Northwest
Micro Mineral
Study Group

MICRO PROBE

FALL, 2009 VOLUME X, Number 10

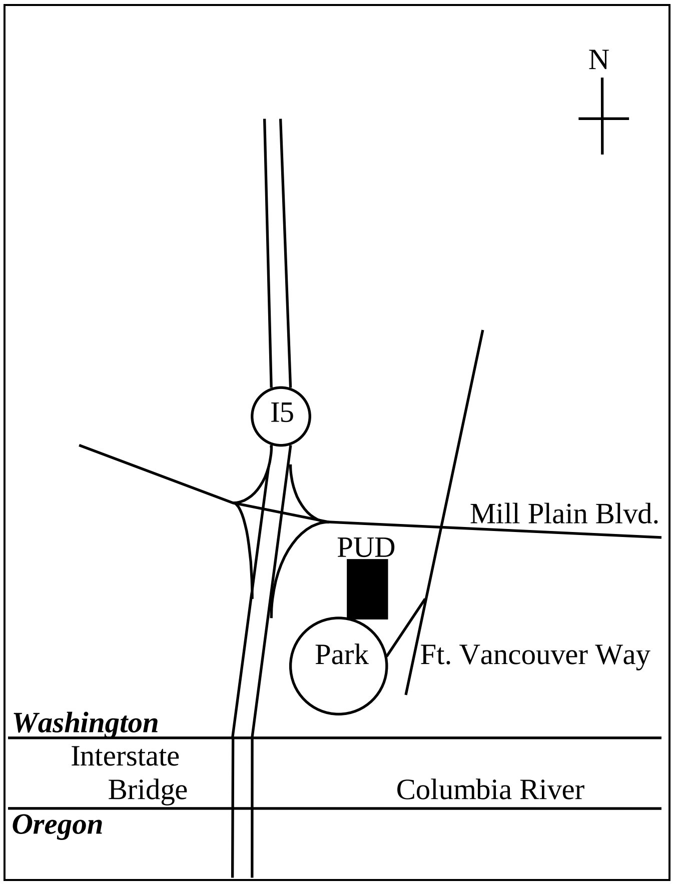
FALL MEETING . . . . .VANCOUVER, WASHINGTON

November 14, 2009 9:00 am to 5:00 pm

Clark County P. U. D. Building
1200 Fort Vancouver Way
Vancouver, Washington

Schedule for the day;

9:00 am	Doors open at the PUD building for table set up. Helpers needed.
9:30 am	Meeting starts: (trading, selling, free tables, viewing specimens, and visiting).
10:30 am	Business meeting, including field trip reports and mineral news.
12:00 noon	Lunch potluck: Club provides sandwich makings (bread, meat, lettuce, cheese, dressings, and coffee, tea, coco). Rudy will bring potato and macaroni salads. Please bring chips, pop, nuts, chili, cookies, pie, or cake to add to the lunch.
1:00 pm	Main Program: We have an extremely good major program on Gadolinite and other minerals from Washington Pass presented by Saul Krotki.
2:00 pm	Additional programs on field trips by the membership.
3:00 pm	Study trays of minerals, free tables, and visiting.
4:00 pm	End of meeting and clean up. Please stay to put away tables and help clean up.
5:00 pm	Dinner will be at the Country Buffet in Vancouver. Please join us if you can.



A New Find of Gadolinite-(Ce)
Washington Pass, Okanogan Co., Washington

Saul Krotki

CRYSTALS IN AN UNUSUAL ALKALI GRANITE AT WASHINGTON PASS

Numerous, minute crystals appearing to have orthorhombic symmetry were collected by the author near milepost 165, North Cascade Highway, Washington Pass, Okanogan County, Washington, in September 2008. The crystals were found in unusual alkali granite, which appears blue-gray, especially when wet, owing to approximately 1.5% dispersed microscopic needles of black amphibole.

The unusual rock is a sparsely scattered rockslide material, carried down each year by the avalanche chutes above the road. Stull (1969) maps the alkali granite, in situ, ½ mile west, and 1000 feet above the road at milepost 165. It occurs in contact with one-feldspar biotite granite, which is generally not productive, but is the most prevalent rock at the road level.

The productive rock displays small miarolitic cavities, generally 1.0-3.0 cm across, with pale yellow-orange iron stains around the cavity rims. Mica, quartz, and microcline with albite overgrowths dominate the appearance of all the cavities.

The crystal components of the cavities in the blue-gray granite are unusually fresh, especially the Mica Group minerals, and so inspire careful examination. Many questions concerning the paragenesis of the minerals come to mind. Boggs (1984a) presents a thorough description of the specific Washington Pass species, as well as a discussion of the physical and chemical conditions of crystal growth in late stage magmatic fluids (Boggs 1984b). Observations gathered in this study agree with the paragenetic sequences offered by Boggs (1984c).

Unlike the pegmatitic granite collected at milepost 166, this rock has no crystallized amphibole within the miarolitic cavities. Descriptions of the species that are found in the cavities are discussed under "Associated Species", near the end of this report.

This report is intended to contribute to our knowledge of the Washington Pass mineral occurrences, as well as offer encouragement to collectors in pursuit of new specimens. Washington Pass is, after all, a locality where, in failing to find a certain sought-after species, a prospector with the right patience is destined to find another!

SPECIES IDENTIFICATION

An initial microprobe examination of numerous crystal fragments, measuring 0.5-2.0 mm, was accomplished at Cannon Microprobe, Seattle, Washington, November 2008, along with zone studies of select single crystals. Relative EDS peaks indicate a cerium-rich, rare earth iron silicate at the core, with yttrium rich outer zones or crusts (Fig.1). EDS spectra also indicate various proportions of other light rare-earth elements as well as thorium. Spectra acquired, however, do not match any species on my list of orthorhombic possibilities. After twenty-two probes, the unknown remains an unidentified species.

With John S. White's recommendation, the author sent a vile of crystals to John Attard in San Diego for examination by X-ray diffraction.

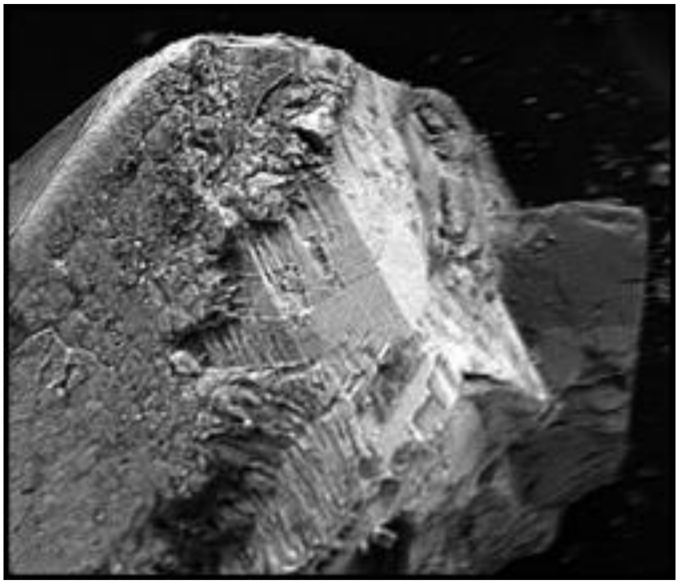


Fig.1. Preliminary EDS spectral analysis of this grain sample revealed a cerium rich interior with a yttrium rich outer crust. BSE/SEM, Cannon Microprobe, Seattle, WA, December 2008. Field of view 1.0 mm in width.

The unknown, found to be metamict, required gradual recrystallization by annealing for forty-eight hours at 700˚ C, before a distinct diffraction pattern could be obtained. Accomplished by John Attard, December 2008, the pattern matched gadolinite-(Ce) Type material from the Bjørkehalen pegmatite, near Buer, Norway, reported by Segalstad and Larsen (1978). The crystals were in fact, monoclinic, but with a beta angle of approximately 90.4˚. Thus, with such a minimal deviation from 90˚, many of the minute crystals would easily be assumed, in error, to have orthorhombic symmetry.

EDS SPECTRA

An X-ray diffraction pattern alone could not reliably discern gadolinite-(Ce) from the analogous species gadolinite-(Y). Which species has been found at milepost 165? This question was investigated by EDS analysis from polished thick sections, which are also essential for discerning core and rim composition variations. Absolute quantitative results, however, could not be achieved, since an analysis of beryllium and boron is beyond the reach of this type of study. Beryllium, with the possibility of boron substitution, is a known constituent of gadolinite, representing perhaps 8% of the composition by weight. In addition, Camara and Oberti (2008) have recently reported lithium as a significant component in gadolinite-group minerals. Therefore, only relative quantitative comparisons are presented for the specific elements studied, which exclude boron, beryllium, and lithium. In view of these omissions, no empirical formula has been calculated.

After the initial examination of twenty-two grain samples, fourteen EDS probes were conducted at Cannon Microprobe from polished thick sections, which compared core and rim compositions. Additionally, a vigorous examination of three select polished crystals was conducted at John Attard's laboratory, including certain discernments by wavelength dispersion.

A typical spectrum from the core of a crystal revealed such complexity (Fig. 2), that without interpretive software, relative quantitative comparisons of the elements would not be possible (Fig. 3). Given equal amounts, neodymium, for example, is known to generate higher peaks than cerium.

Dysprosium is masked by the presence of iron and the two can only be discerned by wavelength dispersion. Overlapping peaks are a general concern in analyses of all rare-earth species.

