

Erdite, a new hydrated sodium iron sulfide mineral

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Abstract

The new mineral erdite, $\text{NaFeS}_2 \cdot 2\text{H}_2\text{O}$, has been found in abundance in a mafic, alkalic diatreme at Coyote Peak, Humboldt County, California. Crystallization of erdite, the first sodium-bearing sulfide to be found in nature, was dependent upon unusual, late-magmatic chemistry; its preservation can be ascribed to the youthful age (29 m.y.) and the remarkably dense and impervious nature of the mafic host rock.

Erdite is a striking mineral, showing a copper-red color on fresh surfaces and an extreme reflection pleochroism, brilliant reddish orange to dark gray, in polished surfaces. Erdite is monoclinic, $C2/c$, $a = 10.693(1)$, $b = 9.115(1)$, $c = 5.507(4)\text{\AA}$, $\beta = 92^\circ 10(2)'$, $V = 536.3\text{\AA}^3$, $Z = 4[\text{NaFeS}_2 \cdot 2\text{H}_2\text{O}]$; density (calc.) 2.216, (meas.) $2.30(1)\text{ g/cm}^3$ for samples containing about 3% magnetite. The six strongest lines of the X-ray diffraction powder pattern are: 6.935, 110, (100); 5.342, 200, (71); 4.556, 020, (41); 3.467, 220, (28); 2.310, 330, (23); 2.902, 221, (15). Reflectance in air at 540 nm is 8.8% for R_1 , 20.7% for R_2 . Microindentation hardness (VHN) at 15-gram load is 22-67, mean 39.

Introduction

During reconnaissance geologic studies of the Redwood Creek area, Humboldt County, California, in 1976, the bizarre, pitted weathering features of numerous exceptionally hard stream boulders attracted attention. Further investigation showed that these rocks originated from a mafic igneous plug (300 \times 500 meters) that cuts the Franciscan assemblage and forms the ridge-top protuberance called Coyote Peak. Subsequent chemical and mineralogic studies have shown this occurrence to be exceptional, if not unique, and have disclosed the presence of at least four new sulfide minerals. Preliminary information concerning the occurrence is reported by Blake (1977) and Czamanske *et al.* (1977). Recent studies by Czamanske *et al.* (1978) have established the age of the body at 29 ± 1 m.y.

The mineral erdite was first noted in polished sec-

tion as tiny, red-orange patches peripheral to a large knot of pyrrhotite and rasvumite. Additional collecting trips have shown that erdite is readily found in certain rock variants and easily identified by its copper-red color. The new mineral and the name were unanimously approved by the Commission of New Minerals and Mineral Names of the International Mineralogical Association in February, 1978. The name honors Richard C. Erd, mineralogist with the United States Geological Survey. It is especially appropriate to name the mineral for him because he synthesized the compound accidentally during research on smythite (Erd *et al.*, 1957, p. 330, Table 4, Experiment 3). Type specimens will be deposited at the Smithsonian Institution.

Occurrence and paragenesis

Erdite is typically associated with other sulfide minerals and magnetite in discrete, late segregations

(0.5 to 8 cm) within the mafic, alkalic host rock. Non-sulfide minerals adjacent to and within these segregations include coarse phlogopite and schorlomite crystals (each to 5 mm across), clinopyroxene, nepheline, natrolite, sodalite, vishnevite, acmite, apatite, and barytolamprophyllite. No well-formed crystals of erdite have yet been found, even in those instances where the mineral lies adjacent to rare small vugs.

Erdite is typically associated with fine-grained, euhedral magnetite crystals (Figs. 1a and 1b) that are interpreted to have formed contemporaneously. Erdite appears to be one of the last formed of the sulfide phases. The earliest formed sulfides in the segregations are ordered-“hexagonal” or monoclinic pyrrhotite (Scott and Kissin, 1973), averaging 47.5 and 46.7 atomic percent Fe, respectively. Sulfides of intermediate stage are djerfisherite [$K_6Na(Fe, Cu, Ni)_{24}S_{26}Cl$], a new K,Fe-sulfide closely related to djerfisherite, and rasvumite (Sokolova *et al.*, 1970; shown to be KFe_2S_3 by Czamanski *et al.*, 1979). Among these, the new K,Fe-sulfide and rasvumite replace pyrrhotite, whereas erdite replaces djerfisherite (Fig. 1a). Erdite has not been found in contact with the new K,Fe-sulfide, and the relations of rasvumite to djerfisherite and erdite to rasvumite are equivocal.

