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FLEETITE, $Cu_2RhIrSb_2$, A NEW SPECIES OF PLATINUM-GROUP MINERAL FROM THE MIASS PLACER ZONE, SOUTHERN URALS, RUSSIA

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Abstract

Fleetite, $Cu_2RhIrSb_2$, a new species of platinum-group mineral (PGM), was discovered intergrown with an Os–Ir–Ru alloy in the Miass Placer Zone (Au–PGE), southern Urals, Russia. A single grain 50 μm across was found. Osmium, ruthenium, and iridium are the main associated minerals; also present are Pt–Fe alloys, laurite, Sb-rich irarsite, Rh-rich tolovkite, kashinite, anduoite, ferronickelplatinum, heazlewoodite, PGE-bearing pentlandite and digenite, as well as micrometric inclusions of forsterite ($Fo_{93.7}$), Cr-rich spinel, and Mg-rich edenite. Under reflected light, fleetite is light gray; it is opaque, isotropic, non-pleochroic, and non-bireflectant. We report reflectance values measured in air. A mean of seven point-analyses (wavelength-dispersive spectrometry) gave Cu 13.93, Ni 8.60, Fe 0.10, Ir 28.07, Rh 7.91, Ru 1.96, Sb 39.28, total 99.85 wt.%, corresponding to $(Cu_{1.41}Ni_{0.58}Fe_{0.01})_{\Sigma 2.00}(Rh_{0.49}Ni_{0.36}Ru_{0.12})_{\Sigma 0.97}Ir_{0.95}Sb_{2.08}$ on the basis of six atoms per formula unit, taking into account the structural results. The calculated density is 10.83 g/cm^3. Single-crystal X-ray studies show that fleetite is cubic, space group $Fd\bar{3}m$ (#227), $a = 11.6682(8)$ Å, $V = 1588.59(19)$ Å^3, and $Z = 16$. A least-squares refinement of X-ray powder-diffraction data gave $a = 11.6575(5)$ Å and $V = 1584.22(19)$ Å^3. The strongest five reflections in the powder pattern [d in Å(I)(hkl)] are:

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6.70(75)(111), 4.13(100)(220), 3.52(30)(311), 2.380(50)(422), 2.064(40)(440). Results of synchrotron micro-Laue diffraction experiments are consistent [$a = 11.66(2)$ Å]. The crystal structure of fleetite was solved and refined to $R = 0.0340$ based upon 153 reflections with $F_o > 4\sigma(F_o)$. It is isotypic with $Pd_{11}Bi_2Se_2$ and best described as intermetallic, with all metal atoms in 12-fold coordination. Fleetite and other late exotic phases were formed by reaction of the associated alloy phases with a fluid phase enriched in Sb, As, and S in circulation in the cooling ophiolite source-rock. The mineral is named after Michael E. Fleet (1938–2017) in recognition of his significant contributions to the Earth Sciences.

Keywords: fleetite, $Cu_2RhIrSb_2$, new mineral species, platinum-group elements, ore minerals, platinum-group minerals, ophiolites, placer deposits, Miass placer zone, southern Urals, Russia.

INTRODUCTION

The Miass placer deposits extend over 90 km along the upper Miass River, located south of Miass (55°0′ N, 60° 06′ E) in the Chelyabinskaya oblast (region), southern Urals, Russia. Historically, the placers are well-known as a source of gold. They also contain platinum-group minerals (PGM), as the watershed drains chromite-mineralized ophiolitic complexes. Three minerals, miassite ($Rh_{17}S_{15}$), palladodymite [$(Pd,Rh)_2As$], and polkanovite ($Rh_{12}As_7$), have been discovered so far in heavy-mineral concentrates of the Miass placers. We report here on a fourth PGM, fleetite, $Cu_2RhIrSb_2$, also found in a concentrate. The new species (IMA 2018-073b) was approved by the Commission on New Minerals, Nomenclature and Classification (CNMNC) of the International Mineralogical Association (IMA) (Barkov *et al*. 2020). It occurs as a single small grain intergrown with a placer grain of Os–Ir–Ru alloy recovered from the Miass Placer Zone. We describe the physical and chemical properties of fleetite and establish its crystal structure.

