned the mines for a long time), has been

known since 1704. The deposit was mined in the 18th–19th centuries. Quartz was extracted here, which was used to construct blast furnaces for the Urals factories and others [9]. The prominent naturalist P.S. Pallas visited the Tochilnogorskoe deposit in 1770. In his work, he described the specimens found on Tochilnaya Mountain as “...the red lead spar (the term for crocoite used at that time, the authors’ note), which had not been found anywhere else in the world, except at the Berezovskoye mines” [9]. Later, the mineralogy of the Tochilnogorskoe deposit was studied by V.I. Vernadsky, and recently by A.F. Bushmakin, V.I. Kainov, and D.A. Khanin [10–12]. This deposit is known for its finds of excellent specimens with crocoite crystals and their druses on clear quartz or beresite.

## MATERIALS AND METHODS

A powder XRD pattern of reynoldsite was obtained at the Center for X-ray Diffraction Studies, the Research Park of St. Petersburg State University,



**Fig. 1.** Schematic geological map of the Rezhevsky district [15] with additions by the authors. Legend: 1, leucogranites, biotite granites, granodiorites, and quartz diorites; 2, dunites, harzburgites, and serpentines; 3, monzodiorites and monzogabbros; 4, volcanogenic-sedimentary rocks: basalts, andesibasalts, and volcanic tuffs; 5, East Uralian tectonic complex, polymictic serpentinite mélange (talc-carbonate rocks, serpentinites, diorites, granodiorites, metavolcanics, and quartzites in lenses); 6, gabbros and gabbrodiorites; 7, biotite-quartz, sericite-quartz schists with lenses of quartzite, tuff sandstones, and marbles; 8, limestones, sandstones, and tuff sandstones; 9, clays and quartz sands (Quaternary deposits); 10, tectonic faults; 11, thrusts; 12, rivers and lakes; 13, populated areas; 14, Tochilnogorskoe deposit.

using a Rigaku RAXIS diffractometer with a cylindrical detector, Debye-Scherrer geometry,  $r = 127.4$  mm,  $\text{CoK}\alpha$ - radiation, 40 kV, 15 mA, exposure for 15 min. Data were processed using *osc2xrd* [13] and *STOE WinXPOW* (Stoe & Cie GmbH), analyst S.N. Britvin. Other analyses were performed at the Laboratory of Physicochemical Methods of Studies at the Institute of Geology and Geochemistry, Ural Branch, Russian Academy of Sciences, the city of Yekaterinburg. The chemical composition of reynoldsite was analyzed in the specimens polished and coated with carbon using a CAMECA SX 100 electron-probe microanalyzer, analyst V.A. Bulatov. Raman spectrum of reynoldsite was obtained from the polished surface of reynoldsite crystals using a LabRam HR Evolution spectrometer (Horiba Scientific) with a solid-state laser at a 514 nm wavelength, analyst E.A. Pankrushina. Mineral images were obtained in back-scattered electron mode with a TESCAN MIRA LMS (S6123) SEM equipped with an INCA Energy 450 X-Max 80 (Oxford Instruments) attachment and AZtecOne software, analyst L.V. Leonova.