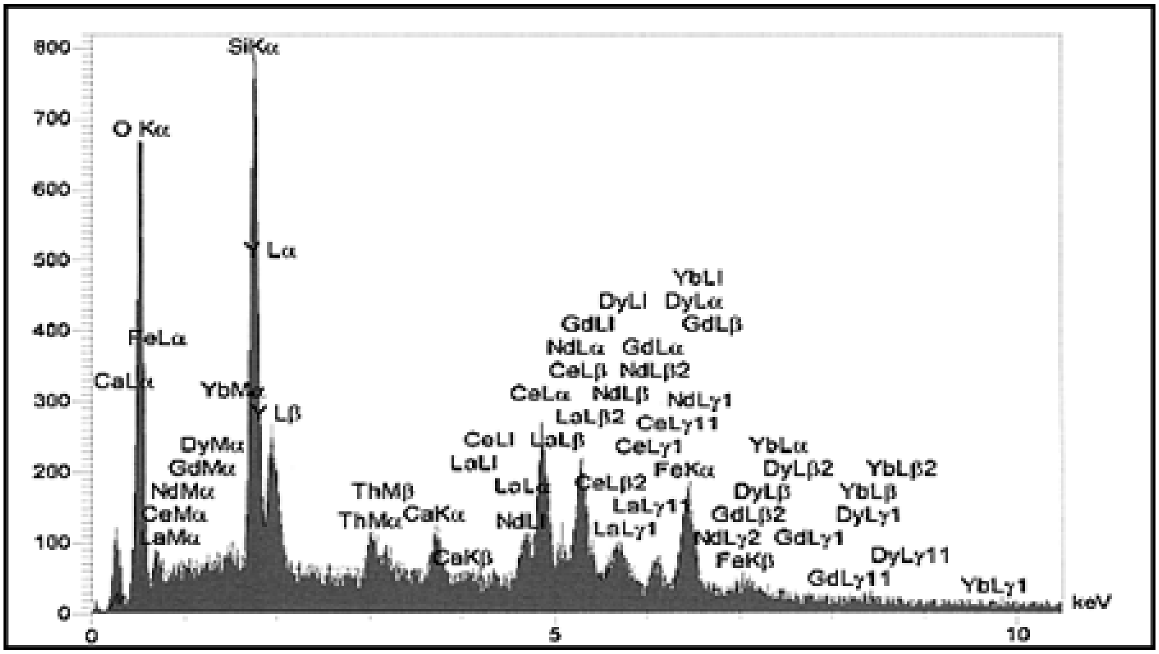
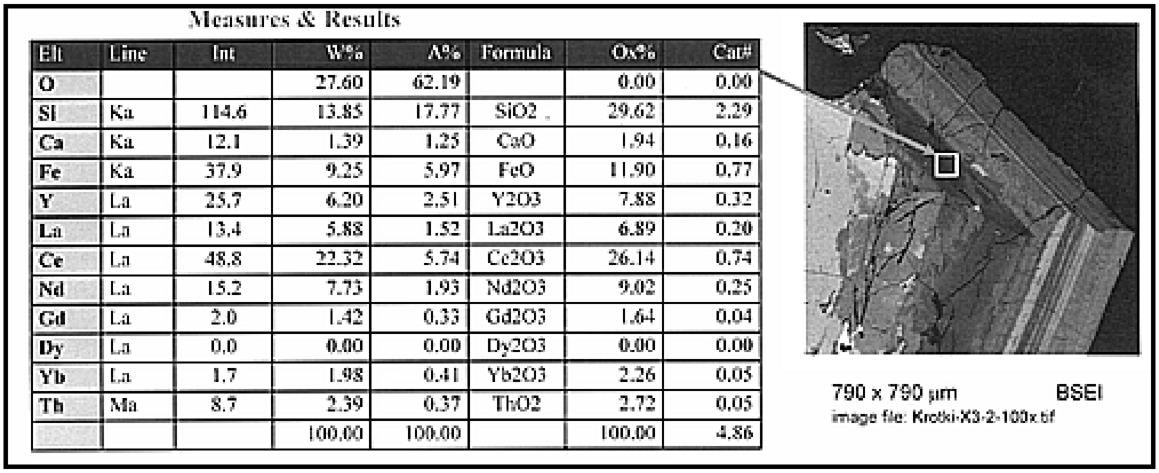


Fig.2. Typical of rare-earth minerals, numerous lanthanide elements jam the spectrum. These substitute for each other in 8-fold coordination along with calcium and thorium.
EDS spectrum, Attard Laboratory, March 2009.

All the crystal zones in the interior of the samples proved to be dominated by gadolinite-(Ce). Cerium dominated yttrium by a factor of as much as 4:1 at the core, and more than 2:1 at the outer oscillatory zones. A sample of one EDS data sheet is shown below (Fig.3).



Measures & Results

Elt	Line	Int	W%	A%	Formula	Ox%	Cat#
O			27.60	62.19		0.00	0.00
Si	Ka	114.6	13.85	17.77	SiO2	29.62	2.29
Ca	Ka	12.1	1.39	1.25	CaO	1.94	0.16
Fe	Ka	37.9	9.25	5.97	FeO	11.90	0.77
Y	La	25.7	6.20	2.51	Y2O3	7.88	0.32
La	La	13.4	5.88	1.52	La2O3	6.89	0.20
Ce	La	48.8	22.32	5.74	Ce2O3	26.14	0.74
Nd	La	15.2	7.73	1.93	Nd2O3	9.02	0.25
Gd	La	2.0	1.42	0.33	Gd2O3	1.64	0.04
Dy	La	0.0	0.00	0.00	Dy2O3	0.00	0.00
Yb	La	1.7	1.98	0.41	Yb2O3	2.26	0.05
Th	Ma	8.7	2.39	0.37	ThO2	2.72	0.05
			100.00	100.00		100.00	4.86

790 x 790 µm BSEI
image file: Krotki-X3-2-100x.tif

Fig.3. Software data indicates cerium as the dominant rare-earth element for the dark outer zone. Location of probe indicated by the white box in the BSE/SEM image on the right.
Attard Laboratory, March 2009.

OSCILLATORY ZONING

Photomicrographs taken by the author reveal broad contrasting bands in the outer perimeters of some polished gadolinite crystal samples. An EDS examination of these rim areas was made to compare with data taken from the core of the crystals. These probes were marked on corresponding SEM backscatter images, which revealed distinct oscillatory zones, represented by micron-level bright and dark bands (Fig. 4). This was a surprise observation. At higher magnification, the bands were observed to be composed of yet finer and finer alternations. An intensive inquiry at the micron level would be required to determine if these oscillations represent any specific shifting in composition in the late magmatic fluids.

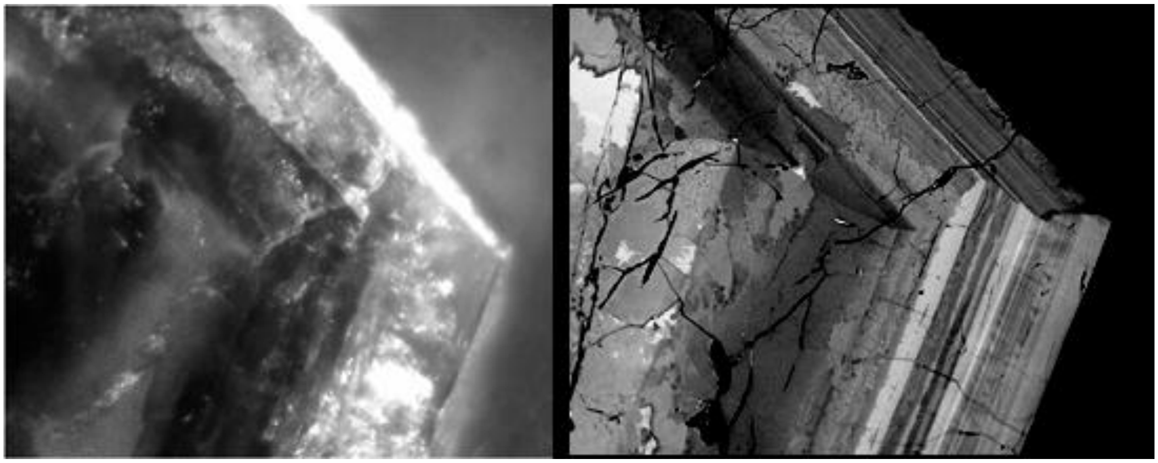


Fig. 4. Oscillatory outer zones in polished gadolinite crystal. Field of view 790 microns in width.
Photomicrograph © Saul Krotki 2009, (left).
SEM/BSE image, Attard Laboratory, San Diego, California, (right).

In their study of color zoning in sphalerite, Oen, Kager, and Kieft (1980a), eloquently expound on the nature of oscillatory zones. From their abstract:

> The oscillatory zoning is explained by a crystallization model depicting cyclic super-saturation at crystal-liquid interfaces due to changes in diffusion and growth-controlled concentration gradients in the boundary layer adjacent to growing crystals during isothermal nonequilibrium crystallization of a supercooled hydrothermal solution. Rapid crystallization eliminates supersaturation at the crystal-liquid interfaces and causes a pause in crystal growth, during which renewed supersaturation of the interface liquid is attained by solute diffusion from the bulk liquid, resulting in a new growth cycle. The model involves true supercooling of bulk liquid as the driving force behind the oscillations and 'constitutional supercooling' of interface liquid as the mechanism by which oscillations are accomplished.

Electron microprobe spacial resolution is on the order of 1×10^{-6} meters. In their study of color zoning in sphalerite, Oen, Kager, and Kieft (1980b) employ three-micron step traverses for detailed analysis, and up to forty-micron steps, for gross analysis. For each step, weight and atomic percentages are calculated. They reflect, "Since the zones are often less than 1.0 micron in width, the true maxima and minima are located in step traverses only by chance. Nevertheless, oscillatory zoning is revealed…by the oscillation of data around an average."

A study of the micron-level alternating bands, however, is not essential for gross comparisons of core and rim. To discern the dominance of gadolinite-(Ce), from the analog species, gadolinite-(Y), and validate this determination, it is instead only necessary to examine and compare many probe volumes from numerous samples. In most of the crystals examined, oscillatory zones were observed only as the last to form rims, representing only a minor proportion of the total sample mass (Fig. 5).

Data from our Washington Pass gadolinite examination included only four probes focused within the dark and light bands, a study far too limited to discern any subtle alternating trends in chemical composition in these zones. These analyses were used only in comparison to data from numerous core crystal spectra.

The sum of all data established cerium as the dominant rare-earth element in the core of the Washington Pass crystals, as well as in both the dark and light outer oscillatory bands. No clear trend in cerium/yttrium proportion between alternating dark and light outer bands has thus far been observed. Only in the outer-most growth-shell is yttrium found to suddenly become dominant (Fig. 6 and 7). Data collected thus far is insufficient to determine if any specific pair of elements alternate concentrations, and so directly result in the oscillatory bands.

Do large-scale shifts in chemical conditions of the progressively cooling fluid field generate the dark and light bands? Do oscillatory relationships exist for any pair of elements? Can something as subtle as a shift, for example, between Fe^{+2} and Fe^{+3}, result in these bands? These are intriguing questions that invite a continued, orderly, and precise analysis of the Washington Pass gadolinite crystals.