OCCURRENCE AND ASSOCIATED MINERALS

The Miass Placer Zone is distributed over about 120 km of strike length along the riverbed of the Miass River (Zaykov *et al*. 2016). The Middle Miass placer (Fig. 1) is the major Au–PGM-bearing deposit. The drainage basin consists of several formations, including a prominent ophiolitic mélange, with bodies and fragments of ultramafic rocks.

Fleetite occurs as a single small grain (~50 × 25 μm) in contact with a placer grain of Os–Ir–(Ru) alloy (Figs. 2a, 2b, 3). The grain of fleetite was found during a careful examination of hundreds of placer grains ranging from 0.2 to ~1 mm in size collected from the area. The alloy minerals of the Os–Ir–Ru system, namely osmium, iridium, and ruthenium, are dominant constituents (>95%) in the concentrates of platinum-group minerals. An overall prominent enrichment in ruthenium in the Miass River placers, documented by Barkov *et al*. (2018) and reflected in the newly discovered minerals, is consistent with the inference of ophiolitic source-rocks.

The main phases associated with fleetite are Os–Ir–Ru alloys, *i.e*., the minerals osmium, ruthenium, and

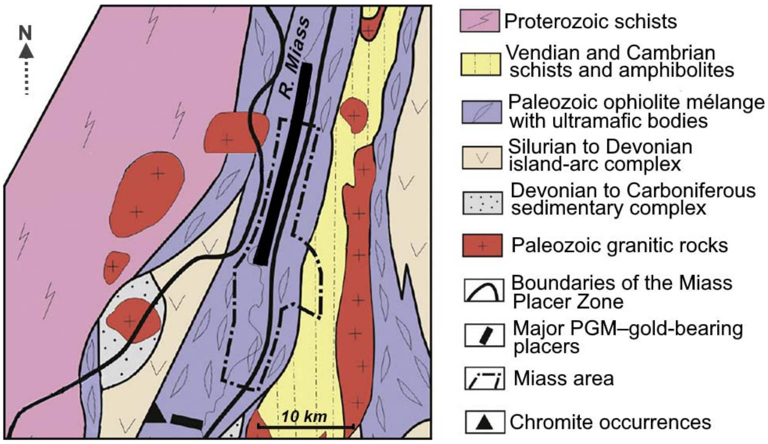

FIG. 1. A schematic geological map of the Middle Miass Au–PGM-bearing placer in the Miass Placer Zone, modified after Lennykh & Nestoyanova (1956) and Zaykov *et al*. (2016).

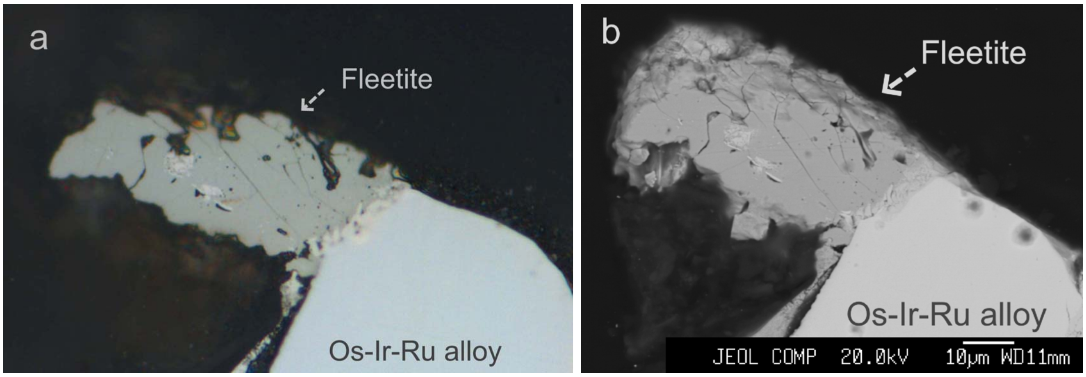

FIG. 2. Optical photograph (a) and back-scattered electron (BSE) image (b) showing the grain of fleetite from the Miass Placer Zone, southern Urals. The difference in the shape of the grain observed between the reflected-light image and BSE image of fleetite reflects the fact that the BSE image displays an unpolished surface at a deeper level covered by the host epoxy.

iridium. Subordinate minerals are represented by Pt–Fe alloys; less common and rare minerals of the platinum-group elements (PGE) include laurite, irarsite (Sb-rich), tolovkite (Rh-rich), kashinite, anduoite, and ferronickelplatinum. The associated base-metal sulfide minerals are heazlewoodite, pentlandite (PGE-bearing), and digenite. In addition, there are micrometric inclusions of forsterite (Fo$_{93.7}$), members of the chromite–magnesiochromite series, and amphibole (a high-Mg variety of edenite) hosted by grains of PGM. Mineral associations observed at Miass also include species that were presumably derived from non-ophiolite lode sources, such as ilmenite, gold, Au–Ag–Hg alloys (amalgam), chlorargyrite, and daubréeite (F-bearing).