## STUDY OBJECT

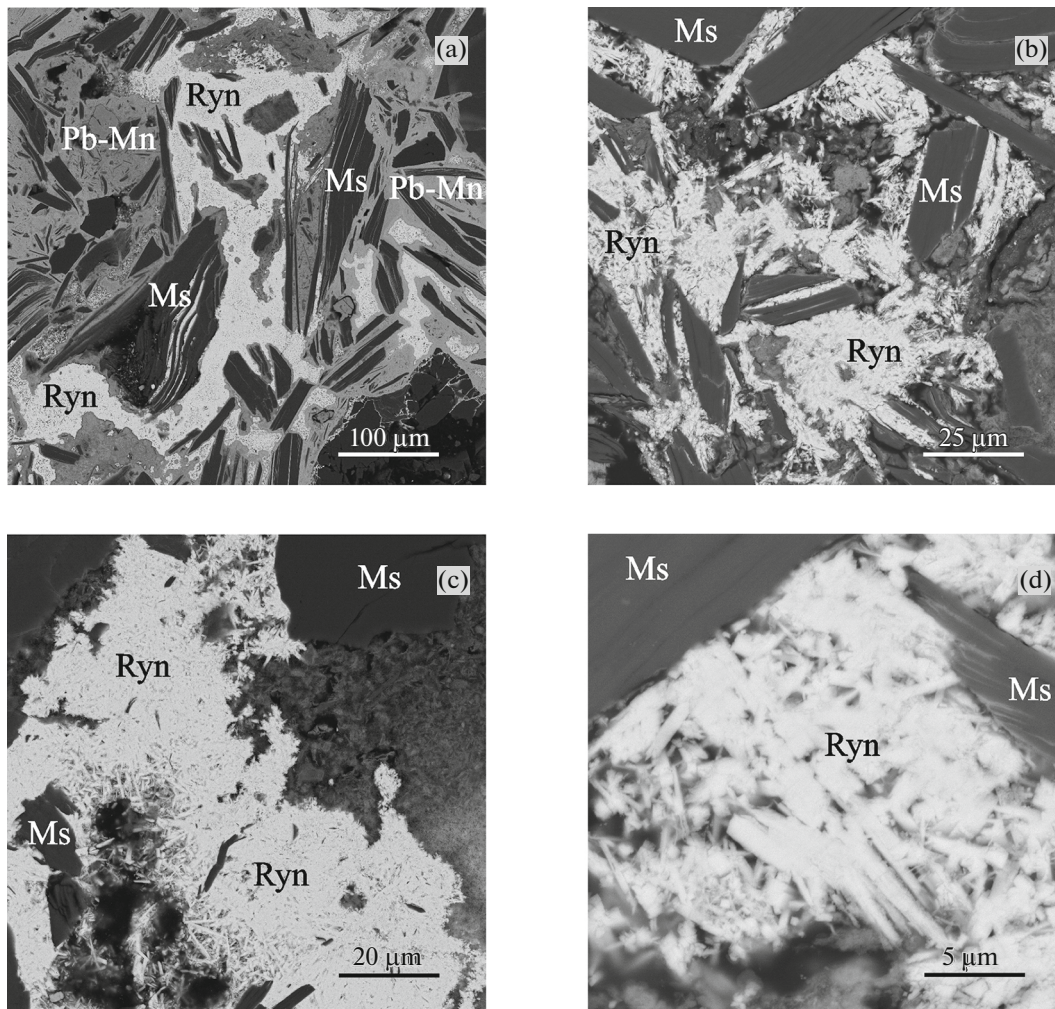
The Tochilnogorskoe deposit of refractory stone is located in the Rezhevsky district of Sverdlovsk oblast,

approximately 500 m north of the village of Tochilny Klyuch. Today, two relatively large open pits up to 50–60 m deep, several pits, trenches, and waste dumps are preserved at this site in the dense forest.

Geologically, the deposit is located within the eastern wing of the Murzinsky-Aduisky anticlinorium, between the Murzinsky and Sokolovsky granite massifs. It is represented by a series of beresitised granite-porphry dykes, intersecting metamorphosed volcanogenic-sedimentary rocks, quartzites, and serpentinites that were transformed into talc-rich and talc-carbonate rocks (Fig. 1). The beresitised dykes contain narrow (up to 1 m) stair-type quartz veins composed of massive and drusoid medium-grain quartz. It is noted that sulfide-quartz mineralization is accompanied by negligible gold content [11]. Rare inclusions of galena, pyrite, sphalerite, chalcopryrite, and very rare fahlore tetrahedrite-(Cd) are observed in the quartz veins [14]. Supergene minerals are crocoite, goethite, anglesite, cerussite, pyromorphite, Mn oxides, and others.

## RESULTS AND DISCUSSION

At the Tochilnogorskoe deposit, we discovered a very rare lead chromate oxide, reynoldsite, which has



**Fig. 2.** Reynoldsite from the oxidation zone of the Tochilnogorskoe deposit: (a, b) drainage aggregate of reynoldsite in a cesarolite-muscovite aggregate; (c, d) elongated individual crystals of reynoldsite in a cavity of a cesarolite-muscovite aggregate. Ryn is reynoldsite, Ms is muscovite, Pb-Mn is cesarolite. SEM image in reflected electrons.

the formula  $\text{Pb}_2\text{Mn}_2^{4+}\text{O}_5(\text{CrO}_4)$ . There are only three known locations of this mineral in the world. Reynoldsite was first described at the Blue Bell deposits in San Bernardino County, California (USA) and at the Red Lead mine in Dundas (Tasmania, Australia). The same study also indicates that reynoldsite was encountered at the Milford mine No. 3 in Goodsprings County, Nevada (USA), though its description is not provided in the published literature [16, 17].