As suggested by Figure 1a, granular masses of impure erdite may reach dimensions of several millimeters. The largest clean single crystals of erdite (*e.g.*, upper portion of Fig. 1a) measure 0.5×1 mm. Because of its striking optical properties, fine blades

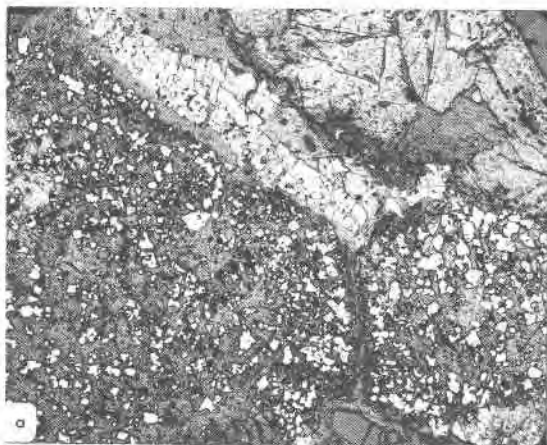


Fig. 1a. Reflected light photomicrograph of an area (0.97×1.23 mm) in which erdite is the dominant phase. An extensively replaced band of djerfisherite extends downward from the upper margin of the photograph, separating a lower region in which erdite grains are studded with fine magnetite from an upper region in which the erdite is free of magnetite.

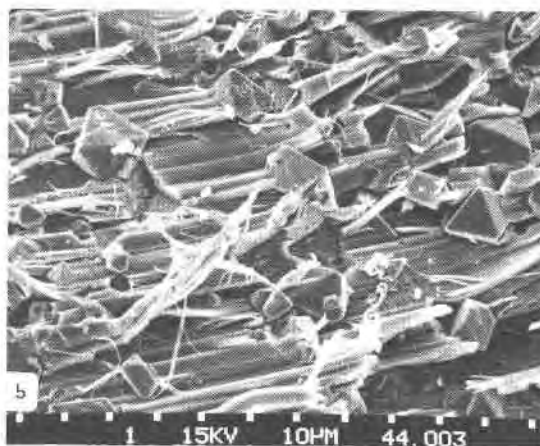


Fig. 1b. SEM photograph of a freshly broken surface showing the fibrous nature of erdite and the intimate nature of its intergrowth with perfect magnetite octahedra. Marks at lower margin are at $10\text{-}\mu\text{m}$ intervals.

down to 0.01×0.075 mm are readily noted in freshly polished surfaces.

Composition

Discovery of a hydrated sodium iron sulfide is, of course, not a routine matter. The first noted grains were small and poorly polished, and many hours of confusion were spared by the fact that these grains were first examined on our microprobe as a multi-channel analyzer was being demonstrated. There, unmistakably, was the spectrum showing Na, Fe, and S! Unfortunately, resolution of the chemical composition of erdite has presented unusual difficulty.

Subsequent analyses were performed on the ARL EMX-SM microprobe at Menlo Park, California at both 10 and 15 kV, using a variety of standards (Table 1). Because erdite polishes poorly and alters readily, several attempts were made to obtain good summations. Nineteen analyses of different grains, utilizing different standards and operating conditions, produced concentration ranges as follows (wt. percent): Na 13.6–15.3; Fe 35.2–37.3; S 39.3–41.9. Sums were typically 91.5 ± 1.5 wt. percent.

