GRECHISHCHEVITE - $Hg_3S_2(Br, Cl, I)_2$ - A NEW SUPERGENE MERCURY SULFOHALIDE V. I. Vasil'ey, L. V. Usova, and N. A. Pal'chik Geo1ogiya i Geofizika, Vol. 30, No. 7, pp. 61-69, 1989 UDC 549.291

A new supergene mercury sulfohalide with the formula $\text{Hg}_{3}S_{2}(\text{Br}, \text{Cl}, \text{I})_{2}$, called grechishchevite, has been discovered in mercury ores from the Arzak and Kadyrel ore occurrence (Tuva ASSR). It is found in leaching caverns in calcite veins together with calomel, Br-calomel, kuzminite, cordierite, Br-cordierite, kadyrelite, lavrentievite, eglestonite, Br-eglestonite, and native mercury (Kadyrel). The mineral's short-prismatic crystals are colored bright-orange or dark-orange. The mineral is unstable when illuminated, heated, or under an electron beam; it has tetragonal syngony and variable composition, especially with respect to Br and Cl. An artificial analog of grechishchevite has been derived by pyrosynthesis, with the unit cell parameters $a = 13.255$ A (5), c = = 8.685 A (5), and $Z = 8$; its experimental density is 7.16 g/cm³.

There are few natural mercury sulfohalides. These are cordierite $(\alpha-\text{Hg}_{3}S_{2}Cl_{2})$, Brcordierite $\text{Hg}_3\text{S}_2(\text{Cl}, \text{Br})_2$, lavrentievite $\text{Hg}_3\text{S}_2(\text{Cl}, \text{Br})_2$, and arzakite $\text{Hg}_3\text{S}_2(\text{Br}, \text{Cl})_2$ [1-3,5J. In comparison with cubic cordierite and its bromine variety, the last two minerals have monoclinic syngony and are representatives of the isomorphic series $\text{Hg}_{3}\text{S}_{2}\text{Cl}_{2}$ - $Hg_3S_2Br_2$, for each of the end members of which several synthetic polymorphic analogs are known [6]. Apparently, synthetic mercury sulfohalides with mixed halogens have not been produced, except for tetragonal $Hg_3S_2Br_0$, $I_{1.5}$ [6]. A supergene mercury mineral*, in which almost all halogens are present along with sulfur, was discovered by V. I. Vasil'ev at the Arzak and Kadyrel mercury ore occurrences (Tuva ASSR). In its properties and Xray characteristics, it differs sharply from the sulfohalides listed above and, for the first time for such natural compounds, besides Br and Cl, ^I is established in an amount from 3.70 to 7.01%. We named the mineral grechishchevite in honor of the engineering geologist Oleg Konstantinovich Grechishchev, who studied mercury deposits in Tuva for ^a long time and gave ^a great deal of help to the author of the mineral find in field work in this region.

At the Arzak deposit [4J, grechishchevite is found sporadically in oxidized cinnabar ores in rhyolite-dacite porphyries subjected to intensive hydrothermal alteration (quartzification). The mineral is localized on the walls of thin exogenous little fissures in the form of films, accumulators of isometric or slightly elongated grains up to 0.2 mm in size, and little crystals of prismatic habit located near relicts of hypogene cinnabar, In such fissures it is accompanied by bromine calomel, eglestonite, lavrentievite, arzakite, cordierite, and native mercury. Very close association with lavrentievite, which often completely "cans" grains or crystals of grechishchevite, is typical.

At the Kadyrel ore occurrence, which is represented by a series of calcite veins with cinnabar and saucovite (Hg, Cd, Zn) S-mineralization cleaving Devonian sandstones, grechishchevite is found much less often and is confined to voids left from cinnabar, saucovite, and pyrite leached in the zone of oxidation. Here, it forms powdery masses, as well as concretions of short-prismatic little crystals up to 0.3 mm in size (Figs. 1

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Fig. 1 $F1g.$ 2

Fig. 1. Concretion of short-prismatic crystals of grechishchevite on calcite. Kadyrel ore occurrence. Scanning microsocpe. 100x.

