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PARADOCRASITE, $Sb_2(Sb,As)_2$, A NEW MINERAL¹

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ABSTRACT

Paradocrasite (Gr. "unexpected alloy") forms an aggregate of silvery-white, striated, stubby prisms replacing calcite in a specimen of supposed "dyscrasite" from Broken Hill, New South Wales.

Paradocrasite is brittle and has perfect {010} parting and several less perfect partings. Its density is 6.52 (measured), 6.44 (calculated). Paradocrasite is monoclinic, space group $C2$, lattice parameters $a=7.252$, $b=4.172$, $c=4.431$ Å, $\beta=123^\circ08.4'$. Major lines of the X-ray powder pattern are 3.72 m, 3.05 vs. 2.219 s, 2.093 s, 1.720 ms, 1.518 Å m. Microprobe analysis gives, in weight %, Sb 82.9, As 18.6, sum 101.5, equivalent to $Sb_{2.98}As_{1.07}$ with $Z=1$.

In polished section paradocrasite is bright white in air and in oil. Reflection-pleochroism in air is distinct—yellowish white with a slightly pinkish tint, to slightly grayish white; in oil, strong—pale pink, white, gray with a very faint blue-green tint. Anisotropism in air is strong; the rather vivid color effects are somewhat intensified in oil. Internal reflection is lacking. Grains are subequant, polysynthetically twinned on {010}, {201}, {001}, and {101}(?). These composition planes are paralleled by partings. Reflectance and optical orientation at 546 nm are: $R_g \sim \perp bc = 73.8\%$, $R_m \approx c = 70.5\%$, $R_p \approx b = 67.8\%$. Mean R at 470, 546, 589, and 650 nm is 71.2, 70.0, 70.0, and $\sim 68\%$, respectively. Microindentation hardness at 100-gram load (HV_{100}) = 104–134, mean 118. Etch reactions are: HNO_3 , HCl , $FeCl_3$, $HgCl_2(+)$; KCN , $KOH(-)$.

Minute bodies of stibarsen (SbAs) are exsolved from paradocrasite, and small radiating clumps of antimonian loellingite replace paradocrasite. Reflectance, microindentation hardness, X-ray powder data, unit-cell dimensions, and microprobe analyses of these associated minerals are reported.

Paradocrasite somewhat resembles native antimony, stibarsen, and dyscrasite in qualitative properties, but its quantitative ore-microscopic properties, twin habit, and powder pattern are unique. The chemical composition of paradocrasite is appropriate for an "allemontite" in the natural mix-crystal series of hexagonal Sb-As alloys, but its monoclinic symmetry is not. The synthetic analogue of paradocrasite has never been reported in experimental studies of the Sb-As system.

INTRODUCTION

In attempting to verify a dyscrasite for comparison with some antimony minerals from Idaho, we discovered the new antimony-arsenic

¹ Publication authorized by the Director, U. S. Geological Survey.

alloy described in this paper. Dyscrasite, Ag_3Sb , was named discrease or "bad alloy" (Gr. $\delta\upsilon\sigma$ —bad $\kappa\rho\alpha\sigma\iota\varsigma$ —alloy) by Beudant (1832, p. 613; Dana's System, 1944, p. 173). Accordingly, we propose the name *paradocrasite* (Gr. $\pi\alpha\rho\alpha\delta\omicron\chi\sigma$ —contrary to expectation, $\kappa\rho\alpha\sigma\iota\varsigma$ —alloy) for the new antimony-arsenic mineral. The name and the mineral have been approved by the Commission on New Minerals and Mineral Names of the International Mineralogical Association.

OCURRENCE AND MEGASCOPIC PROPERTIES

Paradocrasite is the chief mineral in U. S. National Museum specimen USNM R-419, labeled "dyscrasite," from Broken Hill, New South Wales. In hand specimen, paradocrasite is mainly an aggregate of small, diversely oriented, brilliant silver-white, stubby prisms, each about 0.5 mm long, embedded in a matrix of white calcite. Some prisms are striated parallel to the long axis. A few prisms are curved. The ends of the prisms are rough, showing intersecting domains that represent complex, repeated twins. A few nests of curved, grooved pseudo-hexagonal plates are also present. Paradocrasite is brittle, crushing readily to fine dust that contains a few coarser, splintery fragments and a scattering of still coarser perfect cleavage flakes. The streak is black; the luster is metallic. The density of paradocrasite, determined by Berman balance on a 40-mg fragment free of calcite, is taken as 6.5. The measured value is 6.52, but because the fragment was relatively large and may have contained sparse inclusions of associated metallic minerals, the less precise value is probably more reliable.

Mr. Paul Desautels, of the National Museum, kindly verified the source of the specimen as the old Roebbling collection. Other National Museum material under the original Roebbling label R-419 shows a brown carbonate (siderite?) intergrown with the calcite gangue. The appearance of paradocrasite and the nature of its matrix are the same as those reported by Smith (1920) in his description of the original dyscrasite find at Broken Hill. However, Professor L. J. Lawrence of the University of New South Wales subsequently examined our specimen USNM R-419 and expressed doubt that it came from Broken Hill. He believes (oral commun., 1969) that the specimen came from the Lucknow gold mine, Lucknow, 6 miles east of Orange, New South Wales, where the paradocrasite would be called native antimony. The Lucknow mine is about 450 miles from Broken Hill. As Professor Lawrence has investigated the Broken Hill ores in detail (see especially Lawrence, 1968; Markham and Lawrence, 1962) and is now studying the mineralogy of the Lucknow mine, we hope that he may be able to resolve the question he raised regarding the source of specimen USNM R-419.