ETYMOLOGY AND CONSERVATION

The mineral is named in honor of Michael Edward Fleet (January 4, 1938 – December 6, 2017), professor of Mineralogy at the Department of Earth Sciences, University of Western Ontario (now Western University), London, Ontario, in recognition of his important contributions in an unusually wide range of subdisciplines of the Earth Sciences. Appropriate keywords summarizing his eclectic interests might include: sulfides, crystal structures, phase-equilibria in sulfide systems, platinum-group minerals, apatite supergroup minerals, thermodynamics, spectroscopic techniques, and the structure of silicate melts.

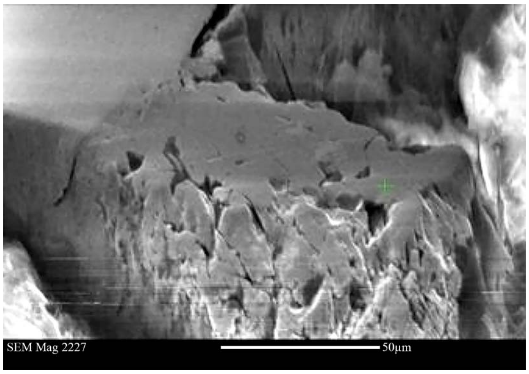

FIG. 3. An SEM image of the specimen of fleetite.

The holotype specimen of fleetite (catalogue # 1/41/11 00 2) is deposited at the State Museum of Geology of Central Siberia, Krasnoyarsk, Russia. The mounted crystal used to establish the structure of fleetite is kept at the Dipartimento di Scienze della Terra, Università di Firenze, Italy.

PHYSICAL PROPERTIES

Fleetite is a light gray mineral; its streak was not observed owing the paucity of material. It is metallic, opaque, and brittle. Fluorescence, cleavage, fracture, and parting were not observed. Its hardness (Mohs or micro-indentation) and density could not be measured owing to the unsuitable grain-size. Its calculated density, 10.83 g/cm^3, was calculated using the single-crystal X-ray data and the empirical chemical formula.

Under reflected light, fleetite is light gray. It is isotropic, non-pleochroic, and non-bireflectant. Internal reflections were not observed. Reflectance values of fleetite (Table 1) were measured in air using a Lomo MSFU-K microspectrophotometer with a SiC standard. Fleetite is a moderately reflective species of PGM. Its reflectivity is comparable to that of cooperite, PtS (*cf.* $R = 47\%$ for fleetite *versus* 45–46% for cooperite at 600 nm; Criddle & Stanley 1985).

TABLE 1. REFLECTANCE VALUES FOR FLEETITE, MEASURED IN AIR

λ (nm)	R (%)	λ (nm)	R (%)	λ (nm)	R (%)
440	45.6	540	46.3	620	46.7
460	45.8	546 (COM)	46.4	640	46.9
470 (COM)	45.9	560	46.5	650 (COM)	46.9
480	46.0	580	46.7	660	46.9
500	46.2	589 (COM)	46.7	680	46.9
520	46.2	600	46.7	700	46.9

Chemical Composition

We present results of three sets of electron-microprobe analyses of fleetite carried out using different facilities, analytical conditions, and standards (Table 2). The results are consistent. Seven ($n = 7$) analyses of fleetite were carried out by means of wavelength-dispersive spectrometry (WDS) using a JEOL JXA–8100 electron microprobe operated at 20 kV and 50 nA, with a beam diameter of 2 μm, at the Institute of Geology and Mineralogy, Siberian Branch, Russian Academy of Sciences, Novosibirsk. Pure iridium (for Ir), rhodium (Rh), ruthenium (Ru), chalcopyrite (Cu and Fe), synthetic FeNiCo (Ni), and synthetic Sb_2S_3 were used as standards. The following X-ray lines were used: Cu$K\alpha$, Ni$K\alpha$, Fe$K\alpha$, Ir$L\alpha$, Rh$L\alpha$, Ru$L\alpha$, Os$L\beta$, Pt$L\alpha$, Sb$L\alpha$, Bi$M\alpha$, Te$L\alpha$, and As$L\beta$. All peak overlaps, including Ru$L\beta_1$–Rh$L\alpha$, were checked and corrected. Osmium, Pt, Bi, As, and Te were sought but not detected. The mean results are listed as entry 1 in Table 2.