Fig. 2. Group of disintegrating crystals of grechishchevite in a leaching cavity in calcite. Kadyrel ore occurrence. Scanning microscopic. *260x.*

and 2). Electron-microscopic study of the powders shows that they consist of elongated individuals, in places fitting tightly against each other. The group of supergene compounds accompanying grechishchevite is approximately the same as the community of its companion minerals at the Arzak deposit and includes calomel, Br-calomel, kuzminite $Hg_2(Br, 01)_2$, Br-cordierite, eglestonite, Br-eglestonite, kadyrelite $Hg_{\mu}(Br, 01)_2$ 0, lavrentievite, native mercury, Fe hydroxides, etc. At this ore occurrence also, grechishchevite is in direct contact with lavrentievite and overgrown by it.

Single crystals of grechishchevite have not been found; concretions of two or more short-prismatic individuals are most often observed, from the habit of which it is hard to determine their symmetry. The planes of such individuals' prisms are covered by numerous accessory growth faces, and distinct cleavage is displayed parallel to them (see Fig. 1).

The color of grains and concretions of grechishchevite crystals is bright- or darkorange with ^a reddish tint; therefore, the mineral is outwardly very similar to montroydite, chursinite, Br-eglestonite, kadyrelite, galchaite, getchellite, realgar, crocoite, and wulfenite. In light, the color slowly darkens and after prolonged exposure becomes brown-orange, and then black. A powder of pulverized grechishchevite grains is deepyellow or yellow with ^a slight orange tint. Accumulations of powdery masses of the mineral in leaching cavities are light-orange. Finely dispersed formations of grechishchevite diffused in milky-white kaolinite have a lemon-yellow color.

The luster of crystalline varieties and grain chips of grechishchevite is strong and glassy to adamantine. The mineral is brittle, with hardness \approx 2.5; its microindentation hardness (MH-3, P 25 g, n = 10) varies from 88 to 116 kgf/mm², with an average value $H_{\text{av}} = 106 \text{ kgf/mm}^2$. Its density was not determined due to the limited number and small size of the grains.

Little pieces of grechishchevite submerged in acids (HC1, HNO₃) for a long time do not change; in KOH (40%) their surface slowly blackens. The black film formed dissolves in a mixture of HCl and $HNO₃$, and afterwards the fragments acquire their own original color. With repeated action of KOH on them, the same affect is observed. In polished sections, the mineral does not react with HC1, HNO₃, or KOH. "Uncanned" grains of grechishchevite, kept in ordinary conditions in a shaded place, in time begin to decompose, are covered with cavities, and get cloudy. This is prevented to some degree by spraying the specimens with ^a thin carbon film. In transmitted light, thin chips of grechishchevite are transparent, display pleochroism from straw-yellow (n_e) to yellow (n_0) , and are anisotropic. Extinction is parallel in relation to elongation and coinciding cleavage. The mineral is uniaxial and positive. Its refraction indices are above
two.

Grechishchevite polishes well. In reflected light it is gray-white when observed

Fig. 3. Reflection spectrum of grechishchevite from the Arzak deposit.

Fig. 4. Artificial crystals of grechishchevite (phase 2). Scanning microscope. 40x.

with dry lenses. Its reflection is higher than for lavrentievite on the whole, but lower than for cinnabar. Its double reflection is distinctive for the grains' different boundaries: for R_0 it is gray, for $R_{\rm e}^t$, gray-white. Reflection values (for R_0^t and $R_{\rm e}^t$,

respectively), measured on ^a POOS-l instrument (STF-3 standard - telluric glass), are: 19.2-22.8% (436 nm), 22.2-24.5 (460), 18.9-21.4 (500), 17.3-19.8 (546),16.5-18.8 (590), 16.7-18.9 (620), and 15.9-18.1% (656 nm). The reflection spectrum is shown in Fig. 3. Greshishchevite is noticeably anisotropic without color effects, but strong light-orange and orange internal reflexes are seen everywhere.

X-Ray photographs of crystalline formations of grechishchevite from the Arzak deposit and the Kadyrel ore occurrence are practically analogous (Table 1) and do not contain additional lines due to the presence of a mechanical admixture of extraneous minerals. However, on many films from powdery masses of it (Kadyrel ore occurrence) reflexes appear which are characteristic of cordierite, lavrentievite, or kuzminite. Only for the purest powders are the X-ray photographs the same as for crystalline varieties.