Because of the consistently low summations, and considering the unusual optics of the mineral, the presence of O, OH, or H_2O was eventually suspected. The presence of “about 10 weight percent oxygen” was established by George Desborough (personal communication), using a TAP crystal on the ARL microprobe in the U.S. Geological Survey laboratories in Denver, Colorado. Concurrently, a small amount of dry erdite was found to liberate water

Table 1. Electron microprobe analyses of erdite compared with theoretical compositions

Element	Electron microprobe wt. % Average for 4 samples*	Theoretical wt. %	
		NaFeS ₂ ·H ₂ O	NaFeS ₂ ·2H ₂ O
Na	14.1	14.28	12.84
Fe	36.0	34.69	31.20
S	40.5	39.83	35.82
O	9.4**	9.94	17.88
H		1.26	2.26
Σ	100.0	100.00	100.00

*Electron microprobe at 10 kV; standards used were (1) synthetic FeS, (2) pure natural rasvumite, (3) natural sodalite containing 17.73 wt. % Na, (4) natural crocidolite containing 4.63 wt. % Na, 27.14 wt. % Fe. Data were corrected by the theoretical scheme FRAME (Yakowitz *et al.*, 1973).

**Oxygen determined by difference. Cl, F, K, Mg, Cu, Ni were not detected.

when heated in a dry tube, indicating the presence of OH or H₂O rather than O.

Several sets of superior microprobe data were recalculated on the basis that oxygen is part of the formula but is determined by difference. A summary of those data appears in Table 1. These data, and the compositional ranges noted above, clearly establish the 1:1:2 stoichiometry of Na:Fe:S and suggest that the formula of erdite is NaFeS₂·H₂O.

It therefore has been a cause of considerable concern that solution of the crystal structure of erdite (Konnert and Evans, 1980) has shown conclusively that the material has two water molecules per unit cell, indicating the formula to be NaFeS₂·2H₂O. This problem has not been resolved, but the following commentary is offered for consideration.

Microprobe analysis involves subjecting polished surfaces to vacuum, both during carbon coating and during analyses. However, the smoothest, brightest surfaces, chosen for analysis by study of freshly polished surfaces, remain smooth and bright throughout the analysis procedure. It seems difficult to imagine that 8 weight percent water (Table 1) could be lost without disrupting polished erdite surfaces. To further check the stability of erdite in the microprobe environment, "burn-out" tests were made at 10 and 15 kV, whereby the same spot was subjected to twenty 10-second counts. At 15 kV, count rates were erratically perturbed, but at 10 kV calculated analyses based on 20 counting intervals on the same spot are indistinguishable from analyses representing 20 distinct beam placements.

Although no Na,Fe sulfide was available as a stan-

dard, considerable experience has been obtained for K,Fe sulfides, and it seems highly improbable that the microprobe determinations of Fe and S are substantially in error. These values in turn have aligned exceptionally well with Na values obtained using a crocidolite standard (containing 27.14 wt. percent Fe) to establish the 1:1:2 stoichiometry of Na:Fe:S. In our experience there is no precedent for the observed situation in which repetitive, careful microprobe analyses point unequivocally to a constant composition that is erroneous.

It is well established that erdite alters readily on broken or polished surfaces. The mineral must be in the process of alteration during X-radiation of powders and small crystals. However, all X-ray data—diffractometer traces, single-crystal precession photos, and single-crystal intensity data—are compatible with one another. Much of the single-crystal data was obtained from one crystal, over a period of nearly four months, yet in two additional months' time the crystal disintegrated!

Clearly, there exist two possible explanations for our dilemma: (1) the mineral contains two moles of water and loses one prior to microprobe analysis; (2) the mineral contains one mole of water and takes up another during preparation for X-ray analysis. Because of arguments presented above, alternative (1) appears unlikely unless the pressure of polishing removes one water molecule and creates a new phase. (H. T. Evans, personal communication, 1979, has assured us that the determined structure cannot accommodate loss of a water molecule.) No totally satisfactory solution to our dilemma is apparent, and we acknowledge that we may describe not one, but two minerals. However, our microprobe, X-ray, and optical data are presumably typical of what others who find the phase will obtain, and further apology is superfluous. The mineral will be defined as the dihydrate, in deference to the crystallographic data.