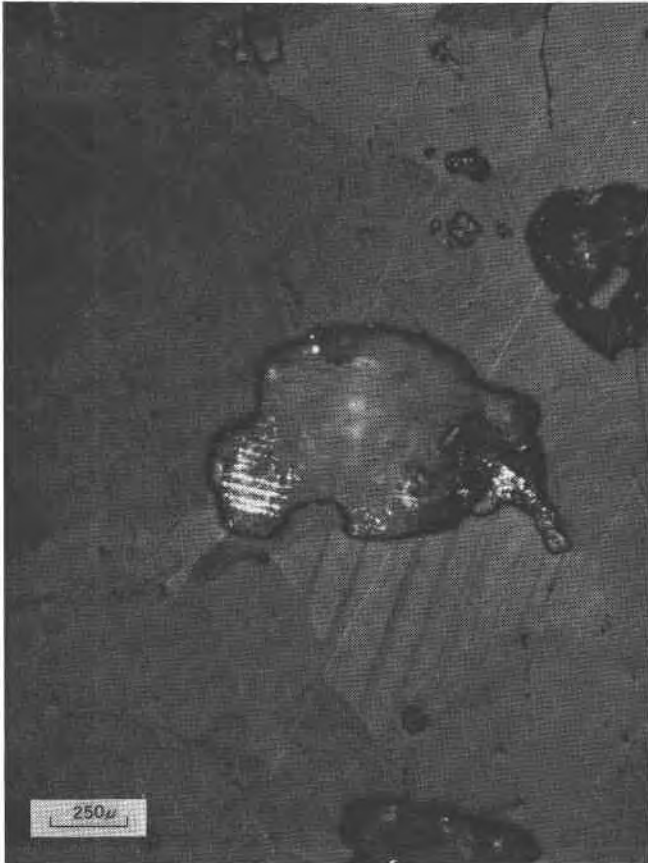


FIG. 1. Photomicrograph of paradocrasite showing polysynthetic twinning. Central area, showing internal reflection, is twinned calcite. Nicols partly crossed.

ORE MICROSCOPY

Paradocrasite is rather readily polished with diamond abrasives, but very fine scratches are ineradicable without severe overpolishing. Unlike dyscrasite, native arsenic, and allemontite, paradocrasite does not tarnish. A polished section kept for 5 years in the relatively dry climate of Denver showed no discoloration.

In polished section, paradocrasite is an aggregate of nearly equant, polygonal grains that replaces calcite irregularly. The grains are conspicuously and polysynthetically twinned (Figure 1). Parallel-sided lamellae, about 25 microns wide, are ubiquitous. In addition, some grains show a set of short, tapering lamellae that intersect the parallel-sided lamellae obliquely. The pattern of the two sets of twins closely resembles that given by combined albite and pericline twinning in plagioclase.

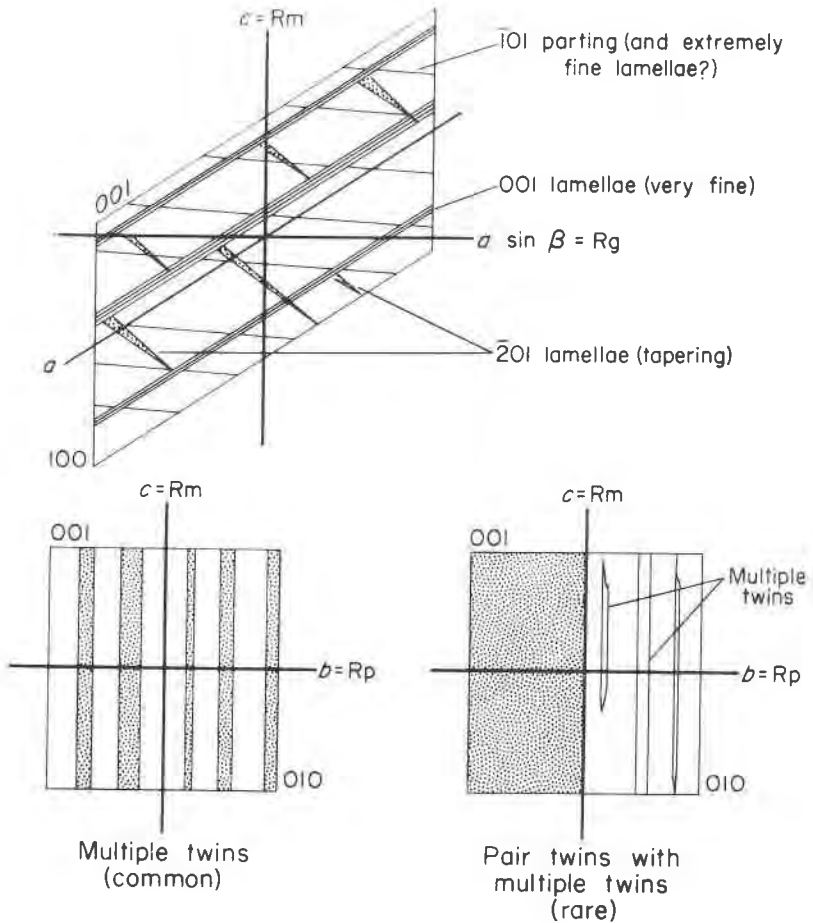


FIG. 2 Optical orientation and twinning in paradocrasite. Apical angle of tapering lamellae is greatly exaggerated in this schematic drawing. On lower half of diagram, trace of lamellae and partings having $[010]$ zone axis is omitted to emphasize dominance of $\{010\}$ twinning. The magnitude $a \sin \beta$ should be read as the direction $\perp bc$.

Very thin lamellae, each about one micron wide, are occasionally found as discrete groups; these lamellae constitute a third set. The orientation of these sets of twins, deduced from optical study and referred to the monoclinic cell, is shown in Figure 2. The composition plane of abundant straight-sided lamellae and rare pair twins is $\{010\}$; the composition plane of tapering lamellae is $\{201\}$; the composition plane of groups of very thin lamellae is $\{001\}$. These composition planes are paralleled by partings, and an additional parting, perhaps paralleled by extremely fine lamellae, is $\{101\}$.

The polishing hardness of paradocrasite is very slightly less than that of stibarsen and much less than of antimonian loellingite, the only additional metallic minerals in the polished section.

These additional metallic minerals, nearly equal in abundance, make up 2 percent of the metallic aggregate. Antimonian loellingite, the more conspicuous minor constituent, forms shreds and clumps of radiating, bluntly acicular crystals, 50–150 μm long, mostly in paradocrasite but partly in calcite. Stibarsen, the homogeneous AsSb component of allemontite, is confined to paradocrasite, in which it occurs as clusters of minute blebs accompanied by a few roundish elongate bodies whose length does not exceed 100 μm . The blebs of stibarsen occur mainly as clusters confined to individual grains of paradocrasite. Most of the blebs are 5–10 μm in diameter, but the smallest are less than 0.5 μm . A few relatively large, irregular bodies of stibarsen, the largest measuring 25 \times 100 μm , are also present within individual paradocrasite grains. Elongate, slightly tapered, occasionally irregular bodies of stibarsen, a few microns wide and 75–100 μm long, are restricted to the boundaries between contiguous grains of paradocrasite.