Due to the absence of satisfactory natural material for single-crystal photographs, the syngony and unit cell parameters were determined on an artificial analog of grechishchevite prepared by our request at the pyrosynthesis laboratory of the Institute of Geology and Geophysics of the Siberian Branch AS USSR. The compound was synthesized in an evacuated quartz vial by heating a mixture of original artificial powders of HgS, HgCl₂, $HgBr_2$, and HgI_2 taken in the proportions, necessary to obtain a homogeneous substance with the formula $\text{Hg}_3.0^\text{S}_2.0^\text{(Br}_1.0^\text{Cl}_0.5^\text{I}_0.5)^\Sigma$, which seemed most likely according to preliminary data.

The final product of the experiment consisted of two phases of the same elemental composition. The first phase, which had ^a greenish-yellow color with an orange tint, contained increased amounts of mercury *(z76%),* sulfur (>9), bromine (~10), and chlorine (>2.5), and a reduced amount of iodine (<3%), and gave a powder pattern different from that of grechishchevite. Less of this phase was formed than of the second, and it was not studied in detail.

The second phase, which had a bright-orange color, included concretions of complexly-shaped crystals and numerous autonomous little crystals of prismatic habit up to 0.5 mm in length, with basic faces of tetragonal prisms {100}, {110} and crowning faces of a tetragonal pyramid, sometimes combined with a pinacoid and faces of a tetragonal bipyramid (?) (Fig. 4). Their powder patterns are identical in appearance to the X-ray photograph of grechishchevite, but are distinguished by small deviations in values of d (see Table 1). Photographing such crystals on ^a "Sinteks" automatic diffractor confirmed that they have tetragonal syngony, with possible space groups $P42m$, $P\overline{4}m2$ or $P4mm$, and P4/mmm; unit cell parameters are a = 13.225 ± (5) A, c = $.8.685$ ± (5) A, V = 1519.01 A: With these cell parameters, the calculated density of the compound with the supposed theoretical formula $\text{Hg}_3.0^\text{S}2.0^\text{(Br}1.0^\text{Cl}0.5^\text{I}0.5)^\Sigma$ 2.0 should be 7.23 g/cm³ with Z = B. Experimental determination of it for crystals which were grown (by the volumetric method in ^a capillary tube at $+18^{\circ}$ C) gives a value of 7.16 g/cm³.

The composition of grechishchevite and its synthetic analog was studied (Table 2) by the micro-X-ray spectral method on a "Camebax" automated microprobe according to an im-