Erdite is easily soluble in cold dilute acids with effervescence of H₂S. When the mineral is heated in a closed tube, in addition to first giving off water, it also gives off sulfur and eventually turns black and strongly magnetic. Its initial decomposition product in the closed tube is a pyrrhotite-type iron sulfide: the end product is magnetite. In polished and broken surfaces erdite alters rather readily, developing a black film that eventually obscures its presence. Microprobe analyses of tarnished erdite show notably lower Na and S contents and support the logical assumption that the alteration process involves loss of sodium.

X-ray diffraction single-crystal and powder data

Crystals of erdite were examined with the Buerger precession camera using Zr-filtered Mo X-radiation; the $h0l$, $h1l$, and $0kl$ nets were taken. Erdite has monoclinic symmetry, and the absence of hkl reflections with $h + k = 2n + 1$ and $h0l$ reflection with $l = 2n + 1$ indicates either space group $C2/c$ or Cc . The crystal morphology does not provide evidence for the lack of a center of symmetry, and the successful crystal-structure analysis (Konnert and Evans, 1980) confirms assignment of centrosymmetric $C2/c$. The cell parameters, obtained by least-squares refinement of powder data using the computer program of Appleman and Evans (1973), are: $a = 10.693(1)$, $b = 9.115(1)$, $c = 5.507(4) \text{ \AA}$, $\beta = 92^\circ 10(2)'$, cell volume = 536.3 \AA^3 , $Z = 4[\text{NaFeS}_2 \cdot 2\text{H}_2\text{O}]$.

The X-ray diffraction powder data are listed in Table 2, where the observed intensities are compared with intensities calculated from the known crystal-structure information using the computer program POWDER by Cyrus Jahanbagloo (1964). The agreement is generally satisfactory, but the observed intensities for the only three general hkl reflections oc-

curing in the list are appreciably lower than the calculated intensities for these reflections. This effect may be due to the fibrous nature of the mineral, because the fiber axis is c , and the observed list is dominated by $hk0$ reflections.

Physical properties

Erdite occurs as small clusters of minute fibers that are prismatic $\{110\}$ with good $\{110\}$ cleavage. The aggregates of fibers and grains are irregular, subhedral, or ragged, the edges of ragged aggregates resembling frayed cloth. On a freshly broken surface, erdite resembles copper, both in its copper-red color and in its metallic luster. The streak is black.

The density measured by suspension in bromoform-acetone mixtures is $2.30(1)$. However, the fragments used in this determination were found, upon dissolution in HCl, to contain about 3% intergrown magnetite (observed specific gravity 5.175). The calculated density for 4 $[\text{NaFeS}_2 \cdot 2\text{H}_2\text{O}]$ per cell, 2.216 g/cm^3 , agrees well with the observed value when the latter is corrected for the small amount of magnetite present.

Erdite appears to be weakly magnetic, but, owing to the omnipresent intergrown magnetite this property is difficult to determine. The purest grains of erdite were the least magnetic, but a powder of fine erdite fragments relatively free of magnetite does show attraction to a magnetized needle.

The microindentation hardness of erdite at a load of 15 g ranges from 22 to 67, mean 39 ± 16 , for seven grains indented with the Vickers pyramid of the Leitz Durimet hardness tester. All indentations are accompanied by fractures parallel to the sides of a figure or radiating from them. Some indentations have short fractures at one corner of the figure, on the extension of the diagonal. The figures are equant or elongate, and their sides are cymoid or concave but jagged. Owing to the small number of grains of suitable size, as well as to the shattering of grains showing high birefractance, we have probably not succeeded in measuring the extremes of microhardness of the mineral.