Reynoldsite was discovered at the Tochilnogorskoe deposit while studying the samples collected during the field works in 2023–2024. The mineral was present in a muscovite aggregate with secondary Pb–Mn oxides (cesarolite, coronadite) within fractures of vein quartz, as well as in druse cavities encrusted by clear quartz together with crocoite and goethite pseudomorphs after pyrite. Reynoldsite forms elongated platy individuals up to 10  $\mu\text{m}$  in length (Fig. 2d) and monomineralic, fibrous-diverse aggregates

(Figs. 2a–2c) up to 0.3 mm that perch cesarolite or aggregate within it. Such clusters of reynoldsite and cesarolite with coronadite frequently occur in the interstices of thin muscovite sheets and their druses on the vein quartz surface.

The X-ray study of the mineral was performed on a specimen sized  $\sim 0.3 \times 0.3 \times 1.0$  mm, consisting of a dense, fine-grained aggregate of reynoldsite with a small amount of muscovite (Table 1). The triclinic (space group  $P-1$ ) unit-cell parameters calculated from the powder data for reynoldsite from the Tochilnogorskoe deposit are as follows:  $a = 5.013(3)$  Å,  $b = 7.589(9)$  Å,  $c = 10.254(5)$  Å;  $\alpha = 91.86(6)^\circ$ ,  $\beta = 99.65(4)^\circ$ ,  $\gamma = 109.08(6)^\circ$ ;  $V = 361.9(4)$  Å<sup>3</sup>,  $Z = 2$ . These results are in good agreement with the reference data for reynoldsite from the Red Lead mine [16].

We obtained the data of the chemical composition of reynoldsite using a wave-dispersion electron-probe microanalyzer, which showed that the mineral has a

**Table 1.** Powder X-ray diffraction data ( $d$ , Å) for reynoldsite from the Tochilnogorskoe deposit

$I_{\text{meas}}$	$D_{\text{meas}}$	$I_{\text{calc}}$	$D_{\text{calc}}$	h k l	$I_{\text{meas}}$	$D_{\text{meas}}$	$I_{\text{calc}}$	$D_{\text{calc}}$	h k l
15	5.58	9	5.582	0 1 1	10	2.043	9	2.043	-2 3 0
27	5.03	14	5.032	0 0 2			2	2.037	-2 3 1
68	4.688	46	4.688	-1 1 0			7	2.027	2 -2 2
		2	4.652	1 0 0			3	2.020	2 1 0
25	4.556	14	4.564	-1 0 1			5	2.012	-2 -1 2
		4	4.475	-1 1 1			5	1.989	-1 0 5
22*	4.303	4	4.308	0 -1 2			9	1.987	1 -2 4
		3	3.949	1 0 1			4	1.974	2 0 2
		19	3.943	0 1 2	17	1.974	12	1.970	2 -3 1
27	3.673	27	3.674	-1 1 2	16	1.951	9	1.952	-2 3 2
3	3.566	4	3.570	0 2 0	6	1.936	5	1.933	-2 1 4
		35	3.469	0 -2 1			14	1.898	-1 -3 1
		2	3.450	-1 2 0	14	1.898	10	1.898	-2 -1 3
		5	3.423	-1 -1 1			11	1.894	-1 4 0
34	3.411	31	3.408	1 1 0			3	1.861	0 3 3
54*	3.351	24	3.355	0 0 3			2	1.853	-1 -3 2
16	3.310	21	3.314	-1 2 1			4	1.847	2 -1 3
		21	3.270	0 2 1			2	1.845	1 2 3
75	3.233	80	3.229	1 -1 2			4	1.798	1 3 1
		13	3.215	1 -2 1			3	1.787	0 -4 1
		8	3.152	0 -1 3			4	1.786	1 -4 2
		3	3.134	1 0 2			3	1.771	-1 3 4
28	3.100	20	3.097	-1 -1 2			4	1.769	1 -1 5
100	3.060	52	3.062	1 1 1	18	1.771	15	1.766	1 -3 4
		48	3.050	0 -2 2	24*	1.763	8	1.762	2 0 3
64	3.004	48	3.003	-1 0 3			6	1.760	-1 4 2
		15	2.933	0 1 3			6	1.755	-1 -3 3
44	2.912	39	2.913	-1 2 2			4	1.733	-2 -2 1
22*	2.793	7	2.791	0 2 2			3	1.730	0 4 1
		5	2.645	-1 -1 3			8	1.707	-2 1 5
		4	2.609	1 1 2	26*	1.708	15	1.701	-1 -2 5
		5	2.576	1 -1 3	11	1.685	5	1.683	2 -3 3
		4	2.568	0 -2 3			4	1.668	0 -1 6
		6	2.516	0 0 4			4	1.659	-3 2 1
53	2.498	40	2.499	-2 1 1			3	1.657	-2 3 4
		8	2.486	-1 3 0			2	1.654	0 3 4
		3	2.486	-1 -2 1	19	1.648	4	1.646	-2 -2 3
		4	2.467	-1 2 3			8	1.641	0 -4 3
27*	2.413	12	2.412	1 -3 1			2	1.635	-2 2 5
		11	2.380	0 3 0			7	1.632	2 2 1
		8	2.369	-1 -2 2			2	1.631	2 -1 4
		3	2.367	-2 0 1	17	1.631	11	1.630	1 2 4
14*	2.344	3	2.344	-2 2 0			6	1.615	2 -2 4
		4	2.338	0 2 3			2	1.609	1 1 5
15	2.330	6	2.326	2 0 0			2	1.607	2 -4 2