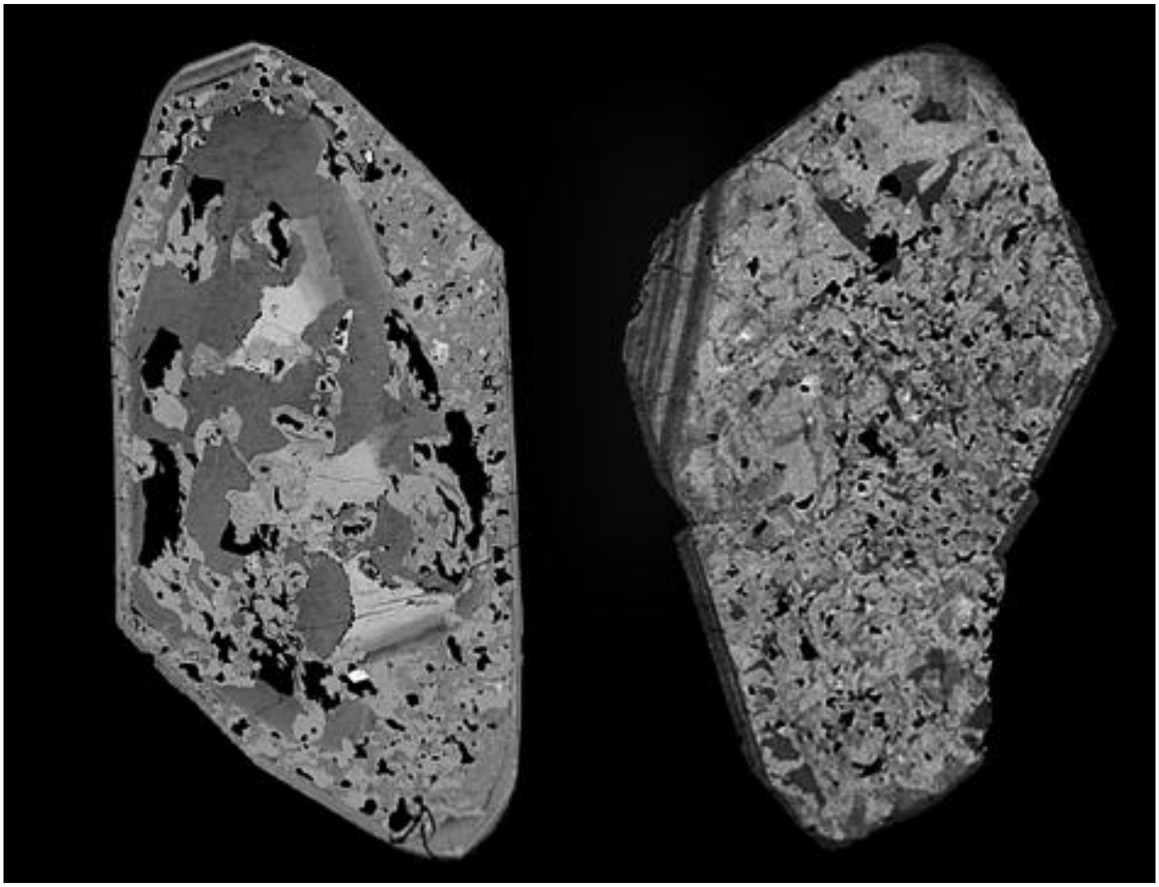


Fig. 5. Complete 1.0 mm gadolinite crystals exhibiting oscillatory zones only in the last growth zones, near the top, of both crystals.
SEM/BSE image from polished thick mount by John Attard, December 2008.

WASHINGTON PASS, MILEPOST 165 GADOLINITE
COMPARISON OF ELEMENTS IN 8-FOLD COORDINATION
(SECTION OF LARGER GRAPH)

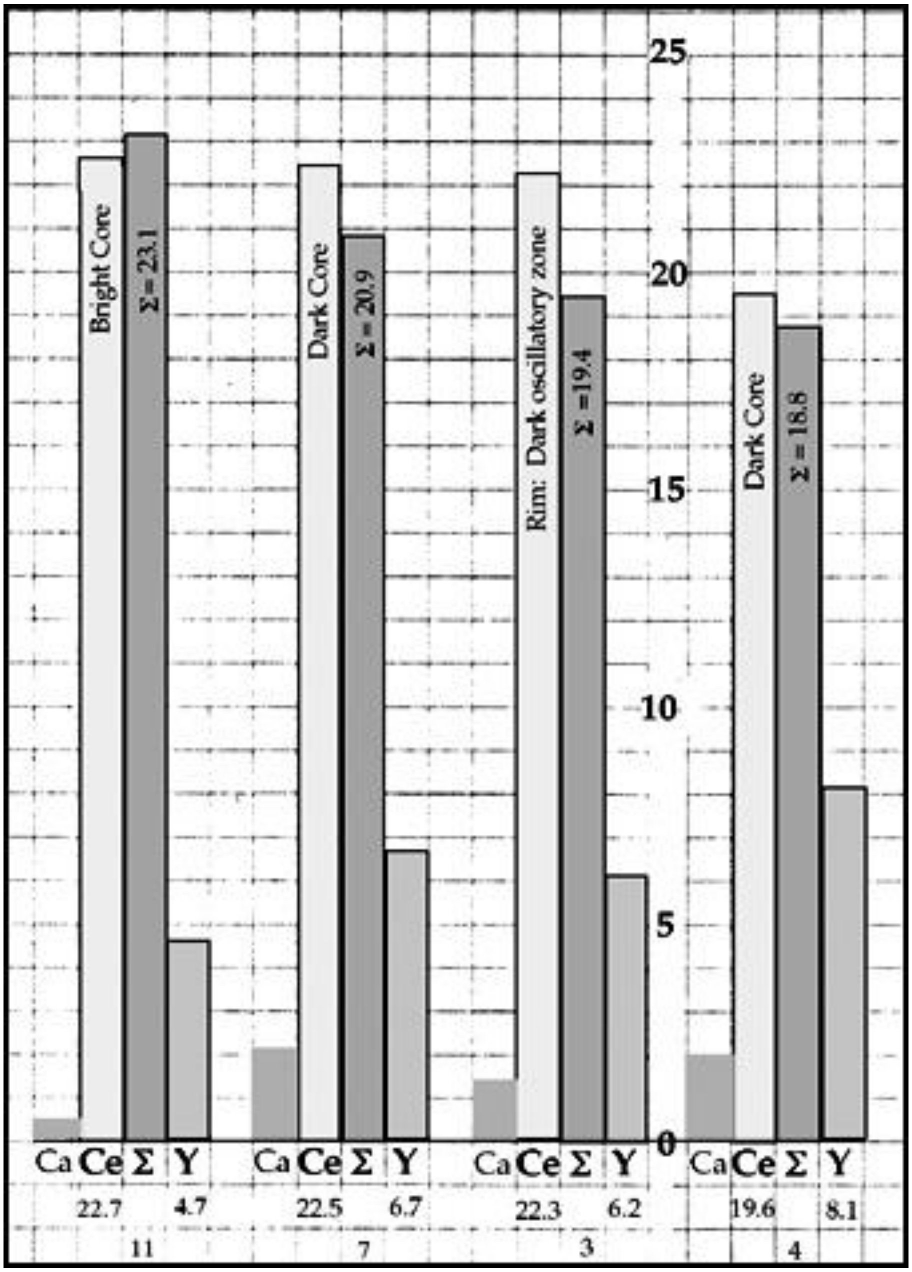


Fig.6. A section of a larger graph showing the relative weight percentages of elements in 8-fold coordination. Here bright and dark core areas are compared. Cerium, second from the left in each set of columns, far dominates all the elements. The lanthanide summation (excluding cerium), the darkest graph area, is almost always dominated by neodymium. Yttrium is shown on the far right. The minor contribution of calcium is shown on the left of each column set.

WASHINGTON PASS, MILEPOST 165 GADOLINITE
COMPARISON OF ELEMENTS IN 8-FOLD COORDINATION
(SECTION OF LARGER GRAPH)

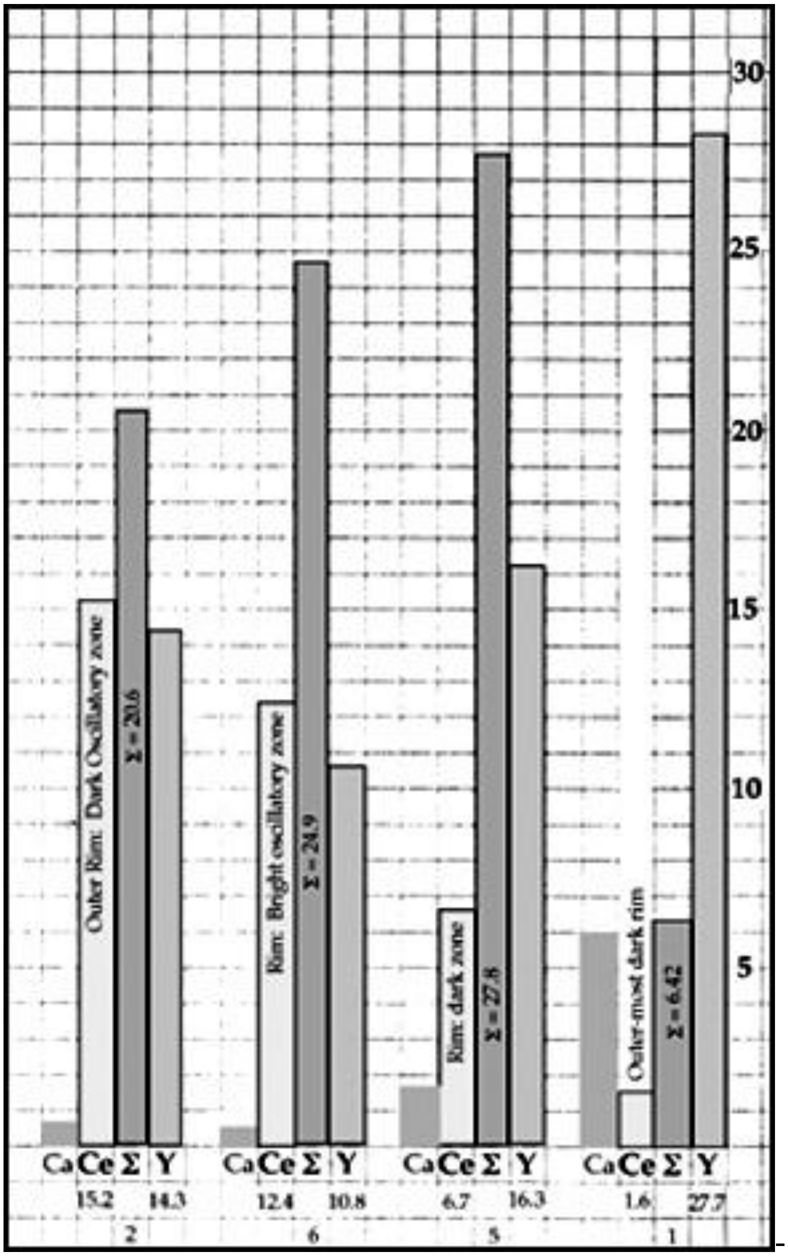


Fig.7. Dark and light areas of the outer-most rims are compared. On the far right, representing the late growth-rim, yttrium suddenly dominates all the rare-earth elements. Calcium content (left of each column set) also increases along with the yttrium dominance in the outer-most rims.