The appearance of antimonian loellingite and stibarsen is shown in Figures 3 and 4, and the properties of these minerals are reported in a



FIG. 3. Photomicrograph of radial aggregate of antimonian loellingite, medium and dark gray, in paradocrasite containing blebs of stibarsen. Nicols partly crossed.

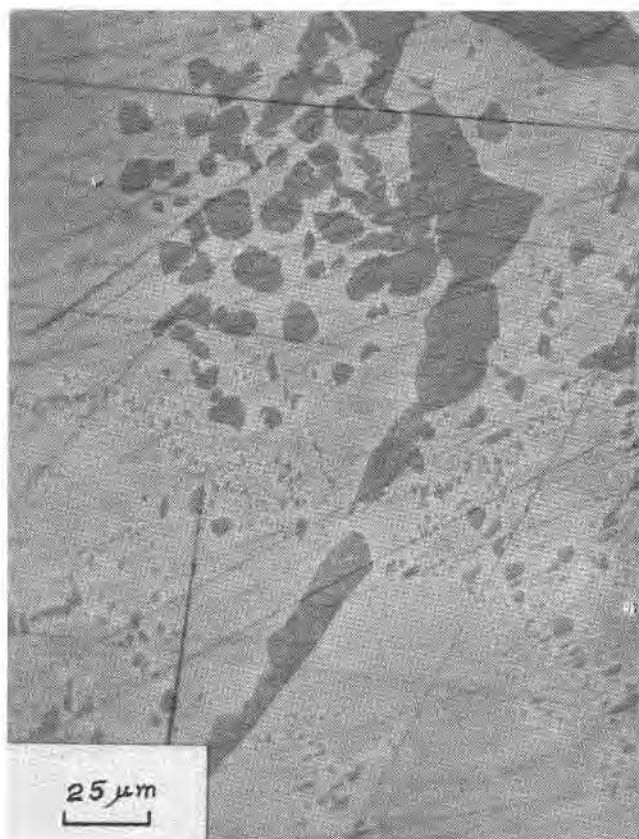


FIG. 4. Photomicrograph of exsolved blebs of stibarsen, the darker constituent, in paradocrasite. Contrast printing has exaggerated the distinction between host and guest and has produced bright halos about the smallest inclusions. Oil; green filter.

separate section. The emulsoid fabric of the stibarsen suggests that it has been exsolved from the paradocrasite host.

Qualitative optical properties. Paradocrasite is white and very bright in air and in oil. In air its reflection-pleochroism is distinct—yellowish white with a slightly pinkish tint, to slightly grayish white. Twinning gives the polished surface a moiré effect that makes it difficult to be sure of reflection-pleochroism until the position of the twin lamellae has been determined between crossed polars. In oil reflection-pleochroism is strong—pale pink, white, gray with a very faint blue-green tint; other grains look yellowish-white, white, gray. Anisotropism in air is strong, with rather vivid tints of pink, pale orange, yellow, pale greenish-blue, and

TABLE 1. REFLECTANCE OF PARADOCRASITE.

Wavelength, nm	R_{air} , percent			
	470	546	589	650
$Rg \sim \perp b c$	75.0	73.8	73.6	70.2
$Rm \approx c$	71.2	70.5	70.7	67.8
$Rp \approx b$	68.6	67.8	67.6	65.9
$Rg - Rp$	6.4	6.0	6.0	4.3
$(Rg + Rp)/2$	71.8	70.8	70.6	68.0
$(Rg + Rm + Rp)/3$	71.6	70.7	70.6	68.0
\bar{R} (statistical)	71.2	70.0	70.0	~ 68

Reflectance measured by B.F.L. with a Reichert photoelectric microphotometer fitted with a continuous interference filter whose half-width at half-height is 13–15 nm. U. S. germanium standard calibrated by National Physical Laboratory, Teddington, England; R at 470, 546, 589, and 650 nm taken as 47.0, 51.3, 52.0, and 47.1%, respectively. Mount press-leveled on plasticine. Reproducibility of measurements for most orientations of paradocrasite is $\pm 1\%$, relatively; for example, 75.0 ± 0.8 . For \bar{R} at 546 nm, the most reliable value reported in the table, the standard deviation of 18 observations is 0.46%. The orientation is for the wavelength 546 nm.

pale green, the last being the dominant color in the 45° position. The effects are somewhat intensified in oil. Internal reflection is lacking in air and in oil.

Reflectance. The reflectance of paradocrasite is given in annotated Table 1. The crystallographic orientation of Rg , Rm , and Rp is necessarily approximate; it can only be determined within a few degrees and therefore does not provide an independent check on the monoclinic or triclinic symmetry of paradocrasite. The value Rm , which in some investigations has to be determined statistically, can be found with some confidence for paradocrasite because grains showing null anisotropism are easily recognized and moderately abundant. The close agreement of the midrange of reflectance, the optical mean $(Rg + Rm + Rp)/3$, and the statistically determined mean indicates that the measurements are internally consistent but does not assure their accuracy. In particular, some reflectance standard brighter than germanium would have been desirable, but none has been approved by the International Commission on Ore Microscopy.² If the optical phenomenon of secondary glare has caused some slight increase in the measured reflectance values of paradocrasite, one supposes that this effect may be approximately compensated by very slight imperfection in polish.

² Germanium has since been withdrawn as a COM provisional standard.

Quantitative determination of color. The reflectance data of Table 1 can be used to specify the color of paradocrasite in quantitative terms by the method applied to ore minerals by Piller (1966) and illustrated elsewhere by some additional examples (Leonard and others, 1969). Piller specified color in terms of a brightness value, Y , and trichromatic coefficients, x , y , and z , of which only x and y need be stated. He supplemented this specification with the derived Helmholtz values of dominant wavelength, λ_f , and excitation purity, p_e . Color values for paradocrasite are given in Table 2. They indicate that paradocrasite is almost a pure white, departing only slightly from I.C.I. standard illuminant C, for which $x=0.3101$, $y=0.3163$. Translated into terms more familiar to observers of ore minerals, the trichromatic coefficients of paradocrasite are virtually the same as those of $R\|b$ of stibnite (whose color is described by

TABLE 2. QUANTITATIVE COLOR DESIGNATION OF PARADOCRASITE.