Optical properties

Reflected light

The properties of erdite in polished section are striking. In air, reflection pleochroism is extreme. The brighter position (R_2) of the freshly polished mineral is brilliant reddish orange; the darker position (R_1) is dark gray with a barely perceptible bluish or greenish tint. One's first impression is that R_2 of

Table 2. X-ray powder diffraction data for erdite

hkl	Calculated*		Observed**	
	d_{hkl} (Å)	I	d_{hkl} (Å)	I
110	6.935	100	6.935	100
200	5.343	84	5.342	71
020	4.557	34	4.556	41
111	4.363	16	4.366	4
111	4.260	5	---	---
021	3.510	1	---	---
220	3.467	28	3.467	28
310	3.318	4	3.317	12
221	2.966	24	2.966	8
130	2.922	3	---	---
221	2.902	61	2.902	15
330	2.312	25	2.310	23
040	2.279	3	2.278	5
240	2.096	1	2.096	7
150	1.797	7	1.797	7
600	1.781	2	1.779	5
530	1.748	11	1.748	11
440	1.734	10	1.734	9
620	1.659	2	1.659	4

*All calculated spacings listed for $d_{hkl} \geq 2.900 \text{ \AA}$. Indices from cell parameters obtained by least-squares analysis of X-ray powder data using the program of Appleman and Evans (1973). Calculated intensities obtained using computer program POWDER (Jahanbagloo, 1964) with the atomic parameters from Evans and Konnert (1980), scattering factors for neutral atoms Fe, S, O, ionized Na^+ , and fixing $I(110) = 100$.

**Specimen No. 77-CYP-125. X-ray diffractometer conditions are: chart no. X-4309; Cu/Ni radiation; $\lambda\text{CuK}\alpha_1 = 1.540598 \text{ \AA}$; silicon used as internal standard; scanned at $1/4^\circ$ minute from 4° - 62° 2θ .

erdite resembles R_o of idaite but is considerably brighter, and that R_i of erdite resembles R_E of graphite. The latter resemblance is enhanced by great softness and by an abundance of minute polishing scratches and tiny pits. R_2 is inclined 3° – 5° to the trace of a perfect cleavage. Fibers are either orange or greenish gray parallel to their length. The anisotropism of erdite, like its reflection pleochroism and bireflectance, is extreme—black to bright orange-yellow between perfectly crossed polars. Internal reflection is absent. In oil, R_2 is little changed but R_1 becomes much darker, anisotropism remains about the same as in air, and internal reflection is probably absent, though minute dents in the polished surface distort the fibers and give an effect that somewhat resembles internal reflection.

The foregoing description of color effects applies to grains of near-maximum bireflectance. Oblique sections show reflection pleochroism in greenish gray, bluish gray-green, pinkish gray, and purple. In oil, in sections that look purple in air, the tint is suppressed in some grains but made murky orange-red in others.

Erdite tarnishes rapidly. The reddish-orange of the position of the maximum bireflectance becomes redder; reddening and darkening progress to give a purplish red resembling that of the vibration direction for the ordinary ray of umangite, but brighter. Simultaneously, the greenish tint of the position of minimum bireflectance is enhanced. In a dry climate, this tarnish is noticeable in one to four days. In a maritime climate, some grains totally blacken within two weeks.

Reflectance data at standard wavelengths are given in Table 3 for 10 grains of erdite, the complete dispersion record for R_1 and R_2 of the mineral is given in Table 4, and the reflectance curves are illustrated in Figure 2.

A comparison of Tables 3 and 4 shows that no single grain among the 10 exhibits both the maximum and the minimum values of reflectance for the mineral. Therefore, the data of Table 4 were obtained from two grains, one showing the lowest reflectance throughout the spectrum, the other showing the highest. R_1 and R_2 throughout the spectrum are here referred to the extinction positions at 546 nm, as the presence of fine scratches made it infeasible to reorient the grains to extinction at other wavelengths. This procedure may cause some truncation of the reflectance reported at other wavelengths.