Results presented as entry 2 in Table 2 were obtained at the same Institute by quantitative energy-dispersive spectrometry (EDS) using a MIRA 3 LMU (Tescan Orsay Holding) scanning electron microscope (SEM) with an attached INCA Energy 450 XMax 80 (Oxford Instruments Nanoanalysis, UK) system operated at 20 kV and 1.6 nA. Pure elements were used as standards. The $K\alpha$ line was used for Cu, Ni, and Fe and the $L\alpha$ line was used for Ir, Rh, Ru, and Sb. The mean results provided as entry 3 in Table 2 ($n = 4$) were also obtained by EDS using a Tescan Vega 3 SBH SEM facility with an attached Oxford X-Act EDS system at the Siberian Federal University in Krasnoyarsk. As in the case of entry 2, pure elements were used as standards, the $K\alpha$ line was used for Cu, Ni, and Fe, and the $L\alpha$ line was used for Ir, Rh, Ru, and Sb.

The empirical formulae, calculated on the basis of a total of six atoms per formula unit (*apfu*) and taking into account the structural results (see below) are: $(Cu_{1.41}Ni_{0.58}Fe_{0.01})_{\Sigma 2.00}(Rh_{0.49}Ni_{0.36}Ru_{0.12})_{\Sigma 0.97}Ir_{0.95}Sb_{2.08}$ (#1, Table 2); $(Cu_{1.38}Ni_{0.62})_{\Sigma 2.00}(Rh_{0.50}Ni_{0.33}Ru_{0.13})_{\Sigma 0.96}Ir_{0.97}Sb_{2.08}$ (#2, Table 2), and $(Cu_{1.33}Ni_{0.67})_{\Sigma 2.00}(Rh_{0.52}Ni_{0.31}Ru_{0.14})_{\Sigma 0.97}Ir_{0.95}Sb_{2.09}$ (#3, Table 2). The simplified formula is $(Cu,Ni)_2(Rh,Ni,Ru)IrSb_2$. The ideal formula is $Cu_2RhIrSb_2$, which

TABLE 2. CHEMICAL COMPOSITION OF FLEETITE

	#1				
Constituent	Mean	Range	σ	#2	#3
Cu (wt.%)	13.93	13.64–14.36	0.2	13.41	13.16
Ni	8.60	8.43–8.71	0.1	8.54	8.93
Fe	0.10	0.07–0.13	0.02	b.d.l.	b.d.l.
Ir	28.07	27.16–28.58	0.5	28.51	28.32
Rh	7.91	7.45–8.48	0.3	7.92	8.26
Ru	1.96	1.86–2.07	<0.1	2.01	2.24
Sb	39.28	39.06–39.78	0.3	38.83	39.69
Total	99.85	99.22–100.46		99.22	100.60

Analysis #1 (with ranges for $n = 7$) was done by wavelength-dispersive spectrometry using a JEOL JXA-8100 microprobe. Analysis #2 are results of quantitative energy-dispersive spectrometry (EDS), with spectra analyzed using a MIRA 3 LMU (Tescan Orsay Holding) scanning electron microscope (SEM) with an attached INCA Energy 450 XMax 80 (Oxford Instruments Nanoanalysis, U.K.) system. Analytical results listed under #3 ($n = 4$) were obtained quantitatively by EDS using a Tescan Vega 3 SBH SEM with an attached EDS Oxford X-Act system. b.d.l.: below detection limits; σ: standard deviation.