	Y	x	y	λ_f	p_e
R_g	73.6	0.307	0.314	—	—
R_m	70.4	0.308	0.315	—	—
R_p	67.7	0.308	0.315	—	—
$(R_g+R_m+R_p)/3$	70.0	0.307	0.314	480 nm	0.016

Ramdohr, 1960, as flat gray, or neutral), but the brightness value Y of paradocrasite is about twice as great as that of stibnite $\|b$.

The quantitative color data for paradocrasite are somewhat surprising, for though they confirm the qualitative observation of whiteness and brightness, they give no hint of the delicate reflection-pleochroism that is perceptible to the eye trained to detect slight color differences. We have no explanation for this discrepancy. Very tentatively we suggest that the effect might be analogous to the one observed in certain monoclinic pyroxenes (Hess, 1949) and in certain hornblendes (Buddington and Leonard, 1953), in which the absorption indicatrix does not coincide with the optical indicatrix. For paradocrasite, a highly absorbing mineral of low symmetry, one might postulate that the orientation or shape of the absorption surface changes markedly with slight change of wavelength. Obviously paradocrasite has color effects too slight to be suited for a test of the hypothesis. However, if the hypothesis is admissible, it might explain why one can detect reflection-pleochroism in aggregates of variously oriented grains but cannot easily specify the tints in a principal section, such as R_g - R_p , whose optical orientation has been determined by photometry. Failure to define the dispersion curve

precisely by measuring R at 20-nm intervals would not account for this discrepancy.

Microindentation hardness. The microindentation hardness (Vickers hardness) of paradocrasite was measured at 100-gram load by means of a Leitz Durimet hardness tester. For 15 indentations, $HV_{100} = 104\text{--}134$, mean 118. The range is based on determination of HV_{100} for each indentation; this is the common convention. The indentations measured were perfect or only slightly fractured. Indentation shape as well as Vickers hardness varies with orientation, from straight-sided to concave, concave-convex, and markedly cymoid. Some indentations show shear cleavage subparallel to the indentation edge. Only one indentation had to be rejected as defective.

To check the effect of a lower load, five grains were indented at 25-gram load. Five indentations at 100-gram load were then made rather close to the first set and, so far as could be determined, on the same grains. For the first set, $HV_{25} = 121\text{--}142$, mean 130. For the second, $HV_{100} = 104\text{--}108$, mean 106. These results suggest that mean HV_{25} for paradocrasite might be about 25 percent greater than mean HV_{100} , or very close to HV_{25} of stibarsen, whose microindentation hardness is reported below.

Etch tests. Etch reactions with the standard reagents of Short (1940) are: HNO_3 (+); instantly darkens the surface and forms a permanent iridescent stain that reveals twinning. HCl (+); fumes produce faint brown halo, soluble in water. FeCl_3 (+); quickly darkens the surface, forms a permanent iridescent stain that reveals twinning, and etches the surface slightly. HgCl_2 (+); forms a brown stain, mostly soluble in water. KCN and KOH (-). The reactions are similar to those reported by Short for antimony, arsenic, and dyscrasite.

CHEMICAL COMPOSITION

Microprobe analysis of paradocrasite gives, in weight percent, 82.9 Sb, 18.6 As, sum 101.5, equivalent to $\text{Sb}_{2.93}\text{As}_{1.07}$ with $Z=1$, or very close to Sb_3As . Other elements, if present, are below the limit of detection—nominally ~ 1 percent. A special search for Ag, Bi, Hg, Pb, Pd, S, and Te failed to detect these elements. The analysis represents the mean of individual determinations of the elements on 10 different, homogeneous areas. The range for Sb is 81.0 to 83.7 wt. percent; for As, 18.3 to 19.0; for the sum, 99.3 to 102.3. Traverses across the width of several polysynthetically twinned grains showed very slight variation within each grain. This confirms the optical evidence that the lamellae are twins in a

single phase of nearly constant composition, not separate phases resulting from exsolution or other intergrowth.

The standards used were GaSb for Sb, and GaAs for As. The analysis was carried out at 25kV. A PET (pentaerythrytol) crystal was used to determine $SbL\alpha$, and an LiF (lithium fluoride) crystal was used to determine $AsK\alpha$. The results were corrected for background, drift, and absorption.

Iron was subsequently found by microprobe in stibarsen, but it was not detected in paradocrasite.

Microchemical tests of paradocrasite by the methods of Short (1940) showed Sb and As positive, Ag negative.

X-RAY POWDER DATA AND LATTICE PARAMETERS

Unit-cell dimensions and indexed X-ray powder data for paradocrasite are given in Table 3.

For single-crystal work, Finney selected a minute fragment of paradocrasite and split it to obtain a single twin lamella. A series of precession photographs was taken using all three axes as precession axes. The photographs showed $h+k=2n$ for hkl , $h=2n$ for $h0l$, $k=2n$ for $0k0$ reflections, with no violations, indicating space group $C2/m$, $C2$, or Cm and a unit cell having $a=7.24$, $b=4.15$, $c=4.42$ Å, $\beta=123^\circ 10'$. The presence in polished section of one paradocrasite grain clearly showing the analogue of the Carlsbad-albite twinning of plagioclase feldspar indicates the absence of a $\{010\}$ composition plane and thus eliminates $C2/m$ and Cm as possible space groups, leaving $C2$ as the logical choice. Without the optical evidence of lowered symmetry, the combined evidence from single-crystal photographs, chemical analysis, and density determination (6.52 measured vs. 6.44 calculated) would be consistent with the formula Sb_2As with $Z=1$. However, an arrangement of odd Sb and As atoms according to the above formula is inconsistent with the space group $C2$. Therefore, we suggest that the appropriate formula is $Sb_2(Sb, As)_2$ with $Z=1$ and $Sb=As=1$ atom each in the bracketed part of the formula. The preferred formula, pointing to partial disordering of the constituent atoms, further suggests that other paradocrasite-like compounds of variable composition might exist, the limits of variation within $2(Sb,As)$ remaining unknown.