The varied concentration ranges of each element reflect the inhomogeneity of the crystal core. In many cases a given cubic micron of one composition, sits adjacent to a cubic micron of a different chemical composition.

Ito and Hafner (1974a) remind us that "natural gadolinites always contain the entire series of lanthanides, different divalent ions, and often Zr^{4+} and Th^{4+}." It will be interesting to see in continued spectral studies how many of the lanthanides make their appearance.

INCLUSIONS AND METAMICTIZATION

Bright, high-mass inclusions were probed, which appear to be cerium carbonates, probably bastnäsite-(Ce) (Fig.8). Other inclusions were determined to be thorium-rich monazite. Uranium was also identified in one probe.

WEIGHT % RANGE FOR ALL ELEMENTS DETECTED IN TWELVE CRYSTAL-CORE EDS PROBE SAMPLES

ELEMENT	WT% RANGE
Oxygen	**18.83-28.48**
Fluorine	**0.00-5.25**
Silicon	**4.51-14.58**
Calcium	**0.42-3.18**
Iron	**2.53-13.00**
Yttrium	**5.36-16.31**
Lanthanum	**0.00-9.49**
Cerium	**6.74-30.44**
Neodymium	**8.03-14.47**
Gadolinium	**0.00-8.41**
Dysprosium	**0.00-7.26**
Ytterbium	**0.00-5.29**
Thorium	**0.00-4.64**

It is generally understood that the change from the crystalline to the amorphous metamict state is brought about by alpha decay of radioactive elements. It is in the high-mass inclusions that we see the root cause of metamictization in these gadolinite-(Ce) crystals. Mitchell (1973a) presents a complete review of the effect of alpha emission on displacing lattice atoms.

The existence of both carbonate and phosphate inclusions offers clues as to the chemical nature of late-stage fluid transport of rare earth elements. Gieré, (1996a), points out that bastnäsite can replace monazite if the late phosphate solutions are present. In fact, the pH-dependent reaction is interchangeable. Gieré presents the reversible equation:

$$\mathrm{REE\ (CO_3)\ F + H_3PO_4{}^{\circ}{}_{(aq)} \leftrightarrows REE\ PO_4 + HF^{\circ}{}_{(aq)} + H_2O + CO_{2\ (aq)}}$$

COMPARISON TO TYPE MATERIAL FROM NORWAY

All zone studies verify that the crystals are, indeed, gadolinite-(Ce). In the Type material from the Bjørkehalen pegmatite, Norway, described by Segalstad and Larsen (1978), cerium increases towards the outer zones and is highest in yttrium in the core. The opposite trend takes place in the Washington Pass samples where cerium is found dominant in the cores and outer zones, while yttrium dominates only in the late-stage overgrowths. What, in principle, does this suggest about the transport of elements in late stage magmatic fluids? Were cerium and yttrium supplied in equal amounts, yttrium would be expected to enter the gadolinite structure preferentially, because of its smaller ionic radii. Since this is not the case in these Washington Pass samples, we must assume that yttrium was simply not available in the early stages of crystallization. Did the gadolinite-(Ce) crystallize in a yttrium-poor environment? Is yttrium taken up by another species? What was the source of the sudden increase in concentration of yttrium in the last stage of crystallization? Perhaps spectral examination of the associated minerals (fluorite, zircon, and mica) will reveal the capture of rare earth elements, including yttrium, at different stages in crystallization. For a comprehensive discussion of the fluid transport of rare earth elements see Gieré (1996b), "Formation of rare earth minerals in hydrothermal systems."

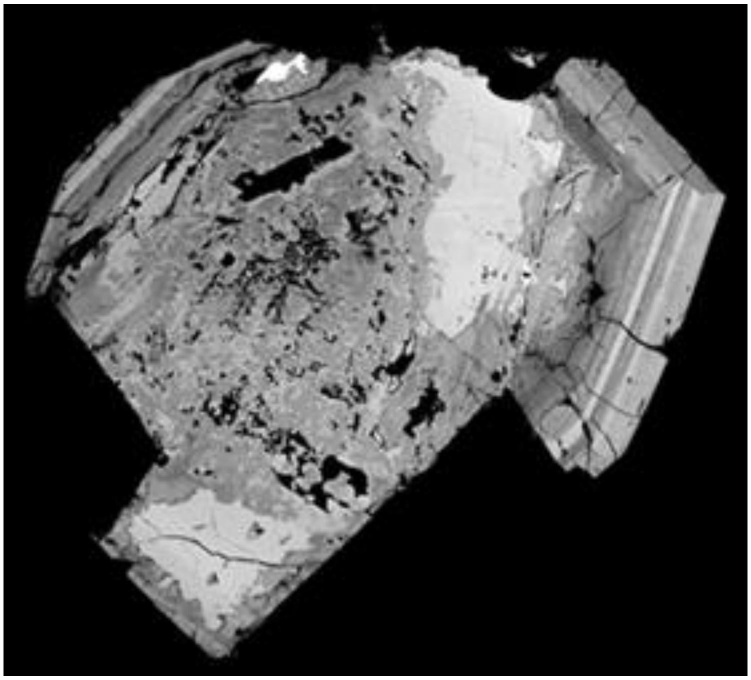


Fig. 8. SEM/BSE cross-section of a 1.2 mm gadolinite crystal accomplished in sections by John Attard, and later assembled as a seamless mosaic by the author, using Adobe Photoshop software. Note the oscillatory, late-stage growth on the right. The large white masses in the crystal interior are relics of the original gadolinite-(Ce) crystal. Black areas are inclusions of hematite. At the top-center left, the brightest inclusion appears to be Bastnäsite-(Ce), which is also dispersed throughout the crystal interior. Yttrium is dominant only in the very outer-most edges.

GADOLINITE VARIATIONS AT WASHINGTON PASS

BART CANNON'S EARLY EXPLORATION AND DISCOVERY OF GADOLINITE IN THE GOLDEN HORN BATHOLITH

Bart Cannon began his field investigation of the Golden Horn Batholith in late 1960's as a field assistant to Robert John Stull. Stull was primarily concerned with geology, bulk rock composition, and geochronology, but not at all with rare mineral species. Nevertheless, Stull's mapping of the relative positions of the various granite types provides a valuable guide to mineral specimen exploration today.

Bart penetrated deep within the batholith examining all the various granite phases. He explored the Willow Creek area of Kangaroo ridge to above 7000 feet, very difficult to traverse zones at Cutthroat Ridge, as well as the more popular roadside areas. By 1976, he had preserved drawers full of rare species including various unknowns. Bart's find of the original zektzerite specimens is well known. See Dunn, Rouse, Cannon, Nelen (1977).

In 1976, Bart consulted with Pete Dunn of the Department of Mineral Sciences, Smithsonian Institution, Washington, D.C., who investigated several of the unknowns. A yellow-green micro-crystal proved to be gadolinite. A specimen in Bart's collection, examined by the author, is a 2.0 mm radial spray of nearly acicular, pale-green, translucent, prismatic crystals from the Liberty Bell talus collecting area.

The crystals are difficult to spot. The radial spray, incidentally, could easily be confused with kainosite, also listed as a Washington Pass species in Pete Dunn's study.

Translucency suggests that these crystals are perhaps not metamict. Mitchell (1973b) points out that metamict species are generally dark brown in color, while non-metamict "dimorphs" of the same species are often more colorful, translucent, and with a higher index of refraction. It would be interesting to demonstrate this as more samples become available. A truly non-metamict crystal would yield an X-ray diffraction pattern without requiring annealing.

The term "noble" has been used to describe gemmy, translucent gadolinite crystals. Bayliss (2000), in *The Glossary of Obsolete Mineral Names*, also notes that the term had been applied to beryl, garnet, and even hornblende among other species. "Noble" signifies only that the crystals are gemmy, but is not a proper species or variety name. Gramaccioli (1977) shows a photograph of an outstanding 2.0 mm transparent blue-green crystal from Böckstein Quarry, near Bad Gastein, Austria that he refers to as "of the 'noble' variety." Are Washington Pass specimens of such limpid quality yet to be discovered?

RUSSELL BOGGS' GADOLINITE REPORT

Russell Boggs (1984d) identified the Washington Pass species as intermediate between gadolinite and gadolinite-(Ce). If you refer to the Fleischer Glossary (1975), you will see the species name, "gadolinite," in use at that time, with no dominant rare-earth element suffix. Refinement to the species name, gadolinite-(Y), was introduced by Miyawaki and Nagashima (1984). Boggs, then, is suggesting that the Washington Pass gadolinites are intermediate between gadolinite-(Y) and gadolinite-(Ce). This may well be the case for some crystals, but perhaps not for all the crystals from the various Washington Pass collecting areas. More crystals need to be examined to validate these discernments.

Boggs (1984d) describes gadolinite from the border granite as pale yellowish green in hue. Among the associated minerals, he lists fibrous alkali amphibole. Collectors will recognize this association as belonging to the Liberty Bell collecting area, where the slightly less peralkaline border phase granite is found.

The milepost 165 crystals, examined in this study, are gray-brown in hue. A scarce few yellow-green, as well as off-white crystals, have also been found. Are these crystals each of a different chemical composition? Examination by microprobe of all the apparent crystal variations would add significantly to this study. Further inquiry will depend, however, on an intensive search for more specimens. This report strongly suggests that gadolinite-(Ce) occurs at milepost 165, as a distinct Washington Pass species. Does a gadolinite-(Y) also occur at milepost 165, other than as a late stage overgrowth?

In his crystal drawings of the Washington Pass gadolinites, Boggs presents three forms: a {100}, s {011}, and m {110} (Fig. 9).

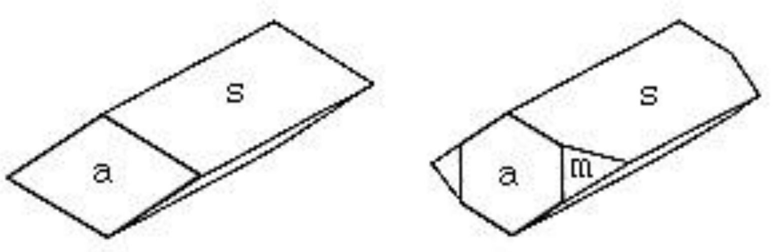


Fig. 9. Washington Pass Gadolinite Crystals after Boggs

RANDY BECKER'S GADOLINITE REPORT

Randy Becker (1985) reported a border phase occurrence of gadolinite-(Y): "In the crocidolite (fibrous riebeckite) pocket, gadolinite commonly occurs as dark, blackish-green to light green micro crystals, diamond shaped in cross section. Some of the crystals found have been transparent." It would be illuminating to see good photomicrographs of these specimens.