Table 1. (Contd.)

$I_{\text{meas}}$	$D_{\text{meas}}$	$I_{\text{calc}}$	$D_{\text{calc}}$	h k l	$I_{\text{meas}}$	$D_{\text{meas}}$	$I_{\text{calc}}$	$D_{\text{calc}}$	h k l
24	2.307	19	2.306	0 1 4			5	1.600	0 1 6
14	2.285	6	2.282	-2 0 2			3	1.585	-2 -1 5
10	2.231	5	2.232	-1 3 2	22	1.578	8	1.579	3 -2 1
		6	2.228	-1 -1 4			3	1.578	-3 0 1
		5	2.226	1 -3 2			2	1.576	0 -2 6
35*	2.200	8	2.198	1 1 3			5	1.573	-3 2 3
12	2.174	10	2.178	2 0 1			2	1.570	3 -1 1
		9	2.171	-2 1 3	11	1.566	5	1.567	-1 3 5
12	2.154	11	2.154	0 -2 4			3	1.566	-2 4 3
18*	2.109	6	2.107	1 -1 4			4	1.551	3 0 0
		6	2.084	2 -1 2			5	1.539	-1 2 6
17	2.079	11	2.082	-1 2 4			3	1.521	-3 0 3
		3	2.079	1 2 2					
		14	2.078	0 3 2					

\* The lines overlapping with the muscovite reflections. The calculated reflection intensities and interplanar spacings derived from the atomic coordinates taken from the structural data for reynoldsite [16] and the unit-cell parameters refined using the powder X-ray diffraction pattern. The calculated intensities are normalized to  $I_{\text{calc}}(111) + I_{\text{calc}}(0-22) = 100$ . The calculated reflections with  $I_{\text{calc}} < 1.5$  are not included in Table 1.

stable chemical composition that corresponds well to the stoichiometry of reynoldsite. The mean of eight analyses (wt %, standard deviation is presented in parentheses) is:  $\text{CrO}_3$  13.18 (0.14),  $\text{MnO}_2$  23.93 (0.32),  $\text{PbO}$  61.92 (0.42), total 99.03 (0.30). The average empirical formula of reynoldsite calculated for nine oxygen atoms is  $\text{Pb}_{2.04}\text{Mn}_{2.02}^{4+}\text{O}_5(\text{Cr}_{0.97}\text{O}_4)$ . Taking the small compositional variation into account, the empirical formula of the mineral is written as  $\text{Pb}_{2.00-2.06}\text{Mn}_{2.01-2.06}^{4+}\text{O}_5(\text{Cr}_{0.96-0.98}\text{O}_4)$ . Reynoldsite

from the Blue Bell deposit is found to contain trace elements, such as Al, Si, Cl, S, Ca, Fe, Cu, As, and Te. Reynoldsite from the Red Lead mine had trace amounts of Na, Ba, Ca, Co, Cu, Zn, Al, and As. The published works report a lowered total sum of chemical analyses for reynoldsite, which is caused by the difficult measurements in thin tiny crystals rather than by the presence of  $(\text{OH})^-$  or  $\text{H}_2\text{O}$ . Chemically, reynoldsite from the Tochilnogorskoe deposit is close to the mineral from the Red Lead mine, except for the presence of strontium: 0.51 wt % SrO in the latter [16]. The presence of strontium in Tasmanian reynoldsite may be related to the occurrence of fine-dispersed coronadite [16].