The lattice parameters read from single-crystal photographs were refined by manual calculation and used to index the powder data. These results were further refined by computer according to a Fortran IV program designed by D. S. Handwerker and D. E. Appleman of the U.S. Geological Survey. The calculated values reported in Table 3 are those obtained by least-squares refinement by computer. Reflections in-

TABLE 3. X-RAY POWDER DATA AND UNIT CELL OF PARADOCRASITE.
 $a = 7.252 \pm 0.001$, $b = 4.172 \pm 0.004$, $c = 4.431 \pm 0.002$ Å, $\beta = 123^\circ 08.4 \pm 01.4'$

	1		2		
<i>hkl</i>	$d_{(obs)}$, Å	$d_{(calc)}$, Å	<i>I</i>	$d_{(obs)}$	<i>I</i>
001	3.72	3.710	m	3.72	w
201, 110 ^a	3.46	3.444	vw	—	—
200, $\bar{1}11^a$	3.05	3.035	vs	3.06	vs
$\bar{2}02$, 111	2.219	2.207	s	2.21	m
311, 020	2.093	2.089	s	2.09	ms
$\bar{1}12$, 201	1.903	1.896	mw	1.912	vvw
002	1.855	1.855	mw	1.829	vvw
312, 310, 021	1.821	1.821	vw		
$\bar{4}02$, 220	1.720	1.722	ms	1.730	w
400, $\bar{2}22$	1.518	1.517	m	1.521	w
203, 112	1.456	1.456	w	1.463	vw
$\bar{4}03$	1.407	1.407	w	—	—
$\bar{3}13$, 311, 022	1.389	1.387	mw	1.392	w
$\bar{5}11$	1.330	1.330	mw	1.330	w
$\bar{1}13$, 202	1.299	1.299	vw	—	—
003, $\bar{5}13$, 420, 131	1.229	1.230	mw	1.232	vw
$\bar{6}02$	1.210	1.209	vw		
401, $\bar{2}23$	1.195	1.195	vw		
$\bar{4}23$, 510	1.167	1.166	w		
$\bar{6}03$, $\bar{6}01$	1.150	1.149	vvw		
$\bar{3}14$, $\bar{2}04$, 312, 023, 113	1.065	1.064	mw		
$\bar{6}04$, 600	1.012	1.013	w		
713	.9900	.9896	w		
711	.9461	.9452	w		
$\bar{6}24$.9112	.9114	vw		
802	.8943	.8945	vvw		
422, 332 ^b , 133	.8628	.8629	vw		
710 ^b , $\bar{5}34^b$, $\bar{3}15$, $\bar{2}43$.8490	.8487	vw		
150, 822	.8230	.8243	vw		
512, $\bar{1}34$, 242	.8132	.8132	vw		
$\bar{6}25$, 621, 043 ^b	.7984	.7981	vw		
$\bar{8}05$.7876	.7879	vvw		
$\bar{2}25$.7746	.7740	vvw		

^a Obscured on single-crystal photographs

^b Missing from single-crystal photographs

1. Powder data for paradocrasite in mixture of paradocrasite and subordinate stibarsen, film D-7413. Spindle prepared from crushed handpicked fragments bound in ethyl cellulose dissolved in toluene. Reflections of stibarsen deducted and reported separately in table 6. Camera diameter 114.59 mm; $\text{CuK}\alpha = 1.54178$ Å; Ni filter; cut-off $2\theta \sim 5^\circ$; Straumanis film arrangement; film corrected for shrinkage. Relative intensities estimated visually—s, strong; v, very; m, medium; w, weak. Plus-or-minus values following lattice parameters indicate standard error.

2. Powder data for paradocrasite, film D-7576. Minute pure sample scratched from polished section and mounted on gelatine sliver. Camera diameter 57.3 mm; $\text{CuK}\alpha/\text{Ni}$. Uncorrected for film shrinkage owing to lack of back reflections.

dexed by computer were checked against those recorded on the precession photographs, and crystallographically invalid assignments made by computer were rejected.

The two powder samples independently prepared and photographed by Finney and Leonard showed stibarsen as a minor phase. Reflections due to stibarsen were identified by directly comparing the powder photographs of the mixture with a photograph of allemontite from Allemont, France (film D-6314, specimen USNM 79127). An additional check was made by photographing a pure but minute sample of paradocrasite (2, Table 3). For photographs of the mixture, all reflections not indexable as part of the paradocrasite structure can be indexed as belonging to stibarsen (Table 6, discussed below), though several weak to very weak reflections could be assigned to paradocrasite by violating the evidence of the single-crystal photographs and assuming that the paradocrasite lattice has the space group $P2_1/m$ or $P2_1$. A computer-refined triclinic cell for paradocrasite ($a=7.262\pm 0.002$, $b=4.182\pm 0.003$, $c=7.822\pm 0.002$ Å, $\beta=108^\circ 21.2\pm 01.0'$) is not only inconsistent with the symmetry shown on single-crystal photographs; it also requires $\alpha=90^\circ 28.8\pm 00.8'$, $\gamma=89^\circ 35.4\pm 00.5'$, values that differ considerably from $\alpha=\gamma=90^\circ 00\pm 05'$ measured on the $0kl$ and $hk0$ single-crystal photographs. Therefore, we believe that the evidence from the single-crystal photographs, from the density determination, and especially from the single grain showing combined multiple and pair twinning in polished section favors space group $C2$, with assignment to stibarsen of all reflections not indexable in $C2$.

PROPERTIES OF MINOR CONSTITUENTS

Antimonian loellingite. Antimonian loellingite USNM R-419 in air is bright, lavender-gray to pink. Against paradocrasite, the gray position of antimonian loellingite is distinctly darker than the average brightness of paradocrasite, and the pink position is slightly darker. In oil, antimonian loellingite is much darker than paradocrasite and shows a bluish to greenish tint. The reflection-pleochroism of antimonian loellingite in air is strong—lavender-gray \perp fiber axis, delicate orange-pink and brighter \parallel fiber axis. The colors are faintly zoned. The lavender tint is less evident where the grains are embedded in calcite. In oil reflection-pleochroism is also strong, but fibers look bluish \perp length, olive \parallel length. Anisotropism is very strong in air, slightly intensified in oil; pink and blue tints are rather brilliant in both media. Extinction is \parallel fiber axis. Internal reflection is lacking. Some aggregates of fibers are perhaps twinned. The polishing hardness of antimonian loellingite is much greater than that of paradocrasite and stibarsen. For antimonian loellingite, $HV_{50}=891-1049$,

mean 949, on four slightly fractured grains. Three other grains fractured badly when indented. Reflectance is reported in Table 4.