TABLE 3. X-RAY DIFFRACTION DATA FOR FLEETITE

hkl	d_{meas}	I_{meas}	d_{calc}	I_{calc}	*hkl*	d_{meas}	I_{meas}	d_{calc}	I_{calc}
111	6.7	75	6.7366	87	551	-	-	1.6339	7
220	4.13	100	4.1253	100	642	1.556	20	1.5592	24
311	3.52	30	3.5181	28	731	-	-	1.5191	6
222	-	-	3.3683	5	800	-	-	1.4585	7
331	2.679	20	2.6769	23	660	-	-	1.3751	4
422	2.380	50	2.3818	47	822	-	-	1.3751	7
511	-	-	2.2455	6	753	-	-	1.2808	4
333	-	-	2.2455	3	664	-	-	1.2438	5
440	2.064	40	2.0627	46	844	-	-	1.1909	12
531	1.969	15	1.9723	23	$\underline{10}20$	-	-	1.1442	3
620	1.843	15	1.8449	21	862	-	-	1.1442	7
533	-	-	1.7794	4	$\underline{10}42$	-	-	1.0652	5
711	-	-	1.6339	5	$\underline{12}40$	-	-	0.9225	3

The *d* values are expressed in Å.

requires Cu 19.09, Rh 15.46, Ir 28.87, and Sb 36.58, for a total of 100 wt.%.

CRYSTALLOGRAPHY

Efforts to characterize fleetite crystallographically began with a synchrotron X-ray scan of the grain with a 1-μm step interval and with Laue microdiffraction measurements at beam line 12.3.2 of the Advanced Light Source (ALS), Lawrence Berkeley National Laboratory, Berkeley, California. The Laue diffraction patterns were collected using a PILATUS 1M area detector in reflection geometry. The patterns consistent with a cubic symmetry were indexed and analyzed using the XMAS v.6 software (Tamura 2014). A monochromator energy scan was performed to determine the lattice parameter *a* of fleetite, which was found to be 11.66(2) Å.

The X-ray powder-diffraction data (Table 3) were collected with an Oxford Diffraction Xcalibur PX

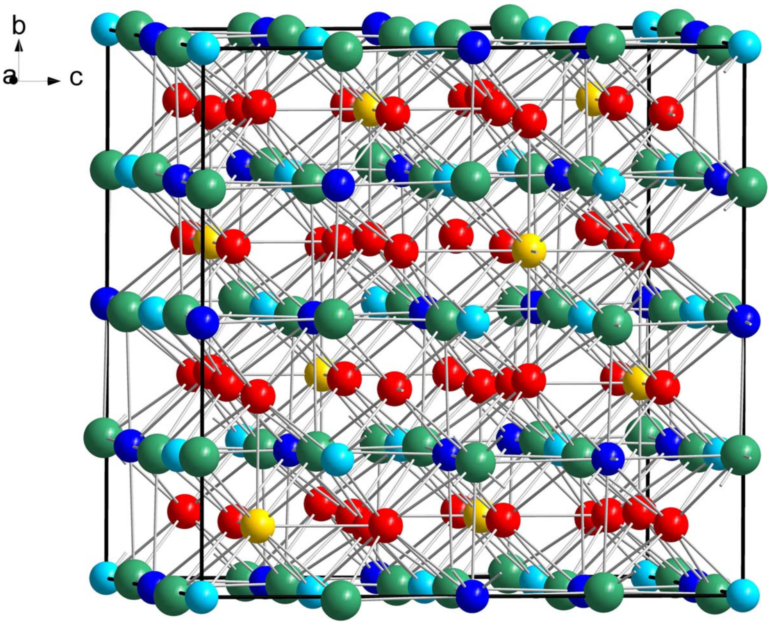

FIG. 4. The crystal structure of fleetite. Orange, red, green, light, and dark blue circles correspond to Cu1, Cu2, Sb, Ir, and Rh, respectively. The unit cell and the orientation of the structure are outlined.

TABLE 4. DATA AND EXPERIMENTAL DETAILS FOR THE FLEETITE CRYSTAL INVESTIGATED

Crystal data	
Formula	$Cu_2RhIrSb_2$
Crystal size (mm)	0.018 × 0.030 × 0.035
Form	Block
Color	Black
Crystal system	Cubic
Space group	$Fd\bar{3}m$ (#227, origin #2)
a (Å)	11.6682(8)
V (Å^3)	1588.59(19)
Z	16
Data collection	
Instrument	Oxford Diffraction Xcalibur III
Radiation type	Mo$K\alpha$ (λ = 0.71073 Å)
Temperature (K)	293(2)
Detector-to-sample distance (cm)	6
Number of frames	662
Measuring time (s)	60
Maximum covered 2θ(°)	62.60
Absorption correction	multi-scan
Collected reflections	17985
Unique reflections	153
Reflections with $F_o > 4\sigma(F_o)$	44
R_{int}	0.0395
R_σ	0.0452
Range of h, k, l	$-16 \le h \le 16$, $-16 \le k \le 16$, $-16 \le l \le 16$
Refinement	
Refinement	Full-matrix least squares on F^2
Final R_1 [$F_o > 4\sigma(F_o)$]	0.0212
Final R_1 (all data)	0.0340
Final wR^2 [$F_o > 4\sigma(F_o)$]	0.0467
Final wR^2 (all data)	0.0528
Number refined parameters	10
GoF	0.958
$\Delta\rho_{max}$ (e Å^{-3})	0.67
$\Delta\rho_{min}$ (e Å^{-3})	–0.88