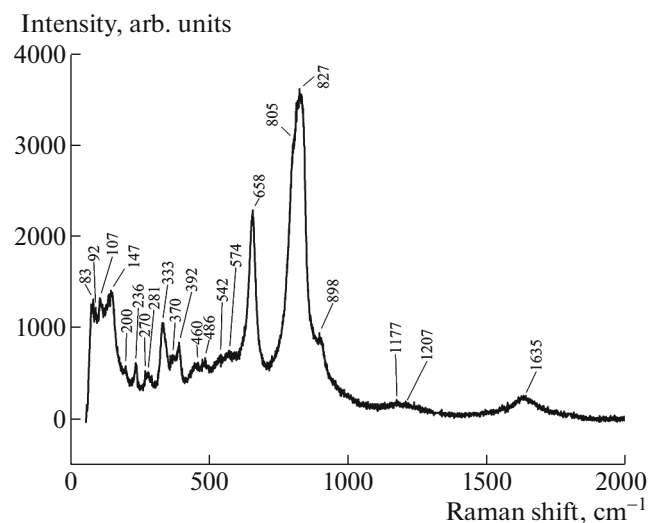


Fig. 3. Raman spectrum of reynoldsite from the Tochilnogorskoe deposit.

We obtained several Raman spectra of reynoldsite from the Tochilnogorskoe deposit. They all are similar. One of them is shown in Fig. 3. It was obtained from the polished surface of an individual crystal sized  $8 \times 4 \mu\text{m}$ . The measurement range was from 50 to  $4000 \text{ cm}^{-1}$ , all bands were located in the  $83-1635 \text{ cm}^{-1}$  range. In the  $4000-2000 \text{ cm}^{-1}$  range, the mineral spectrum had no bands, which indicates the absence of water in the mineral. The Raman spectrum of reynoldsite is characterized by the following peaks ( $\text{cm}^{-1}$ ): 83, 92, 107, 147, 200, 236, 270, 281, 333, 370, 392, 460, 486, 542, 574, 658, 805, 827, 898, 1177, 1207, and 1635.

By using the published data for Raman spectra of natural lead chromates [18, 19], we interpret the reynoldsite spectrum. The bands within  $805-898 \text{ cm}^{-1}$  correspond to the  $\text{Cr}^{6+}-\text{O}$  vibrations in the tetrahedra ( $\text{CrO}_4$ ). The bands within the  $333$  and  $658 \text{ cm}^{-1}$  range

most likely related to the  $\text{Mn}^{4+}\text{-O}$  vibrations in the  $\text{MnO}_6$  octahedra, like in layered oxides of Mn (ranciéite, chalcophanite), being similar in structure to reynoldsite [16, 20]. The bands below  $333\text{ cm}^{-1}$  have a complex nature and are related to the valence vibrations of  $\text{Pb-O}$  bonds. A broad weak band with a peak at  $1635\text{ cm}^{-1}$  is recorded in the reynoldsite spectrum, which is consistently attributed to  $\text{H-O-H}$  vibrations. A similar weak peak in the  $1640\text{--}1680\text{ cm}^{-1}$  range was present in the Raman spectra of the other lead chromates (evanichite, georgerobinsonite, and vauquelinite), which do not contain  $\text{H}_2\text{O}$  in their chemical formulas. It was suggested that these minerals can contain small amounts of  $\text{H}_2\text{O}$  [19].

Reynoldsite is a supergene mineral formed as a result of the weathering of primary minerals, including oxides and sulfides, in the presence of acidic groundwaters. At the Blue Bell deposit, reynoldsite is associated with coronadite, fluorite, goethite, opal, pyromorphite, quartz, and wulfenite. At the Red Lead mine, the mineral is associated with coronadite, lithiophorite, and crocoite [16]. At the Tochilnogorskoe deposit, reynoldsite is found in the specimens with cesarolite, coronadite, crocoite, and goethite. The formation of reynoldsite and the entire supergene association particularly is related to the fact that the galena-bearing quartz veins cut through ultramafic rocks, which are characterized by the presence of accessory chromespinelide. The oxidation of this sulfide occurs in the hypergenesis zone, involving the removal of lead, while the ultramafic rocks supply chromium and manganese. As a result, the supergene deposit enriched in lead, chromium, and manganese is formed.

Thus, a very rare lead chromate oxide, reynoldsite, was found for the first time in Russia in the historical Tochilnogorskoe deposit of refractory stone in Middle Urals. The powder X-ray analysis of the mineral was performed to reliably identify the mineral and determine unit-cell parameters. The study of the chemical composition showed its stable composition and the absence of any impurities. The Raman spectrum was presented for reynoldsite for the first time, which can be used for mineral identification. Reynoldsite is extremely rare at the Tochilnogorskoe deposit. It is a supergene mineral formed as a result of the weathering of primary minerals.

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#### CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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