Microprobe analysis of antimonian loellingite gives, in weight percent, Fe 28.7, As 60.9, Sb 10.9, sum 100.5, equivalent to $Fe_{1.09}(As_{1.72}Sb_{0.19})$ or $Fe_{1.00}(As_{1.58}Sb_{0.17})$. No other elements were detected. This loellingite contains almost twice as much Sb as the antimonian variety from Chalanches, near Allemont, Isère, France, for which Palache, Berman, and Frondel (Dana, 7th ed., 1, 304–305) report 5.61 weight percent Sb, 3.66 percent S. The As-Sb deficiency evident in the analysis of loellingite USNM R-419 is consistent with the findings of Radcliffe and Berry

TABLE 4. REFLECTANCE OF ANTIMONIAN LOELLINGITE AND STIBARSEN.

Wavelength, nm	R_{air} , percent							
	Antimonian loellingite				Stibarsen			
	470	546	589	650	470	546	589	650
Rg'	57.8	56.4	56.7	—	62.5	65.2	64.5	—
Rp'	50.8	52.8	53.0	—	58.8	61.6	59.3	—
$Rg' - Rp'$	7.0	3.6	3.7	—	3.7	3.6	5.2	—
$(Rg' + Rp')/2$	54.3	54.6	54.8	—	60.6	63.4	61.9	—
\bar{R}	53.4	54.4	55.1	^a ~53?	60.2	63.1	62.3	^a ~52?

^a Red-light sensitivity of microphotometer is too low to yield reliable measurements on the minute areas available.

(1968) for the series of non-antimonian loellingites which they investigated.

X-ray powder data and approximate lattice parameters for loellingite USNM R-419 are given in Table 5. The powder data permit a choice between loellingite and safflorite (*cf.* Berry and Thompson, 1962, nos. 128–133), but the probe analysis shows that the mineral is antimonian loellingite. Its cell dimensions are so close to those reported by Radcliffe and Berry (1968) for intermediate members of the loellingite-safflorite series that we would have erred if we had interpreted the composition of this loellingite from diffraction data alone.

Loellingite as a minor constituent in Broken Hill ore was described 30 years ago by Stillwell (1939), who commented on the presence of Sb with Co and Ni in a suite of analyzed samples rich in arsenopyrite. He tested the loellingite microchemically and found Co in it, speculated on the presence of an additional, unidentifiable Co-Ni mineral in the samples, but evidently did not identify the source of the Sb. The problem is further complicated by the occurrence of willyamite, pseudocubic (Co, Ni)SbS

TABLE 5. X-RAY POWDER DATA AND CELL DIMENSIONS OF ANTIMONIAN LOELLINGITE.
 $a=5.25$, $b=6.01$, $c=2.93$ Å

hkl	$d_{(obs)}$, Å	$d_{(calc)}$, Å	I	
—	2.83	—	mw	β of 011, 120
011, 120	2.61	2.63, 2.61	s	
101	2.56	2.56	mw	
210	2.43	2.41	w	
111	2.36	2.36	m	
130	1.873	1.873	m	
310	1.689	1.682	vw	
031, 221	1.648	1.654, 1.640	m	
230	1.582	1.594	vw	

Film D-7568. Camera diameter 57.3 mm, unfiltered $FeK\alpha$ radiation, gelatine-tip mount. Inadequate film record from minute sample. Cell dimensions approximate, calculated from strongest reflections.

in which $Co > Ni$, with some of the loellingite from Broken Hill (Cabri *et al.*, 1970, 96).

Stibarsen. Two terms, allemontite and stibarsen, are still current for the mineral called stibarsen in this paper. As we use the term, stibarsen is the naturally occurring homogeneous, single-phase, hexagonal alloy whose composition is essentially AsSb. Stibarsen corresponds to the "homogeneous subspecies" of allemontite of Quensel, Ahlborg, and Westgren (1937, p. 138) and to "allemontite of type II" of Wretblad (1941, p. 28). Our usage of the term stibarsen follows the unambiguous usage of Ramdohr (1960, p. 348) and Hey (1955, no. 1.38). Ramdohr concisely summarizes Wretblad's findings as follows: allemontite I is an intergrowth of native antimony and stibarsen; allemontite II is stibarsen, homogeneous AsSb; allemontite III is an intergrowth of native arsenic and stibarsen. Whether one regards stibarsen as a species or as a variety is of no concern in the description that follows; our stibarsen is a single phase, not a mixture, and for this reason we prefer to avoid the blanket term allemontite.

Stibarsen USNM R-419 by itself is bright white in air, somewhat darker in oil. Against paradocrasite, stibarsen in air looks slightly pinkish and very slightly darker than its host; in oil it looks slightly grayish and distinctly darker. The pinkish tint is seen in air only if the section has been polished so that the slight positive relief of stibarsen against paradocrasite is accentuated. If the section is polished with a truly plane surface, the pinkish tint is not detectable. Reflection-pleochroism of stibarsen in air is weak, perhaps obscured by the stronger reflection-pleo-

chroism of the enclosing paradocrasite; in oil the reflection-pleochroism of stibarsen is distinct, generally untinted, rarely with an extremely faint pink tint. Anisotropism is distinct in air and in oil; faint pinkish to bluish tints are visible in oil. Internal reflection is lacking. Twin lamellae are detectable between crossed polars if illumination is intense. $HV_{25} = 133-159$, mean 143, measured on 4 grains. This determination of microindentation hardness is not of first quality because the largest areas are only 25 microns wide; this places the indentation less than one diagonal length inside the area boundaries. Reflectance of stibarsen is reported in Table 4.

Semiquantitative microprobe analysis of stibarsen gives, in weight percent, 38 As, 57 Sb, sum 95. In addition, Fe is definitely present, but the quantity could not be reliably determined, owing to the smallness of the stibarsen inclusions and to the difficulty of recognizing an adequate number of them in the carbon-filmed specimen viewed with the probe microscope. Other elements were not detected. The atomic proportions of As and Sb are 0.51 and 0.47, respectively, equivalent very nearly to AsSb if Z is taken as 2.

