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# A first occurrence of euclase in the Swiss Alps: Discovery and refinement of the crystal structure

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**Abstract**

A first finding of the rare beryllium mineral euclase $BeAlSiO_4(OH)$ has been reported in the Swiss Alps (Pizzo Giubine near the St. Gotthard Pass, Canton Ticino); until recently, the presence of this species in the Alpine region was known only in the Eastern part (Südtirol, Rauris, Kärnten). Taking advantage of the good quality of the crystals, a suitable set of X-ray diffraction data has been collected for structure refinement (final R index = 0.019 on 1636 independent observations). In this structure the Be–OH bond distance (1.608[1] Å) is definitely shorter than the Be–O distances (average 1.654[1] Å). The hydrogen bonds are particularly weak (shortest O...O distance = 2.933[1] Å).

*Keywords:* Euclase, x-ray diffraction, structure refinement, beryllium minerals, Canton Ticino, Swiss Alps.

**Riassunto**

Il primo ritrovamento nelle Alpi Svizzere di un minerale raro di berillio, l'euclasio $BeAlSiO_4(OH)$, viene segnalato al Pizzo Giubine presso il San Gottardo (Canton Ticino); finora, la presenza di questa specie nelle Alpi era soltanto nota nella regione orientale (Alto Adige, Rauris, Carinzia). Data la qualità molto buona dei cristalli, mediante diffrazione di raggi X su cristallo singolo sono stati ottenuti 1636 riflessi indipendenti, con cui si è raffinata la struttura del minerale (indice di disaccordo finale R = 0.019). In questa struttura il legame Be–OH (1.608[1] Å) è decisamente più corto dei legami Be–O (media 1.654[1] Å). I legami ad idrogeno risultano particolarmente deboli (distanza O...O più corta = 2.933[1] Å).

## Introduction

The presence of euclase in the eastern Alpine region has been known for over a century (BECKE, 1882; BREZINA, 1884; KOECHLIN, 1886, 1905; CAVINATO, 1929; MEIXNER, 1957, 1958; BRENDLER, 1956; NIEDERMAYR, 1976; WENINGER, 1974; HUBER and HUBER, 1980). The crystals are usually well-shaped, colourless, slightly greenish or yellowish, a few millimetres long (although exceptional specimens are known with crystals reaching 2 cm in length); sometimes, they contain inclusions of chlorite.

Although some of the occurrences are known exactly (Rosshufgletscher in Windtal, Ahrntal; Grieswies Alp, Hoher Goldberg and Krumlkeeskopf, Rauris), some others (Mölltal in Kärnten, etc.) are not sufficiently documented, and it seems that the situation may be similar to that which occurred for the first findings of milarite (PARKER, 1973). In any case, euclase continues to be one of the greatest rarities in the Alps, in spite

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of the recent considerable increase in number and skill of "strahlers" and collectors in general.

Some time ago (autumn 1990), during a mineral show at Ispra, one of us (C.M.G.) was contacted by Mr. Giuseppe Raineri, a collector from Bellagio, who wanted to know the nature of some interesting colourless transparent crystals he had noticed in a large fissure at Pizzo Giubine, Canton Ticino. These crystals (Fig. 1) attain a maximum length of 3 millimetres and are found together with pericline and quartz; their habit is nearly ideal, practically identical to the "classical" drawing reported by BECKE (1882) for Alpine euclase (see also figure 56, table 153 in volume 3 of GOLDSCHMIDT's Atlas, 1916).

Since the crystals were so perfect and so few, it was a pity to damage them, so we waited until one partly damaged crystal was available for X-ray study. At the same time, during our work on Alpine gadolinite (DEMARTIN et al., in press) we had come across the problem of determining the content of lighter elements, such as boron or hydrogen, using refined crystal-structure data. By comparing the structures of gadolinite and datolite, it can be seen that the Be in gadolinite may be replaced by B and that the oxygen atom (corresponding to the OH group in datolite) may be partly replaced by a hydroxyl group, thereby accounting for the presence of hydrogen or "water" in the crystal structure (see also hingganite: SEMENOV et al., 1963; VOLOSHIN et al., 1983; YAKUBOVICH et al., 1983).