Some discrepancies exist between the values initially measured and reported in Table 3 and those

Table 3. Reflectance (R^{air} ,%) of erdite for weakly to strongly bireflecting grains, measured at standard wavelengths

Grain no.	Wavelength, nm							
	470		546		589		650	
	R_1	R_2	R_1	R_2	R_1	R_2	R_1	R_2
1	8.9	9.6	8.7	10.8	9.9	10.4	11.9	14.5
2	7.8	9.4	9.1	12.9	8.8	16.6	8.1	28.1
3	-	-	10.3	16.2	-	-	-	-
4	-	-	10.7	11.1	-	-	-	-
5	-	-	11.0	13.5	-	-	-	-
6	-	-	11.0	13.8	-	-	-	-
7	9.5	14.5	11.5	20.1	10.3	26.6	13.6	36.8
8	-	-	12.1	15.6	-	-	-	-
9	-	-	12.8	16.6	-	-	-	-
10	-	-	15.0	17.0	-	-	-	-

Reflectance measured by B. F. Leonard with a Zeiss MPM microphotometer fitted with a Smith vertical illuminator and a Veril type S running interference filter whose half-width at half-height is 10 nm. Zeiss-calibrated SiC standard no. 052. Mounts press-leveled on plasticine. Final polish with 0.05 alumina in water on Buehler microcloth. Reproducibility of individual measurements is $\pm 1\%$ relatively.

subsequently measured and interpolated from Table 4. The discrepancies are attributed mainly to the difficulty of precisely recovering the sites of the initial measurements; the sites were recovered within a few micrometers, but recovery within a micrometer or less would be desirable to eliminate the microphotometer's seeing minute differences in the size or density of the inescapable polishing scratches. Thus, the discrepancies in R are probably greater at shorter and longer wavelengths because in the middle of the spectrum the microphotometer is less affected by polishing imperfections.

Before discussing Figure 2, we note that the average value, $R(546)$, for the more strongly bireflecting grains (nos. 3, 7, 8, 9, 10) of Table 3 is $14.7 \pm 3.0\%$, in close agreement with $0.5(R_1 + R_2) = 14.5\%$, the value interpolated at 546 nm from Table 4. The mean or mid-range value is useful for placing the data on er-

Table 4. Reflectance of erdite measured at 20nm intervals

Wavelength nm	R^{air} , %		Wavelength nm	R^{air} , %	
	R_1	R_2		R_1	R_2
440	6.6	15.3	580	8.8	22.8
460	6.8	15.8	600	8.7	26.7
480	7.0	13.5	620	9.6	31.1
500	7.4	11.7	640	9.9	34.7
520	7.6	16.7	660	10.0	34.5
540	8.8	20.7	680	9.9	30.4
560	8.5	19.4	700	9.9	26.5

R_1 measured on grain 2, R_2 on grain 7 (Table 3). Extinction positions of R_1 and R_2 referred to 546 nm. See footnote, Table 3, for other conditions of measurements.