X-ray powder data and cell dimensions of stibarsen are given in Table 6. The powder data were obtained from a mixture of paradocrasite and stibarsen because pure stibarsen could not be extracted from the polished section. The d values and relative intensities of reflections attributable to stibarsen agree closely with those reported for allemontite by Berry and Thompson (1962, no. 21). The film record for our stibarsen is relatively incomplete, of course, because stibarsen is the minor phase in the X-rayed mixture.

An estimate of the composition of our stibarsen based on a comparison of its hexagonal lattice parameters with those precisely determined by Skinner (1965) for synthetic members of the As-Sb solid-solution series gives mildly contradictory results. The composition estimated from a is 48 mole percent (or atom percent) As, in close agreement with the value determined by microprobe; the composition estimated from c is 75 mole percent As. Some of this discrepancy might result from the imprecise determination of c for our stibarsen, for which no 000 l reflections of high order were recorded on the powder photograph. However, to eliminate the discrepancy, one would have to suppose that $2\theta_{01\bar{1}2}$ was incorrectly measured by more than four times the vernier reading of 0.05 mm. We do not think the measurement was that bad. Instead, we believe that some—perhaps most—of the discrepancy is due to the lattice-contracting effect of the indeterminable but small amount of Fe found by microprobe to be present in this stibarsen. Quensel, Ahlborg, and Westgren (1937, 142-143) likewise attributed to Fe substitution a similar discrepancy in

TABLE 6. X-RAY POWDER DATA AND CELL DIMENSIONS OF STIBARSEN.
 $a = 4.052$, $c = 10.815$ Å; $a_r = 4.298$ Å, $\alpha = 56^\circ 15'$

<i>hkl</i>	$d_{(\text{obs})}$, Å	$d_{(\text{calc})}$, Å	<i>I</i>
0003	3.60	3.604	vw
1010	3.33	3.337	vw
0112	2.944	2.944	ms
1014	2.138	2.142	mw
1120	2.026	2.026	w
1123	1.758	1.766	w
2022	1.667	1.669	vw
0224	1.471	1.472	w
1126	1.353	1.347	vw
1232	1.288	1.288	vw
0118	1.250	1.262	vw
1235	1.134	1.131	vwv
2028	1.078	1.071	vw
2240	1.020	1.013	vw
3036	.9751	.9812	w
3142	.9596	.9579	vw
—	.9260	—	vwv
0339	.8375	.8382	vw
—	.7809	—	vwv

Reflections not assignable to paradocrasite, film D-7413. Experimental conditions are those reported in table 3. Intensities uncorrected for dilution caused by paradocrasite, the admixed major phase. Hexagonal cell edges of stibarsen calculated from best reflections, 1120 and 0112.

the cell volume of analyzed Varuträsk stibarsen relative to the cell volume of their synthetic As-Sb alloys.

If we now compare the rhombohedral cell edge of our stibarsen with the monoclinic cell edges b and c of the host paradocrasite, we find a striking relation: a_r of stibarsen, 4.298 Å, is almost equal to $\frac{1}{2}(b+c)$ of paradocrasite, 4.307 Å. In addition, since β of paradocrasite is $123^\circ 10'$ or $\sim 120^\circ$, we can calculate a pseudorhombohedral cell for paradocrasite. Its pseudo $a_r \approx 12.75$ Å, which is very nearly equal to $3a_r$ of stibarsen ($3 \times 4.298 = 12.89$ Å). And the volume of the monoclinic cell of paradocrasite is approximately twice that of the rhombohedral cell of stibarsen. This sort of relation of the lattices of guest and host, here requiring minimum accommodation of their constituent Sb and As atoms, is so well documented for other exsolution pairs that it strongly supports our interpretation of the microscopic fabric; stibarsen has been exsolved from paradocrasite as blebs within the individual grains of paradocrasite and along the intergranular boundaries. The total quantity of exsolved stibarsen is so small that the bulk composition of the pre-existing single phase must have been close to the present composition of paradocrasite itself.

POSSIBLE MISIDENTIFICATIONS

The *qualitative physical and optical properties* of paradocrasite, as well as its behavior toward standard etch reagents, fail to distinguish the mineral with certainty from other soft, bright, white minerals such as native antimony, stibarsen, dyscrasite, and allargentum. However, examination of these minerals with the aid of a comparison microscope reveals some significant differences in qualitative optical properties. These are discussed before the more diagnostic quantitative properties of reflectance, microindentation hardness, X-ray diffraction behavior, and chemical composition are considered.

By direct comparison³ in white light, in air, under a comparison microscope, paradocrasite looks very similar to native antimony. Paradocrasite, having greater bireflectance, looks slightly brighter and darker than antimony, and the brightest position of paradocrasite is very slightly yellower than the corresponding position of antimony. Against stibarsen, paradocrasite is distinctly brighter and shows greater bireflectance; details were reported in a separate section of this paper. Against each of the four dyscrasites, paradocrasite looks almost equally bright but is distinctly white against the cream or yellow tint of the dyscrasites; the difference in reflectance is difficult to assess in white light, but the difference in color is pronounced in the large areas available for comparison. Against allargentum, paradocrasite looks white; allargentum looks yellow and considerably brighter. The slight qualitative distinctions among these minerals are emphasized by direct comparison. They might easily go unnoticed in small, isolated grains.

The quantitative properties of *mean reflectance* and *mean microindentation hardness*, taken together, help to identify paradocrasite. Indeed, if the data for all ore minerals were absolutely correct, the unique *R-HV* number pair for paradocrasite would fully distinguish this mineral from its closest neighbors in an updated Bowie-Taylor diagram. However, absolute correctness of the data, including our own, is beyond expectation. Figure 5 shows the relations with generous allowance for overlap. The diagram contains averaged data for all ore minerals whose R_{air} in green light is 70 ± 7 percent and whose extremes of *HV* overlap those of

³ Specimens verified by X-ray powder diffraction and used for direct comparison were: native antimony, USNM 90597, Sarawak, Borneo; stibarsen sparsely intergrown with native arsenic ("allemontite"—both components verified by X rays), USNM 79127, Allemont, France; stibarsen intergrown with paradocrasite and described in detail above; dyscrasite (pure), USNM R-419-1, Broken Hill, New South Wales; dyscrasite (pure), RTO 318, Andreasberg, Harz; dyscrasite sparsely intergrown with a fine net of allargentum, RTO 319, Calliope mine, Ouray County, Colorado; dyscrasite abundantly intergrown with lamellae of allargentum (both components verified by X rays), RTO 320, Cobalt, Ontario. The dyscrasites differ among themselves in qualitative optical properties and in interplanar spacings, but the differences are slight.