DONALD HOWARD'S GADOLINITE REPORTS

Donald Howard (Spring 1990) reports: "Another unusual occurrence in the border granite is gadolinite. At most occurrences, this mineral is black and completely embedded in the quartz and feldspar matrix. At Washington Pass it occurs as pale green, simple prisms, in miarolitic cavities. Again, although a monoclinic mineral, the oblique angle is so close to 90˚ that the prisms appear to be orthorhombic." (Fig.10, left).

In a follow-up report, Donald Howard (Fall 1990) adds, "Gadolinite of quite a different habit and color is found in the same border granite phase, closely associated with clinochlore. These crystals are white in color and possess a pearly luster." (Fig. 10, right). "Crystals of this type often form in radiating clusters. They are very hard to see against a back-ground of white minerals, such as microcline and quartz."

What differences might EDS zone studies reveal between the yellow-green crystals and the white crystals? This is an enticing question and one that we hope to answer, in detail, as more material becomes available.

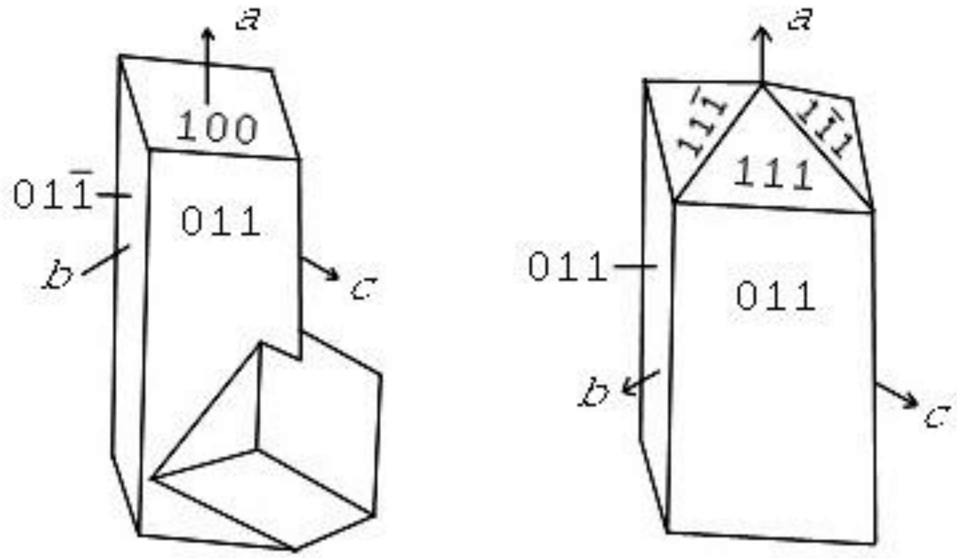


Fig. 10. Pale green gadolinite crystal morphology reported by Donald Howard, Spring 1990 (left).
Pyramidal gadolinite form observed in white crystals from Howard's Fall 1990 report (right).

CONSIDERING CALCIOGADOLINITE

Calciogadolinite is one theoretical end-member proposed by Ito and Hafner (1974b) in their study of synthetic gadolinites. This composition, however, is not to be expected in natural gadolinites. No specimen has been found with high enough calcium in any zone to suggest calciogadolinite. Here we find relatively low calcium for much of the internal mass of the crystals. Only where yttrium dominates at the late stage overgrowths, or crusts, is the calcium concentration elevated. At best, we can refer to certain zones as calcium-rich gadolinite, but this has not been clearly determined in this study. The calcium content needs to be evaluated in all the Washington Pass gadolinites of various colors. Calciogadolinite is not, at this time, an accepted mineral species name.

IDEAL CHEMICAL FORMULAS FOR GADOLINITE-(Ce)

General, $A_2BC_2Si_2O_{10}$
Where A = REE, Ca, Th, K in 8-fold coordination.
B = Fe^{2+}, Mg, Mn in 6-fold coordination.
C = Be, B, Al in 4-fold coordination.
(REE is here used to mean the 15 elements La through Lu and yttrium.)
Segalstad and Larsen (1978).

$REE_2Fe^{2+}Be_2Si_20_{10}$
Segalstad and Larsen (1978).

$(Ce_,La,Nd,Y)_2Fe^{2+}Be_2Si_20_{10}$
Fleischer and Mandarino (1980-2008), Glossary of Mineral Species.

$Ce_2Fe^{2+}Be_2O_2(SiO_4)_2$
Nickel and Nichols (2008), IMA/CNMNC, List of Mineral Names.

$Be_2Fe^{2+}Ce_2\,Si_20_{10}$
RRUFF Database (2009).

PHOTOMICROGRAPHS AND CRYSTAL MORPHOLOGY

Thirty specimens have been preserved for photomicrographs and crystal morphology studies. The crystals range in size from less than 1.0 mm to just over 2.0 mm. These crystals are more complex than those previously reported by Boggs (1984), and Howard (1990).

Photomicrographs were created by the author, using a Nikon Coolpix 4500 digital camera, mounted on an Optem 12.5, video zoom tube. This system successfully images down to a 0.5 mm field of view. Image-plane blending, color, and contrast adjustments, were accomplished in Adobe Photoshop, version 3.0, Knoll and others (1989-1995).

Distortions and partial emergence from the matrix make orienting the minute crystals perplexing. Because of the low *beta* angle, a given crystal may give the impression that it can be oriented horizontally or vertically. Only one choice, however, for the placement of the unique monoclinic mirror plane of symmetry is absolutely correct.

Most crystals in this study are oriented with an elongated *b* axis. Some are elongated on the *c* axis. Crystallographic forms are named for those crystals illustrated, but are not absolutely certain. The placement of the mirror plane of symmetry cannot be arbitrary. In the absence of a reasonable way to conduct optical morphological examination, choices had to be made based on what could be learned from comparisons of many crystals.

SHAPE 71 software, Dowty (2002), provides an idealized 3D model for a gadolinite crystal, which can be turned in virtual 3D and modified to match specific crystals as they are examined. The software is capable of enlarging or reducing any given face. Forms can be added or deleted as necessary to model a specific crystal being studied. Most helpful, the unique mirror plane of symmetry is coordinated with miller indices for the individual crystal forms.

Gieré (2008), in his study of metamict fergusonite-(Y), reminds us that the crystalline-to-metamict transition is associated with macroscopic swelling. For certain of the Washington Pass gadolinite crystals, the swelling certainly results in curved surfaces and raised, meandering, crystal face edges. This distortion makes establishing a unique plane of symmetry impossible (Fig. 12). Gieré (1996c) describes the effects of metamictization in a study of a polished zirconalite crystal, "the principal effects of the crystalline to aperiodic transformation are volume expansion and microfracturing, the fractures visible in (the zirconalite crystal) are probably a direct consequence of the radioactive decay of Th and U."

Photomicrographs of a wide range of gadolinite morphological types, preserved in this study, are shown below (Fig. 13, 14, 16). Illustrations of select crystals, oriented with symmetry operators and miller indices are included (Fig. 15, 17, 18).

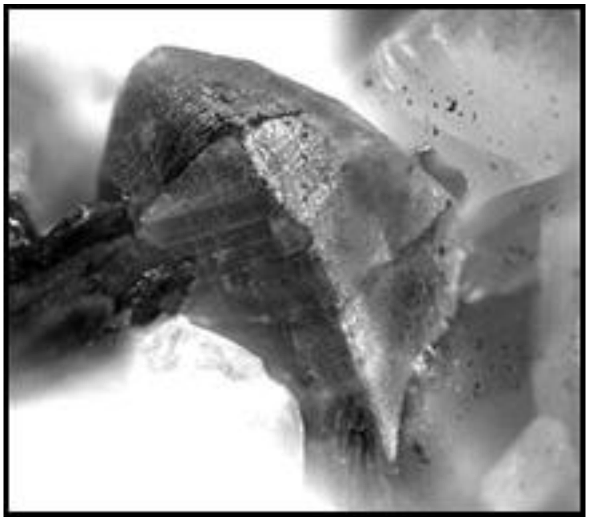


Fig.12. A gadolinite-(Ce) crystal from milepost 165 showing external deformation resulting from internal metamictization. Photomicrograph © Saul Krotki 2008. Field of view: 2.0 mm.

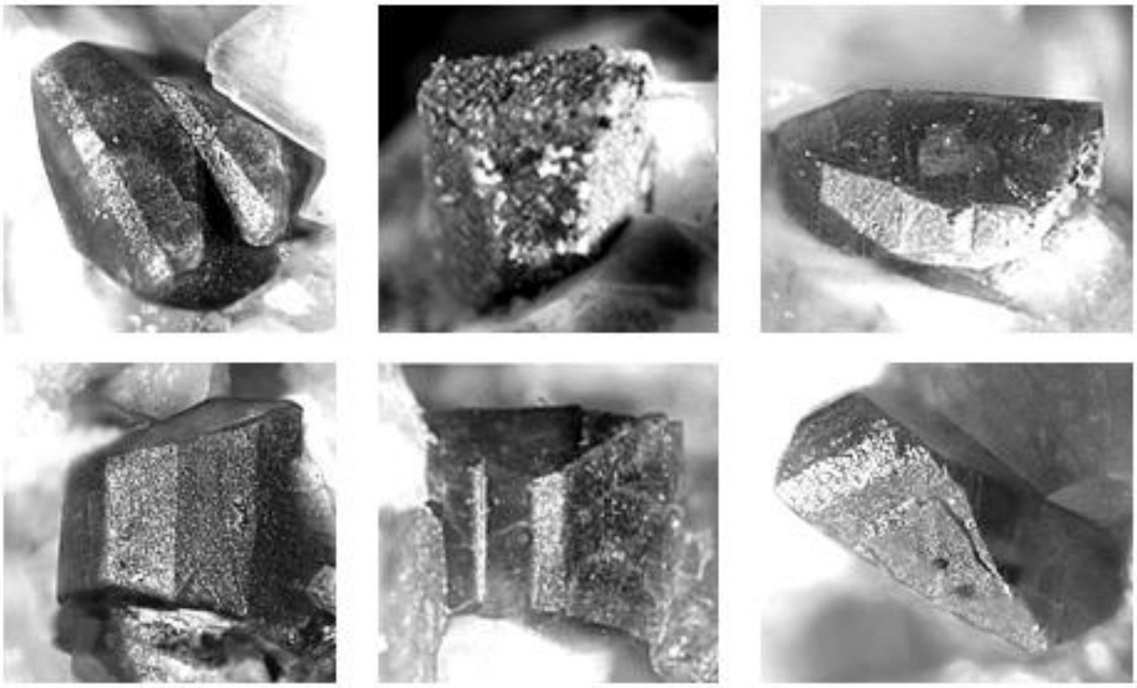


Fig 13. Gadolinite-(Ce) crystals collected by the author at WA Pass, milepost 165, exhibit a wide range of morphological variation. Note the orthorhombic appearance of the top center crystal. The crystals are all approximately 1.0 mm in their longest dimension.
Photomicrographs © Saul Krotki, 2008-2009**.**