Ultra diffractometer fitted with a 165 mm diagonal Onyx CCD detector and using copper radiation (Cu$K\alpha$, λ = 1.54138 Å). The program Crysalis RED was used to convert the diffraction rings to a conventional powder-diffraction pattern. The least-squares refinement gave the following values: a = 11.6575(5) Å, V = 1584.22(19) Å^3.

Single-crystal X-ray studies were then carried out using an Oxford Diffraction Xcalibur diffractometer equipped with an Oxford Diffraction CCD detector, with graphite-monochromatized Mo$K\alpha$ radiation (λ = 0.71073 Å). The single-crystal diffraction-intensity data were integrated and corrected for standard Lorentz polarization factors with the CrysAlis RED package (Oxford Diffraction 2006). The program abspack in CrysAlis RED was used for the absorption correction. A total of 153 unique reflections were collected. Systematic absences were found to be consistent with the space group $Fd\bar{3}m$. These results show that fleetite crystallizes in the cubic system, in space group $Fd\bar{3}m$ (#227; origin #2). The unit-cell parameter a is 11.6682(8) Å [V = 1588.59(19) Å^3, and Z = 16], confirming the earlier determinations.

The structure was solved and refined using the program SHELX-97 (Sheldrick 2008) to R = 0.0340 based upon 153 reflections with $F_o > 4\sigma(F_o)$. In detail, the site-occupancy factor (s.o.f.) at the cation sites was allowed to vary (Cu, Rh, Ir, Sb *versus* structural vacancy) using scattering curves for neutral atoms taken from the *International Tables for Crystallography* (Wilson 1992). Although it is very hard to give a cation distribution in the presence of elements with similar scattering powers, our best interpretation of the data is that the Cu2 position is half-occupied by copper, whereas the Cu1, Ir, and Sb sites are fully occupied by Cu (Ni, with almost the same scattering power), Ir, and Sb, respectively. Such a distribution was then fixed in the subsequent cycles of the refinement. Indeed, the Rh site exhibits a mean electron number of 38.3, which is in good agreement with $Rh_{0.49}Ni_{0.36}Ru_{0.12}$ (mean electron number 37.4), the site population obtained from the electron-microprobe data. Experimental details and R indices

TABLE 5. ATOMS, SITE-OCCUPANCY FACTORS (s.o.f.), COORDINATES OF ATOMS, AND THEIR ISOTROPIC TEMPERATURE-FACTORS IN THE STRUCTURE OF FLEETITE

Atom	s.o.f.	x	y	z	U_{iso}
Sb	$Sb_{1.00}$	0.25625(9)	0.25625(9)	0.25625(9)	0.0103(6)
Ir	$Ir_{1.00}$	0	0	0	0.0193(14)
Rh	$Rh_{0.85(2)}\square_{0.15}$	½	½	½	0.022(3)
Cu1	$Cu_{1.00}$	⅛	⅛	⅛	0.0159(13)
Cu2	$Cu_{0.52(2)}\square_{0.48}$	0.3619(5)	⅛	⅛	0.015(2)