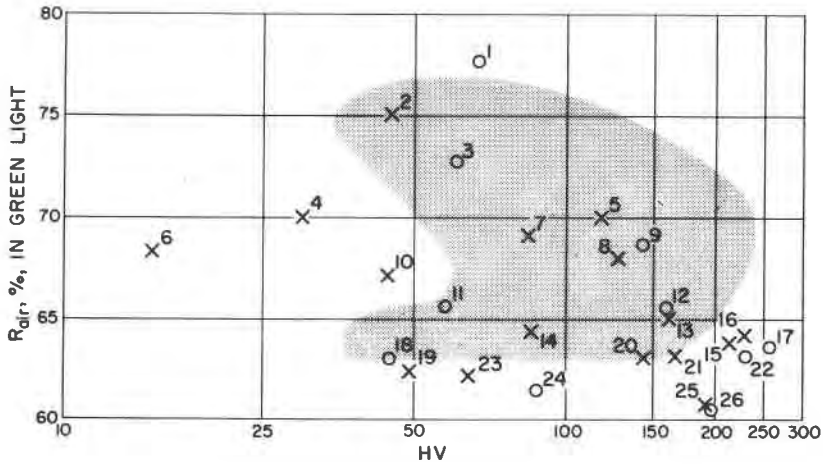


FIG. 5. Mean reflectance *versus* mean microindentation hardness for some relatively soft, bright ore minerals. Semilogarithmic plot. Circles—optically isotropic; crosses—optically anisotropic. Shaded area: $R=70\pm 7\%$ and range of HV overlapping that of paradocrasite (no. 5). Clear area: R similar to above but no HV overlap.

1 Electrum (~700 fine)	14 Krennerite
2 Zinc (artif.?)	15 Calaverite
3 Gold	16 Algodonite
4 Potarite	17 Pd_3Pb of Cabri and Trail (1966)
5 Paradocrasite	18 $(Pb,Bi)_3Te_4$ of Rucklidge (1969)
6 Bismuth	19 Tellurium
7 Antimony	20 Stibarsen
8 Allargentum	21 Arsenian copper, phase II
9 Platinum	22 Nickel (artif.)
10 Altaite	23 Hedleyite (synth., in part)
11 Arsenian copper, phase I	24 Copper
12 Iron	25 Melonite, var. I
13 Dyscrasite	26 Kamacite

paradocrasite. The “uncertainty overlap” is not plotted, for the lines or ellipses indicating range of properties would make the diagram unreadable. For most minerals, the range for R is perhaps 2 to 5 percent, relatively, and for HV about 10 percent. Data for all minerals except paradocrasite and stibarsen are from the literature; for several minerals, including allargentum, these data are sketchy.

Figure 5 shows that in addition to the minerals one intuitively supposes should resemble paradocrasite—antimony, stibarsen, allargentum, and dyscrasite, whose main white-light qualitative properties have already been directly compared—four other close neighbors in the diagram might cause confusion. These are zinc(2), platinum(9), iron(12), and krennerite(14). Zinc as a mineral is rare, and even the properties of the

artificial substance are not fully recorded. Platinum and iron are optically isotropic and thus readily distinguished from paradocrasite. Krennerite is, according to Ramdohr (1960, p. 398), cream-white; its reflection-pleochroism in air is weak; its anisotropism is quite strong under intense illumination. Judicious use of the information compiled in Figure 5, supplemented by good qualitative observations, should therefore be of considerable help in identifying paradocrasite. Quantitative color data would be a valuable supplement, but these are not now available for any of the minerals likely to be mistaken for paradocrasite.

The *powder pattern* of paradocrasite resembles those of stibarsen and the telluride calaverite. (Compare data of Tables 3 and 6, and Berry and Thompson, 1962, nos. 21 and 147.)

The resemblance between the powder patterns of paradocrasite and stibarsen is close enough to permit mistaking one for the other if the comparison is made too casually, or if either mineral is the minor constituent in a mixture. Confusion should not arise if measurements of interplanar spacings are made with care, or if powder photographs of the two minerals are compared directly and carefully. Direct comparison is especially instructive, for the pattern of paradocrasite shows many additional weak reflections that are absent from the pattern of stibarsen. If one were to overlook this obvious difference in symmetry and place too much reliance on the intensities and spacings of the three major reflections, one might mistakenly interpret the two different minerals as members of a single solid-solution series.

The resemblance between the powder patterns of paradocrasite and calaverite is less close, and the difference in the intensity of the reflection at $2\theta_{\text{Cu}} \approx 43^\circ$ (the third strong line on the photos) is conspicuous. In paradocrasite, the intensity of the third strong line ($d_{020} = 2.093 \text{ \AA}$, s) is nearly the same as that of the second ($d_{111} = 2.219 \text{ \AA}$, s), whereas in calaverite the third strong line ($d = 2.09 \text{ \AA}$, vs) is considerably stronger than the second.

The *chemical composition* of paradocrasite is, of course, appropriate for a member of the hexagonal Sb-As series, but the single-crystal data prove that paradocrasite is monoclinic.

Paradocrasite stands as an independent species adequately defined by the properties reported here. However, no one class of properties except the lattice parameters and the X-ray powder data dependent thereon will uniquely identify the mineral at present.

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logical Survey, made and read the original X-ray powder photograph of the unidentifiable species whose composition was shortly determined by Mrs. Mead. Other associates in the Geological Survey contributed as follows: R. L. Parker and W. N. Sharp made special X-ray equipment available; R. B. Taylor made the photomicrograph shown as figure 1 and advised us on photographing other areas of the specimen; R. E. Van Loenen prepared the diffraction data for processing by computer. We are grateful to these people for their generous help.

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