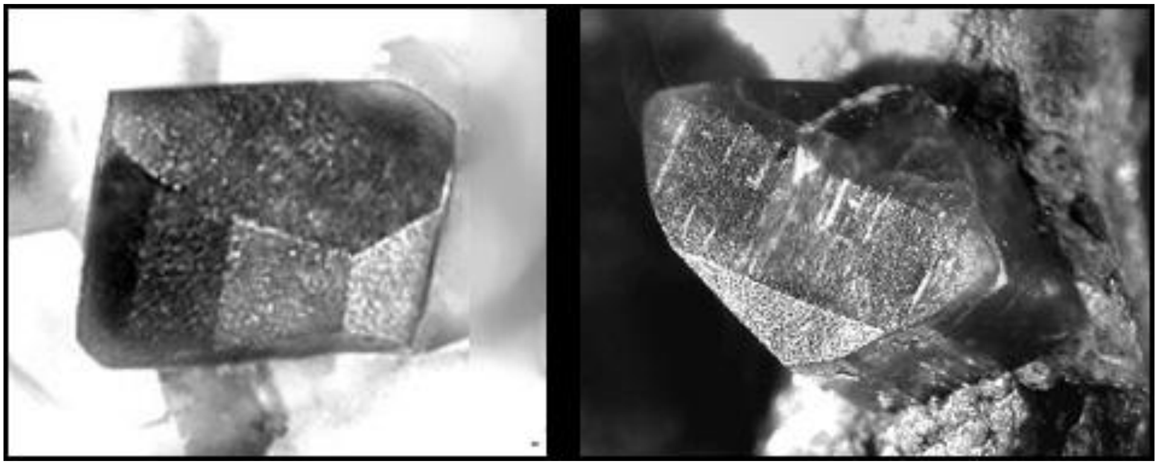


Fig. 14. WA Pass gadolinite crystals oriented with elongated *b* axis. Crystals are 1.0 mm.
Photomicrograph © Saul Krotki 2009.

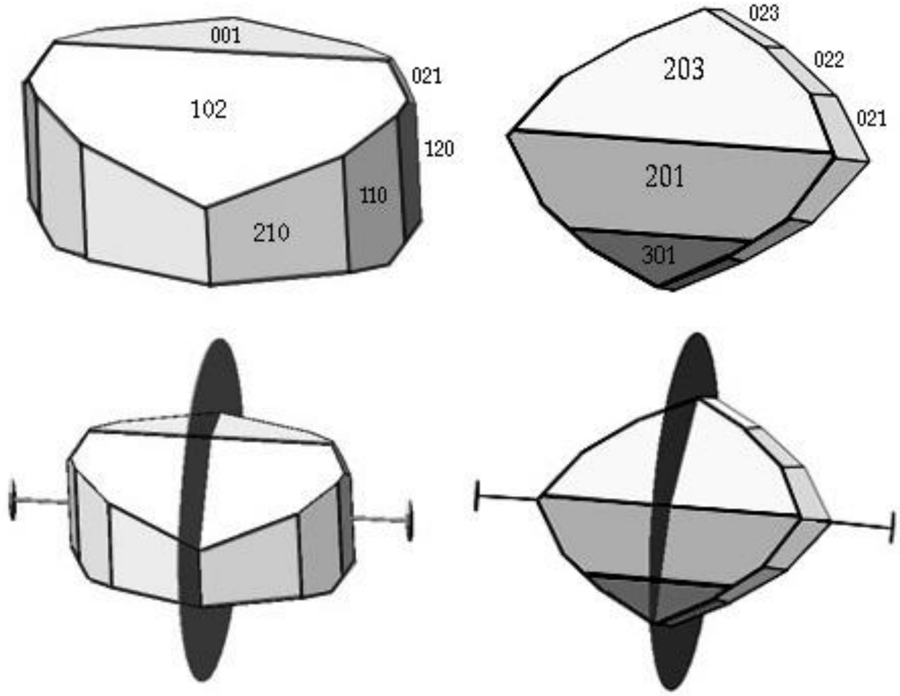


Fig. 15. Crystallographic diagrams, created by the author, in SHAPE software, indicating mirror plane of symmetry and choice of crystal faces which approximate the forms of the crystals shown above in Fig. 14. Relative axial lengths: a: .06261; b: 1.32; c: 1. *Beta* angle assigned as 90.6˚. Other crystals tend to be elongated vertically on the *c* axis (Fig. 16, 17).

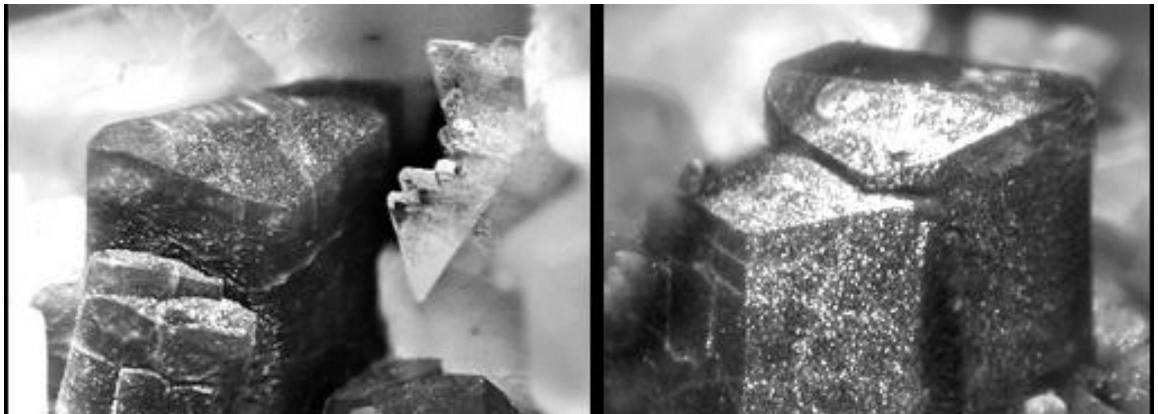


Fig. 16. Parallel crystals of gadolinite collected by the author at WA Pass milepost 165 oriented with the *c* axis elongated. Photomicrograph © Saul Krotki 2008. The crystals are approximately 1.0 mm in the longest dimension. Note the late stage serrated albite crystal (upper right) and the well-formed quartz crystal, so typical of many of the cavities at milepost 165 (Fig.16).

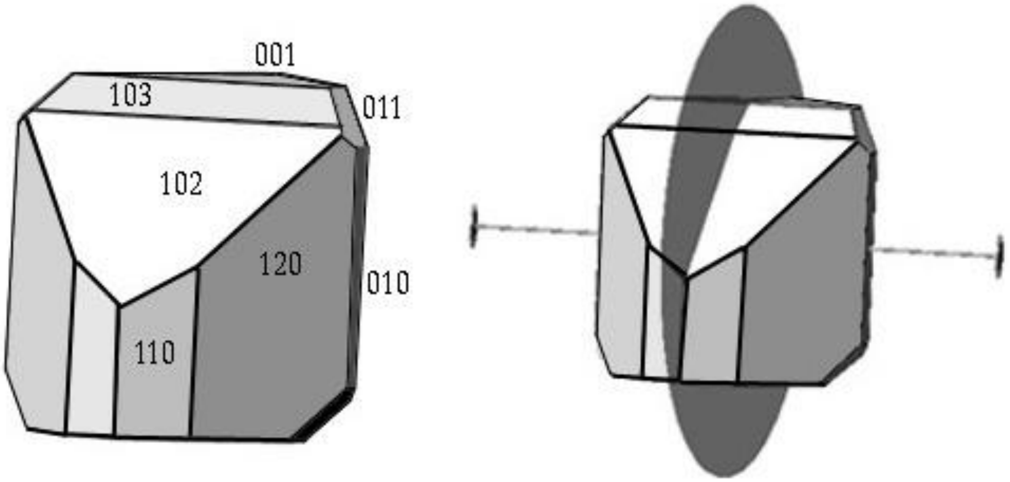


Fig. 17. Idealized crystallographic drawings, rendered by the author, with SHAPE 71 software, indicating the position of the mirror plane of symmetry, the axis of rotational symmetry, and the miller indices for the forms suggested by the crystals in Fig. 16 above. Here the c axis is elongated instead of the b axis.
a: .06261; b: 1.00; c: 1.32 Beta angle assigned as 90.6˚.

Two other forms of gadolinite-(Ce) have been observed at milepost 165 (Fig. 18).

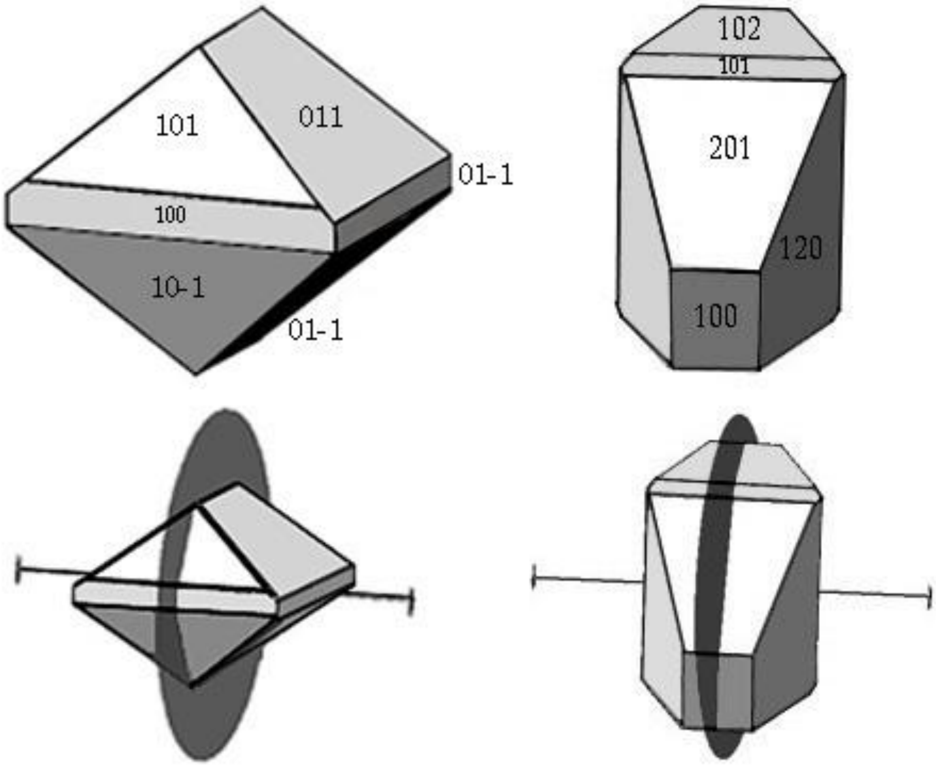


Fig. 18. Morphology idealized from several specimens of similar form. Modeled with Shape 71 software. The illusions of certain views are difficult to fully comprehend. It is necessary to turn the virtual crystal through various views in the 3D software to fully comprehend the illusion of any one orientation.