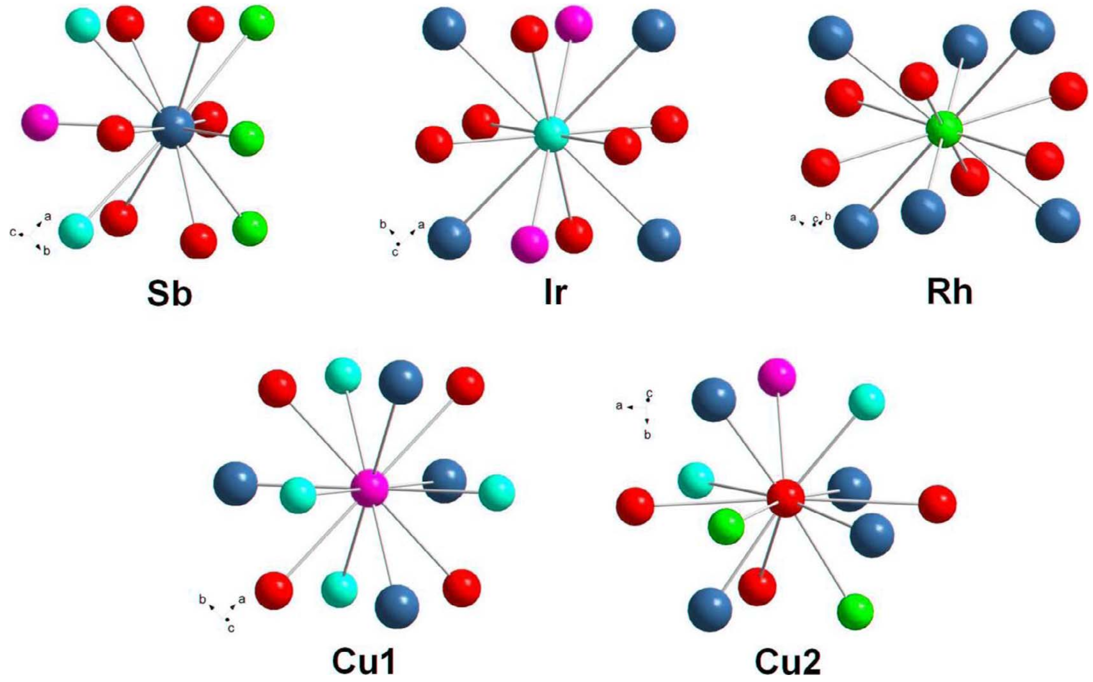

FIG. 5. Coordination environment of atoms in the crystal structure of fleetite. Colors as in Figure 4.

are given in Table 4. Final coordinates and equivalent isotropic displacement parameters of the atoms are given in Table 5, and selected bond-distances are shown in Table 6.

The crystal structure of fleetite is shown in Figure 4. It is best described as intermetallic, with all the metal atoms in 12-fold coordination (Table 6, Fig. 5). Fleetite is isotypic with $Pd_{11}Bi_2Se_2$ (Götze *et al*. 2018) and displays, to a certain extent, some structural similarities with synthetic CoMnSb (Ksenofontov *et al*. 2006).

TABLE 6. BOND DISTANCES (Å) IN THE STRUCTURE OF FLEETITE

Sb–Cu2 (×3)	2.492(3)	Cu1–Ir (×4)	2.5262(2)
Sb–Cu2 (×3)	2.584(4)	Cu1–Sb (×4)	2.6526(18)
Sb–Cu1	2.6526(18)	Cu1–Cu2 (×4)	2.764(6)
Sb–Rh (×3)	2.8460(10)		
Sb–Ir (×3)	2.9918(11)	Cu2–Ir (×2)	2.441(3)
		Cu2–Sb (×2)	2.492(3)
Ir–Cu2 (×6)	2.441(3)	Cu2–Sb (×2)	2.584(4)
Ir–Cu1 (×2)	2.5262(2)	Cu2–Rh (×2)	2.617(3)
Ir–Sb (×6)	2.9918(11)		
		Cu2–Cu1 (×6)	2.764(6)
Rh–Cu2 (×6)	2.617(3)	Cu2–Cu2 (×4)	2.9250(6)
Rh–Sb (×6)	2.8460(10)		

ORIGIN

The Miass placer deposits of PGM are inferred to have been derived from ophiolite bodies, mostly from the Karabash and Talovka (Talovskiy) complexes. At its lode source, fleetite formed after the crystallization of chromitite. As in the case of the majority of the platinum-group minerals in the Miass placer, such as Ru–Os–Ir disulfide, Ir–Rh sulfarsenide, Pt sulfide, Ir–Rh–(Cu) sulfoarsenoantimonides, and Ru diarsenide, fleetite likely formed at a late subsolidus stage of fluid-mediated transformations as a result of the buildup of S, As, and Sb in the fluid phase. The replacement reactions locally affected the primary Ru–Os–Ir and Pt–Fe alloys, which constitute the host minerals of these intergrown exotic phases (Barkov *et al*. 2018).

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