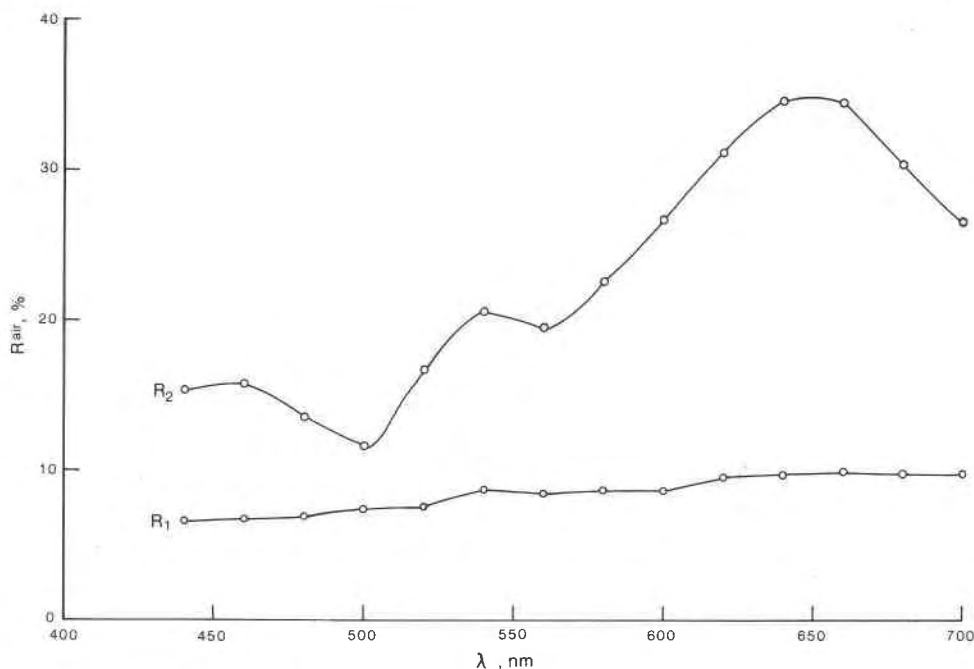


Fig. 2. Reflectance (R) curves for erdite.

dite in a table of generalized reflectance values. The mean or mid-range value is not, in general or in this case, equivalent to R_m , the reflectance in the vibration direction perpendicular to the R_g - R_p plane. In monoclinic substances, this vibration direction is along the unique symmetry axis (here, b), and the unequivocal determination of R_m requires having an oriented section that is parallel to b . However, because the angle β of erdite is close to 90° , the overlap method of Cameron (1963) should be applicable, with little uncertainty, to the statistical determination of R_m for the new mineral. The value of R_m' so obtained from the data of Table 3 is $11.5 \pm 0.5\%$ at 546 nm. If this nearly constant value closely approximates R_m , the sign of the bireflectance of erdite is (+) at 546 nm. The suggested sign of the bireflectance obviously requires confirmation by measurements made on oriented sections, but these cannot be had from the material at hand.

The reflectance curves shown in Figure 2 are remarkable for their disparity, but they are consistent with qualitative observations and with the initial measurements of reflectance at standard wavelengths. The shape of the curves is diagnostic and, we think, reliable, though absolute values obtained on sections of indeterminate crystallographic orientation are always open to question. The presence of in-eradicable scratches and the speed with which the mineral tarnishes lend some uncertainty to the qual-

ity of the measurements in spite of their high precision, which is generally less than one part in 200 for R_2 and approached one part in 100 for R_1 . The curves for R_2 and R_1 do not cross within the 440–700 nm range, and it appears unlikely that they would cross between 400 and 440 nm. However, the observations may be biased by the adoption of the extinction positions at 546 nm as lines of reference.

Color values derived from R_2 and R_1 are given in Table 5. For R_2 , the agreement between quantitative and qualitative observations is good: the dominant wavelength, $\lambda = 591$ nm, is on the red side of orange, and the color seen by eye is pinkish orange. For R_1 , the agreement is not so good; λ_d of 578 nm is yellower than the color seen by eye—dark gray with a barely perceptible bluish or greenish tint, as previously described. While one might fault either the instrumental skill or the eye of the observer, a rea-

Table 5. Quantitative color designation of erdite referred to the International Commission on Illumination's illuminant C

	Trichromatic coefficients		Brightness value	Dominant wavelength	Saturation
	x	y	Y, %	λ_d , nm	P_e , %
R_2	0.376	0.341	21.2	591	24.1
R_1	0.337	0.342	8.5	578	14.0

Method of 30 selected ordinates. For details, see Piller, 1966.

sonable view is that the instrumental and visual results, though different, are equally valid. Very likely the difference is due to the color memory of the observer. Having seen and remembered the bright pinkish orange of R_2 , the observer senses the complementary color—blue—in the rather dark gray of R_1 . The x - y coordinates of R_1 , R_2 , and illuminant C are not collinear, but the points are close enough to being so to indicate that the phenomenon of complementary color comes into play when one observes R_2 and R_1 in rapid succession.

Transmitted light

Light is transmitted by very fine fibers of erdite; the largest non-opaque crystal measured 60 μm in length by 3 μm in diameter. It was not possible to measure the indices of refraction of erdite, as the mineral is immediately attacked by high-index immersion media forming a reaction product that rims all grains. Erdite has extremely high birefringence and shows inclined extinction up to a maximum of 5° to the fiber length. The mineral also exhibits a very strong pleochroism—reddish brown perpendicular to the length and very dark reddish brown (almost opaque) along the length of the crystal.

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