Table 1

Results of X-Ray Investigations of Grechishchevite

$\mathbf 1$		\overline{a}		J			4		5
I	d.X	I	$d\overset{\circ}{A}$	1	$d\overset{\circ}{\mathbf{A}}$	hni	I	ďÀ	ه ده
3 6 2	4,19 3,95 3,66	2 5—6 >l	4,19 3,95 3.63	3 6 2	4,195 3,955 3.677	130 112 230	3 Ü >۱	4.18 3.95 3.67	4.182 3,939 3,668
3 6	3.12 3.02	2 6	3,13 3,01	$\ddot{2}$ 6	3,125 3,022	330 132	>2 $6 - 7$	3,12 3,02	3.117 3,012
10	2,65	10	2,65	10	2.645	023 340 050	10	2,65	2.652 2,645 2,645
4	2,60	>3	2,59	4	2,590	123 150	4	2.60	2,600 2,593
>1	2,460	1	2,459	0,5	2,457	223 250	<1	2,459	2,462 2,456
4	2.341	<4	2,342	3	2,342	440	3	2,341	2,338
$_{0,5}$	2,271	0.25	2,271	${<}0.25$	2,268	233 350	0,5	2,273	2.272 2,263
3 wd*	2,180	3 wd*	2,179	3 wd*	2,180	043	>3	2,179	2.178
						014	< 0.25	2,143	2.142
< 0.25	2,103	÷	\div	0,25	2,103	161 260 243	÷	÷	2.109 2,091 2.069
< 0.5	2.066	0.25	2,067	0,25	2,065	450 U24	$_{0,5}$	2,065	2.065 2,063
ا>	2,011	0,5	2,010	1	2,018	352 451	1	2,010	2.010 2,009
				$0.25d*$	1,931	153	< 0.5 w*	1,933	1,032
3	1,873	3	1,873	3	1,874	253 550	3	1,872	1.873 1,870
0,25	1,820	0,25	1,819	${<}0.25$	1,821	443 270	< 0.5	1,820	1,819 1,817
0,25	1,784	0,25	1,784	$<$ 0.5	1,784	353 334	<1w*	1,784	1,785 1,782
-4	1.682	4	1,679	4	1,683	453 025 344	4	1,682	1,681 1.680 1,678
>1	1,641	1	1,639	0,5	1,646	470 180	>1	1,641	1,640 1.640
0.5 wd*	1,599	0.5 wd*	1,597	0,5	1,597	230 444	$>\!\!1$	1,593	1,604 1,591
1 wd*		$1,544$ 0.5 wd*	1,546	1 wd*	1,544	380 064 032	1 wa*	1,544	1,548 1,547 1,545
1 wd*	1.462	0.5 wd*	1,465	0.5 wd*	1,463	662 563	1 wd*	1,462	1,467 1,462
						191 016	0.25	1,438	1.440 1,439
There are 10 more wide, diffuse lines with low intensity after 1.462							35.∪>	1,422	1.425 1,418
						283 580 464	≤ 0.5	1,401	1,403 1,402 1,401
						136 383 ເໝົວ	0.5 ₁	1,367	1.368 1,365 1.364

Note. Columns: 1) grechishchevite from the Arzak deposit (chemical analyuses 4-8, Table 2), calculated
unit cell parameters: $a = 13.208(6)$ Å, $c = 8.698(9)$ Å; grechishchevite from the Kaderyl ore occurrence: 2)
crystais,

proved program. We investigated 11 crystals of the mineral from ores of the Arzak deposit, two from the Kadyrel ore occurrence (up to 0.3 mm in size), and four synthesized ones. With a voltage of 20 kV and a current of absorbed electrons of about 6 nA, Hg, Cl, Shes. Which a voltage of 20 K. and a carrent of absorbed electrons of disage 3 km, has, HgS (HgM_a- and SK_a-lines) $Pb_7S_2Br_{10}$ (BrL_a-line), HgI₂(IL_a-line), HgSe (SeL_a-line), and natural calomel $\text{Hg}_{2} \text{Cl}_{2}$ (Cl K_{α} -line) with a Br content of no more than 0.01% by weight. Grechishchevite and its synthetic analog were unstable under electron bombardment; therefore, the analysis was done with an electron probe expanded to 5 um, with continuous movement of the specimen.

Crystals of the second phase which were grown differ little in their composition from the supposed final product of synthesis and practically coincide with grechishchevite from both mercury deposits (see Table 2).

The analysis results and theoretical ideas about the permissible bonds and mutual replacements of elements allow us to pick out two pairs of halogen combinations in grechishchevite in the group of them: Br-Cl and Br-I, isomorphism between the elements of which in the mineral is most likely on the strength of the likeness of their ions' radii. In comparison with the total atomic number of the elements in each pair, there is always less of the third halogen of the group, ^I or Cl. There is usually less chlorine than bromine. In only three grains were the Br: Cl ratios almost the same (see Table 2, analyses 1, 3 and 4), and in one other (analysis 2) chlorine was even somewhat predominant over bromine. The atomic content of chlorine is more often higher than that of iodine, but there are grains with opposite ratios: I > Cl (analyses 7-10). Also typical is a more or less consistent concentration of iodine within a narrow range, in contrast to the concentrations of bromine and chlorine, which apparently may indicate a strictly limited quantitative interval of the replacement, most likely of bromine, by iodine.

Thus, the inconsistency of the mineral's composition stands out, primarily with respect to the contents of halogens, which determines the distribution of these elements' places in formulas calculated according to analyses of the corresponding grains. The instability of composition is evidently due not only to ^a change in the degree of the compound's disintegration under the probe, but also to natural variation in the concentration of halogens on account of quantitatively varying replacement of one element by another during crystallization of independent, spatially separate formations of grechishchevi te.