Summary of all crystal forms encounter in the WA Pass Gadolinite crystal is as follows:

{001}, {010}, {011}, {01-1}, {021}, {022}, {023}
{100}, {101}, {10-1}, {103), {111}, {102}, {120}
{210}, {201}, {203}, {301}

ADDITIONAL SPECIES FOUND ASSOCIATED WITH GADOLINITE-(CE):

Allanite: Shiny black crystals up to 1.5 mm, in primitive crystal forms. Rare.

Albite: Clean, sharp, serrated, overgrowths on microcline (Fig. 16).

Bastnäsite-(Ce): Occurring in pale pink clusters of minute platelets to 4.0 mm. Observed associated with fluorite. Rare.

Fluorite: Isolated, nearly colorless, octahedral crystals, with cube modifications. The crystals are generally less than 2.0 mm.

Genthelvite: Isolated bright pink tetrahedrons to 1.2mm. EDS verified, Cannon Microprobe, November 2008. Perhaps the rarest of all the milepost 165 species.

Mica Group minerals: Unusually fresh mica crystals to 4.5 mm, which exhibit delicate growth hillocks. They vary from water-clear, or white, to green, but also occur as dark micas.

The light micas resemble the lithium-bearing polylithionite from milepost 166. They do not show the annite-chamosite intergrowths typical of those found at milepost 164, although some surface alteration does occur. Lithium has not been determined for these micas.

Microcline: Well-developed white crystals up to 5.0 mm, most often with late stage albite overgrowths.

Opal var. hyalite: Late-stage overgrowths on quartz, often as beautiful botryoidal formations. The hyalite fluoresces under short-wave ultraviolet light in a hue similar to fluorescent zektzerite, but slightly greener.

Quartz: Terminated clear quartz crystals up to 1", frequently containing curious inclusions.

Siderite: Shiny crystals in clusters up to 10.0 mm. Siderites found in the cavities at milepost 166 are almost always altered to goethite, whereas the siderite crystals at milepost 165 show little alteration.

Zircon: Occurs as oddly flattened crystals, frequently side by side with easily overlooked gadolinite. Because of the odd morphology, EDS spectra was required, at times, to discern the zircon species. Zircon also occurs in odd clusters that appear to be polycrystals in which four parallel, prismatic elements make up one tetragonal form. Zircon is also found as scepter crystals in radial sprays. The clusters observed of these types have all been less than 1.5 mm in diameter.

SUMMARY

X-ray diffraction, coupled with EDS analyses, appears to have validated the occurrence of gadolinite-(Ce) as a Washington Pass species. Only a first impression of the composition of various internal zones, inclusions, and late stage incrustations has been achieved. We will want to subject any conclusions suggested in this study to scrutiny involving analysis of a reasonable number of new samples.

A full investigation, for example, would logically seek to determine the composition of crystals of different color or morphology, or of similar appearance, but from different alkali granite.

It is always reasonable to suggest that for a given species, more extensive investigation is warranted. Unique, however, in this continued study is the possibility of verifying that both gadolinite-(Ce), and gadolinite-(Y) occur at Washington Pass. This preliminary report suggests that Washington Pass may be the only known locality where both analog species of gadolinite occur.

Newly acquired specimens and support research will be presented by the author at a meeting of the Northwest Micro Mineral Study Group in November of 2009.

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What? Another type of twin in Pseudobrookite?

Donald Howard

After all the exciting developments that have happened recently, I pretty much keep my eyes open for new locations for pseudobrookite. So at the NCMA meeting in El Dorado, California this June, I picked up a couple of pieces from a location I had never heard of before. It was labeled:

Along Highway 78, Big Lue Mountains, Greenlee Co., Arizona

The minerals listed were: tridymite, hematite, titanite, hollandite, pseudobrookite, phlogopite, other stuff.

The hematite looks rather corroded, the hollandite is black and fuzzy, and there are numerous small black blades of pseudobrookite. There is tridymite lining every cavity. The other minerals I did not find in my pieces.

The little black blades of pseudobrookite show striation on the a-face, which are common for the species. When I got to looking closely at one of the blades (shown below), I became confused because it looked at first as if it had striations perpendicular to the c-direction instead of parallel to it. And then I realized that it was a twin crystal. Though crystals of pseudobrookite are normally elongated in the c direction, the cell dimensions are really much shorter along that axis, in fact, only about 1/3 of the dimension along the other two axes. As a result, twinning on {031} will yield two related crystals with a common a-face that are 89.3^{o} apart. Since they are so close to 90^{o}, it is difficult to tell that you are not looking at a single orthorhombic crystal. The picture below was taken with light reflected off the a-face so as to show the two directions of striations that mark the two members of the twin.

So that rather completes the set. We now have twins on planes containing all three of the symmetry axes. Pseudobrookite seems to be able to twin in an amazing number of ways. It will be very interesting if someone figures out how it can possible do all that.

Pseudobrookite on tridymite
Twin on {031}
Size of crystal is about 1mm

Big Lue Mountains,
Greenlee Co., Arizona

Priceite, Oregon's Own Borate

Donald Howard

It was quite by accident that I ran across the reference in Fleischer while looking up something else on the same page. At first I only noticed that the type locality list was in Oregon. A borate in Oregon, I thought, it must be somewhere out on the eastern side in the desert. And then I read the whole line: "8 km north of Chetko, Curry County, Oregon"[1]. Wait a minute, Curry County is on the coast! What a strange place to find a borate.

The 7th edition of Dana[2] gives more information: "Originally found on the seacoast five miles north of Chetco, Curry County, Oregon, both compact and as roundish masses up to several hundred pounds in weight with aragonite in what appears to be a hot-spring deposit." The mineral was first described by Benjamin Silliman Jr. in the 1870s.[3]

So where is Chetco? No such town exists, and as far as I can tell never has. The reference appears to be to the Chetco River, which empties into the sea at Brookings. So the logical place to start was with the Chamber of Commerce in Brookings, which quickly referred me to the Chetco Valley Historical Society. This is a small museum in Brookings that is open to the public Saturday and Sunday afternoons. Unfortunately, I was there on a Tuesday. Fortunately, the caretaker was just pulling in as I was. I read him the above reference, and fortunately he recognized the place that was being described, and ushered us inside to see a photo on display of the place, called "Lone Ranch" (see below).

Aerial view of Lone Ranch in 1948. The light spots on the hill to left of Lone Ranch house are the only visible remains of the borax mine workings.

Photo courtesy of the Chetco Valley Historical Society.

Priceite is a calcium borate, $Ca_4B_{10}O_{19}\cdot 7H_2O$. In addition to the Lone Ranch occurrence, it has been found at Furnace Creek in Death Valley, California; New Brunswick, Canada; Inder, Kazakstan and at Sultan Tschair, Brussa Province, Anatolia, Turkey. At all the locations, it is as a white, fine-textured mass that resembles porcelain.

The material was first noted in the early 1860s by the owner of the ranch at that time. The nodules of the material looked and felt like chalk, and was used as such by the local carpenters and coopers. At one point, the owner tried to dig a 75 foot shaft for more of the “ore”, but gave up when he encountered considerable water. He eventually sold the property in the early 1890s to Pacific Coast Mines.

Some priceite was shipped as ore of borax, but operations ceased by 1893 because of difficulties in processing. Pacific Coast Mines later became U. S. Borax, the company that operates the huge mine at Boron in Southern California, and more recently has taken the name Rio Tinto Minerals. Most of the property is still under their ownership. In the 1950s, the company donated the land lying west of highway 101 to the Oregon State Parks Department to become a part of Samuel H. Boardman State Park. The site is now called Lone Ranch Beach, and is easily reached from highway 101 just north of Brookings.

As can be seen in the accompanying photographs, the appearance of the hillside has changed very little in the last 61 years. The bare rock exposures are still there devoid of grass and vegetation, though the willows along the creek have grown to a considerable size. One building (not visible in the pictures) still remains hidden deep in the willows, surrounded by a sturdy fence. A parking lot, lawns, and picnic facilities have been added, as have paths and walkways and a stone marker.

The rock that composes those exposures is bluish in color and has numerous seams, some of which have the smooth greenish greasy gloss that characterizes serpentine. I could find no evidence of any nodules of the type described as being the priceite. The white seams lacing through the bluish rock analyze out to be composed mostly of magnesium and silicon with some carbonate present, and are probably a mixture of brucite, magnesite and some form of silica. They contain a little iron and aluminum, but no detectable calcium. Since boron is not detectable in XRF, the lack of any calcium is the best indication that none of it is priceite.

The site is a lovely place for a day at the beach, and interesting as a piece of history, but not a very good bet to find priceite. Moreover, if found, the mineral is really pretty ugly, though it would be interesting to see what the “type material” really looked like.

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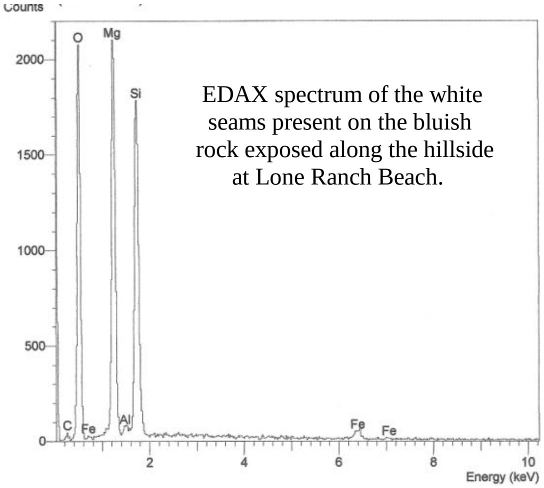


EDAX spectrum of the white seams present on the bluish rock exposed along the hillside at Lone Ranch Beach.