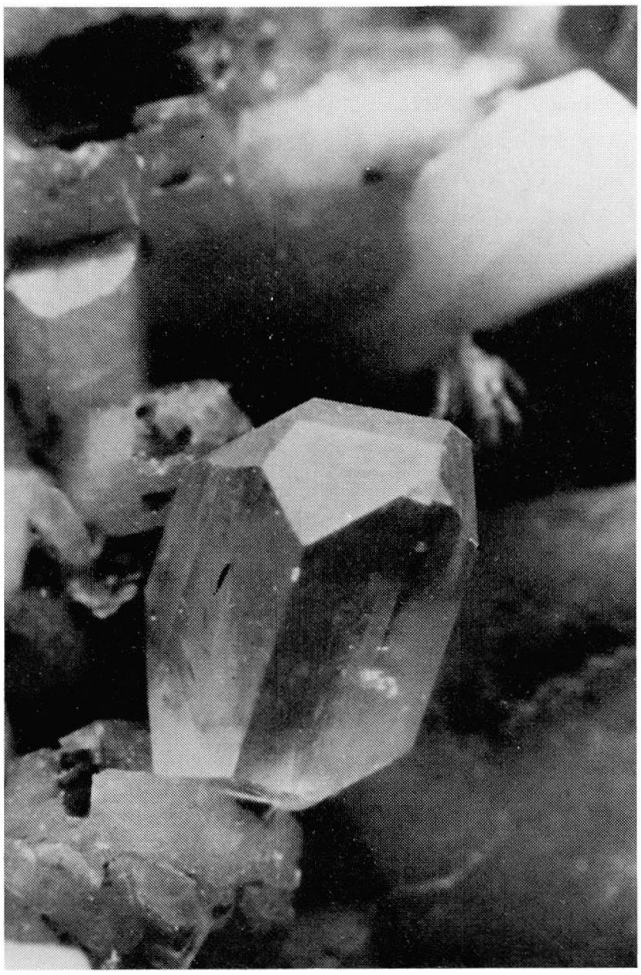

*Fig. 1* Euclase crystal from Pizzo Giubine (×15).

In order to have a meaningful comparison, accurate measurements of Be–OH as well as Be–O bond lengths (and their equivalents for tetrahedral boron) would be useful. Whereas there are no particular problems concerning boron, accurate measurements of Be–OH bond lengths are scarce in the literature, and often controversial. For instance, there are two well-studied beryllium minerals, euclase and bertrandite, containing Be–OH bonds. For euclase, MROSE and APPLEMAN (1962) find the Be–OH bond longer than the Be–O bonds (1.68 against 1.60 to 1.64 Å, respectively). In a more recent study, however, HAZEN et al. (1986) find the Be–OH bond shorter than the Be–O bonds (at ordinary temperature and pressure 1.612[4] against 1.633[3] to 1.667[3] Å), which contrasts with the general case where bonds with OH groups are longer than the corresponding ones with O groups. Similarly for bertrandite, HAZEN and AU (1986) find an average Be–OH bond distance of 1.628(5) Å (in the structure at ordinary temperature and pressure) and an average Be–O bond distance of 1.645(6) Å.

Thus, we thought it appropriate to undertake careful redeterminations of the crystal structures of selected samples of beryllium minerals containing both O and OH groups in order to obtain comparative data for Be–O and Be–OH bond lengths. At the same time, concerning the particular problem of identifying the Alpine sample in question, in our opinion nothing could be better than a crystal-structure refinement (particularly as such a method requires only a small fragment of material, definitely smaller than any sample suitable for ordinary powder-diffraction procedures, and because of the presence of lighter elements such as beryllium and hydrogen, difficult to determine quantitatively by the usual routines).