Analytic data and the probable pair combinations of halogens in the mineral which were mentioned above permit us to write several types of formulas for it, which are supposedly satisfactory when figured for seven atoms in the unit cell, including $Hg_3S_3[Br,$ Cl], I)₂. However, due to the absence of structural determinations, it is not yet possible to choose a definitive variant taking into account actual replacements among halogens or their occupation of specific positions in the crystalline lattice. Therefore, the mineral's formula can be given only in ^a general form, i.e., with ^a combined group of halogens and their position according to the amount of each element. For the speci- mens analyzed, it is as follows (see Table 2).

Analysis no. Formula

 $\mathbf{1}$

- Hg_3 ,03 S_2 ,03 (Clo.77 Br_0 ,73 I_0 ,43) x_1 ,93
- 2 $Hg_2.98S_2.06 \left(\text{Cl}_{0.82} \text{Br}_{0.75} \text{I}_{0.339} \right)$ $\text{I}_{1.93}$
- 3 Hg_{3.01}S_{2.09} (Br_{0.75}Cl₀.75I_{0.39}) $z_{1,90}$
4 Hg_{3.07}S_{2.03} (Br_{0.77}Cl_{0.74}I_{0.39}) $z_{1,90}$
- $Hg_{3.07}S_{2.03}(Br_{0.77}Cl_{0.74}I_{0.39})$ $x_{1.90}$
-
- 5 Hg_{3.07}S_{2.04} (Br_{1.06}Cl_{0.44}I_{0.39}) x_{1.89}
6 Hg_{3.09}S_{2.05} (Br_{1.12}Cl_{0.39}I_{0.35}) x₁ as
- 6 Hg_{3.09}S_{2.05} (Br_{1.12}Cl_{0.39}J_{0.35}) x_{1.86}
7 Hg_{3.03}S_{2.09} (Br_{1.14}J_{0.41}Cl_{0.34}) x_{1.59}
- 7 Hg3.03 $S_2.09$ (Br₁,14Io.41Clo.34) x1.89
S Hg_{3.02}S_{2.09} (Br₁,13Io.43Clo.32) 1.84
- 8 Hg_{3.02}S_{2.09} (Br₁,13I₀,43Cl_{0.32}) 1.58
9 Hg_{2.05}S_{1,09} (Br_{1,15}I_{0,47}Cl_{0.32}) 71.01
- 9 Hg_{3.05}S_{1.99} (Br_{1.15}I₀,47Cl_{0.33}) $z_{1.96}$
10 Hg_{3.03}S_{2.09} (Br_{1.19}I_{0,42}Cl_{0.33}) $x_{1.95}$
- 10 Hg_{3.03}S_{7.09} $(Br_{1,19}I_{0,43}Cl_{0,25}) z_{1,37}$
11 Hg_{3.05} (S_{2.03}Se_{0.01}) Tr_{2.4} (Br_{1.19}Cl₀
- 11 Hg_{3.05} (S_{2.03}Se_{0.01}) $x_{2.04}$ (Br_{1.19}Cl_{0.39}I_{0.34}) $x_{1.92}$
12 Hg_{3.05}S_{2.03} (BreadClossIo.a) the
- 12 Hg_{3.06}S_{2.02}(Br_{0.93}Cl_{0.56}I_{a.43}) 1.92
13 Hg_{3.05}S_{1.09}(Br_{0.93}Cl_{0.79}I_{a.24}) _{21.9}
- 13 Hg_{3,05}S_{1,09}(Br_{0,93}Cl_{0,79}I_{0,24})_{21,95}
19 Hg_{3,03}S_{2,07}(Br_{1,07}Cl_{0,49}I₀,24)_{71,00}
- Hg_3 ,03 S_2 ,07 $(Br_1$,07 C lo.49 I_9 ,34) z_1 ,90

Formulas for the mineral calculated according to the best analyses demonstrate inequality of the ratios Br:Cl and Br:I. and inverse correlation between the Obvious deficiencey of halogens. This also seen in other formulas, including those of the synthetic