Lone Ranch as it looks in July, 2009.

Above, the north side of the creek, showing the rock exposure above the willow trees that choke the creek.

Right, the rock exposed on the south side of the creek.

Below, the view looking south from the grassy slope beside the north exposure, showing the car park, the path and lawn area, and the beach and rocks that are now part of Samuel H. Boardman State Park.

Zircon twins from Liberty Bell, Washington Pass, Washington

(Each view is approximately 3 millimeter side to side.)

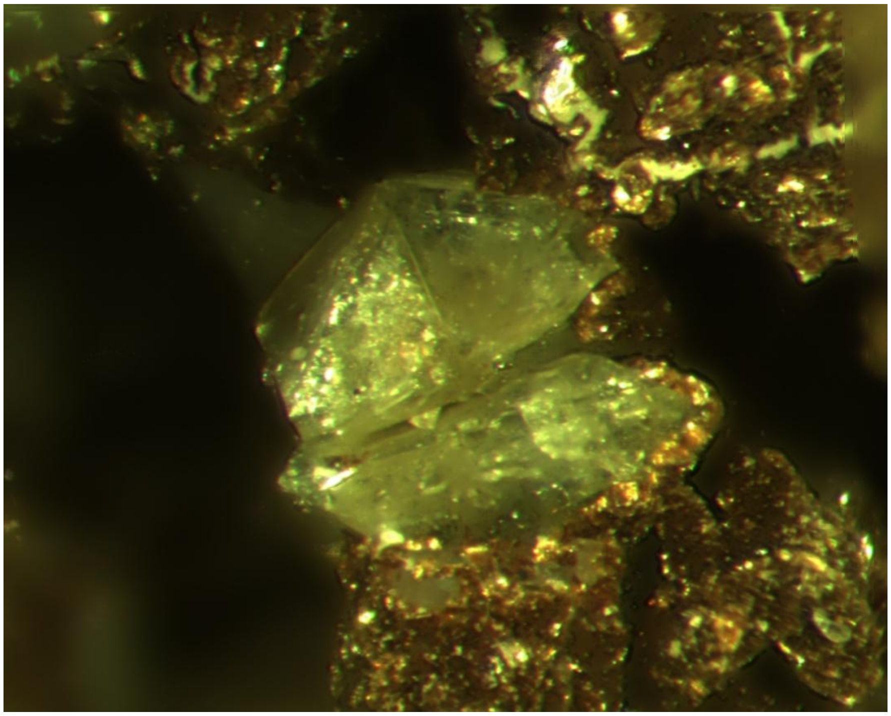
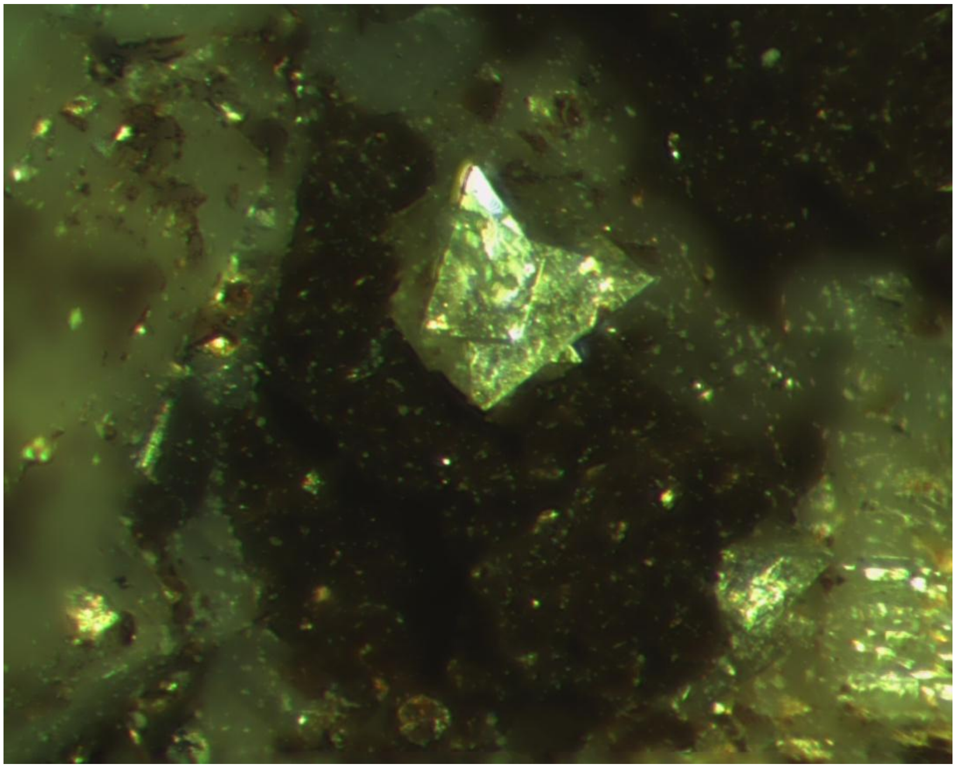


Plate 1: Twin on {101} (upper crystal)

Plate 2: Twin on {111}, taken with reflected light to show the inter-penetration.



Twinning in Zircon, Washington Pass, Okanogan Co., Washington

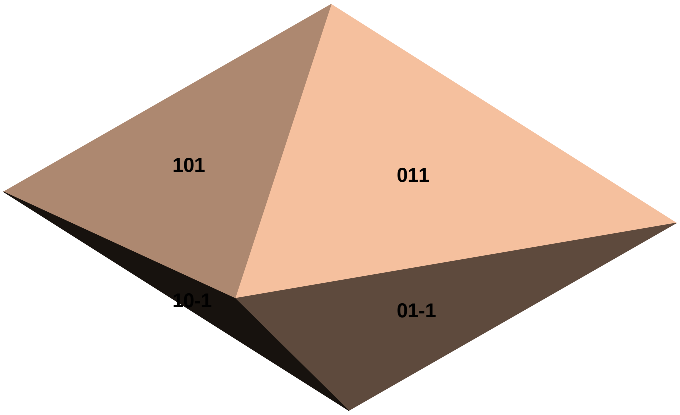
by Donald Howard

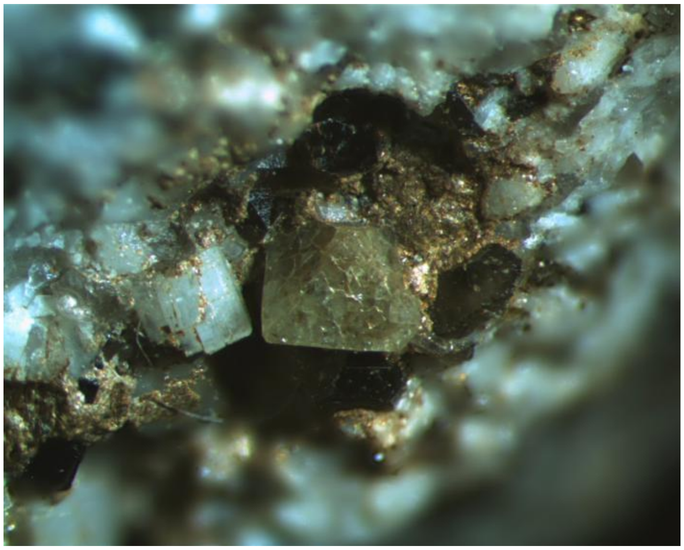
Recently, while looking through some material from the boulders that contain kainosite found on the talus at the base of Liberty Bell, I ran across some interesting twin crystals of zircon. Zircon crystals from these boulders are usually in the form of octahedrons – tetragonal bipyramids with no prism faces at all. All eight faces of a crystal are therefore equivalent and form the set represented by {011}. These zircons are green in color, so they contrast nicely with the orangey-brown smectite that usually lines the cavities. They are generally more numerous than the kainosite and allanite crystals that are also present in some of the cavities. A photograph of a single crystal is shown below right, with a diagram of this particular form shown directly above it.

The usual form of twins in zircon have one of the {011} faces as the composition plane. That means, you take two of the shapes shown at right and glue them together so that their faces just fit together. Twins of this type occur in the kainosite boulder; a color photograph of just such a twin is shown in plate 1.

A more complicated and more interesting twin is shown in plate 2. The form of the individual members is the same, but this time the composition plane is (11-1). If the c and a cell dimensions in zircon were the same, this plane would be perpendicular to the (011) plane, and reflection would map it into itself, but rotated through 90^o. Since the c-dimension is slightly less than the a-dimension, the rotation is actually through 112^o, and the resulting image plane is tilted 9^o from the original. This allows the zircon shown in plate 2 to be a penetration twin, with the tip of the left-hand member just showing on the right of the pair.

I have not seen twinning on {111} listed for zircon in the literature. This is one of the things that make looking for and studying twins so interesting. It is often possible to discover new twinning rules that in turn have something to say about the atomic structure of the minerals that compose them. I encourage you all to keep your eyes open to new possibilities in the crystals that you are examining.





A single crystal of zircon in a cavity from the boulder containing kainosite, located in the talus at the base of Liberty Bell, Washington Pass, Washington.

President's Message

During the Spring 2009 meeting, previous president Doug Merson stepped down as president of the Northwest Micro Mineral Study Group (NWMMSG), due to increasing difficultly in his getting away from work to attend meetings. Rudy Tschernich was elected the new president and conducted the remainder of the meeting.

Doug graciously has donated his Toshiba laptop computer with Power Point installed and his digital Epson projector to the NWMMSG so that we will always have the equipment to show programs at our meetings. I have donated a projection table and a Kodak slide projector with reels to the NWMMSG for those programs using slides. This equipment will be stored with the president and brought to all NWMMSG meetings.

Field Trip Sunday November 15th, 2009, weather and Weyerhaeuser permitting. We will try a field trip to the Wolf Point area near Mt. Saint Helens. The weather has to be clear and dry. Rainy weather in that area is horrible. Also, Weyerhaeuser only opens some sections during deer hunting season. Therefore, we will not know if this area is open until a few days before the meeting.

The Spring 2010 issue of the Micro Probe will contain the new membership list with photos attached. At the coming Fall 2009 meeting, Pam and Kirby Harris will be photograph all the members attending the meeting so that faces can be attached to the names in the membership list. For those **not** attending the meeting, please send your digital portrait photos (head shot) by email attachment to bikeklein@yahoo.com, or mail them to Kelly Starnes, 1276 SE Goodnight Ave, OR 97333.

Rudy Tschernich, president

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