Table 2

Chemical Composition of Grechischevite and Its Synthetic Analog (% by Weight)

	Arzak deposit										
Element		2	3	4	5	6		8			
Hg	73.60	73.68	73,70	74.42	72.40	73.20	72,30	72.74	71.82		
Br	7.09	7.32	7,44	7,46	9.99	10.60	10,76	10,85	10,84		
Cl	3,31	3.57	3,24	3,18	1,84	1,62	1.43	1,40	1.40		
	6,55	6,13	5,91	5,98	5.87	5,20	6,25	6,56	7,01		
s	7.89	8.14	8,15	7,89	7,70	7.79	7.94	8,04	7,52		
Se	0,03	0.02	Not detected	0.02	0.02	0.03	0,01	0.02	Not detected		
otal	98,47	98,86	98,44	98,95	97.82	98,44	98,69	99.61	98,59		

 \vert 98,31 | 98,57 | 99,08 | 98,37 | 98,61 | 99,83 | 100,35 | 99,51 | 100,17 | 99,96 Total

Note, Average data are given for no fewer than two independent analyses of each specimen. The analysis numbers correspond to the numbers of individual specimens arranged in order of increasing contents of Br. Analyses 1 and 2 are possibly of a new mineral and were not used in figuring the average amounts of elements
(14); 19) average for analyses 15-18 of the synthetic analog of grechishchevite.

compound. The main reason for the deviation from stoichiometry, in our view, is an error in determining the amounts of elements, connected with the extreme instability of the object being studied under the probe and a possible variable rate of scanning over its small area, which leads to different degrees of destruction of the substance in individual segments of the total time of each scan and, consequently, to unequal release of metallic mercury, accompanied by more or less intensive volatilization of halogens, especially iodine.

Keeping in mind the probability of mistakes in recording the concentrations of elements in the specimens of grechishchevite investigated, it is better at present to write the mineral's calculated formula according to the average analytical data: Hg_{3.05}S_{2.04}- $(Br_{1.03}c1_{0.49}I_{0.39})\Sigma_{1.91}$ (see Table 2, analysis 14). The formula for the synthesized substance, also calculated according to average figures from the analyses (see Table 2, analysis 19) practically does not differ from it: the order of places among halogens in the group of them is maintained and, according $/cm³$. It agrees well with its measured density (7.16 $g/cm³$) and the calculated density of grechishchevite from the Arzak ore occurrence, which is equal to 7.23 $g/cm³$. In the idealized variant, the general formula for the mineral and its artificial analog will be $Hg_3S_2(Br, Cl, I)_3$, where $Br > Cl > I$.

If the additional patterns $CL > Br$ and $I > Cl$, noted thus far on a limited amount of material, are valid, then one must suppose the existence of isomorphic series of natural sulfohalide mercury compounds: $Hg_3S_2(Br, Cl, I)_2 - Hg_3S_2(Cl, Br, I)_2$, $Hg_3S_2(Br, I, Cl)_2$ - $\text{Hg}_{3}\text{S}_{2}(\text{Cl}, \text{Br}, \text{I})_{2}$, and $\text{Hg}_{3}\text{S}_{2}(\text{Br}, \text{I}, \text{Cl})_{2}$ - $\text{Hg}_{3}\text{S}_{2}(\text{I}, \text{Br}, \text{Cl})_{2}$ (tetragonal syntony).

In ores, grechishchevite is overgrown by lavrentievite, which contains little or no iodine. There is reason to believe that the crystallization of lavrentievite is preceded by formation of arzakite, sometimes with a small isomorphic admixture of iodine. In other words, in a natural situation conditions are created locally for formation first of sulfohalide mercury compounds with the maximum number of halogens in their composition and a quantitative predominance of one pair (Br-Cl or Br-I) among them, and then minerals of the same type with ^a reduced group of halogens, in which the most active of them (iodine) is often completely absent, while another (bromine) gradually yields to chlorine the leading place with respect to content in the group or pair of halogens.

Specimens of ore with grechishchevite have been turned over for exhibition and storage to the Central Siberian Geological Museum at the Institute of Geology and Geophysics of the Siberian Branch AS USSR, and the Mining Institute's museum in Leningrad.

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