## Crystal structure refinement

A crystal measuring 0.07 × 0.10 × 0.12 mm was mounted on an Enraf-Nonius CAD-4 diffractometer, and twenty-five intense reflections having a $\Theta$ value in the range 11.3–20.3° were centered using graphite-monochromated MoK$\alpha$ radiation ($\lambda$ = 0.71073 Å). The unit-cell parameters were obtained by refinement of their setting angles,

*Tab. 1* Comparison between unit cell parameters for euclase.

| SAMPLE | (1) | (2) | (3) | (4) | (5) | (6) |
|---|---|---|---|---|---|---|
| a (Å) | 4.632(1) | 4.618 | 4.64 | 4.62 | 4.63 | 4.6335(2) |
| b (Å) | 14.334(1) | 14.29 | 14.33 | 14.31 | 14.27 | 14.322(1) |
| c (Å) | 4.781(1) | 4.763 | 4.72 | 4.77 | 4.76 | 4.7800(3) |
| β (°) | 100.33(1) | 100.25 | 100.26 | 100.25 | 100.26 | 100.310(5) |

(1) Present work.
(2) MROSE and APPLEMAN (1962), sample from Villa Rica, Minas Gerais, Brazil.
(3) GOSSNER and MUSSGNUG (1929), sample from Minas Gerais, Brazil.
(4) KNORRING et al. (1964), sample from Muiane, Mozambique.
(5) BISCOE and WARREN (1933), sample from Minas Gerais, Brazil.
(6) HAZEN et al. (1986), sample from Minas Gerais, Brazil.

together with an orientation matrix relating the crystal axes to the diffractometer axes. A total of 3967 diffracted intensities (3288 with $I > 3\,\sigma\,[I]$) were collected at room temperature with variable scan speed (maximum scan time for each reflection: 90 sec.), by exploring the hemisphere of the reciprocal lattice with $-8 \le h \le 8$, $0 \le k \le 25$ and $-8 \le l \le 8$, out to a maximum 2 Θ angle of 80°. The diffracted intensities were corrected for Lorentz, polarization and background effects. An empirical absorption correction was applied by performing a psi-scan correction (NORTH et al. [1968]). After averaging the symmetry related data, whose agreement was 1.3% on Fo basis, 1928 independent reflections were obtained; of these 1636 with $I > 3\,\sigma\,(I)$ were considered for the structure refinement. Scattering factors for neutral atoms and anomalous dispersion corrections for scattering factors were taken from CROMER and WABER (1974) and CROMER (1974) respectively. The structure was refined, starting from the atomic positions reported in HAZEN et al. (1986), by full-matrix least-squares, minimizing the function $\Sigma w(|Fo|-k|Fc|)^2$. Weights assigned to individual observations were $1/\sigma^2(Fo)$, where $\sigma(Fo) = [\sigma^2(I) + (kI)^2]^{1/2}/2FoLp$, $\sigma^2(I)$ is the standard deviation for each reflection as derived from counting statistics, k (= 0.04) is a coefficient for improving the "goodness" of fit and Lp is the Lorentz-polarization factor. Anisotropic thermal parameters were assigned to all the non-hydrogen atoms. A secondary extinction correction was applied as described in STOUT and JENSEN (1968); the maximum extinction correction (y = 0.65) was applied to the 0 2 0 reflection. All the calculations were performed on a PDP11/73 computer using the SDP-Plus Structure Determination Package (FRENZ et al. [1980]). Maximum residual in the final difference Fourier synthesis: 0.46 e/Å$^3$.

*Tab. 2* Crystal data.

| | |
|---|---|
| Crystal system | monoclinic |
| Space Group | $P2_1/c$ |
| Z | 4 |
| $D_{calc.\,d}$ ($gcm^{-3}$) | 3.085 |
| μ(Mo–Kα) ($cm^{-1}$) | 8.80 |
| Min. transmission factor | 0.83 |
| Scan mode | Θ/2Θ |
| Scan width (°) | 0.80 + 0.35 tanΘ |
| Θ-range (°) | 1–40 |
| Octants of reciprocal space explored | ±h, +k, ±1 |
| Measured reflections | 3967 |
| Unique observed reflections with $I > 3\,\sigma\,(I)$ | 1636 |
| Final R and Rw indices $ | 0.019, 0.030 |
| Extinction coefficient g | $1.86 \cdot 10^{-5}$ |
| No. of variables | 78 |
| GOF & | 1.100 |

$ $R = [\Sigma(|Fo|-k|Fc|)/\Sigma|Fo|]$ $R_w = [\Sigma w(|Fo|-k|Fc|)^2/\Sigma w|Fo|^2]^{1/2}$
& $GOF = [\Sigma w(|Fo|-k|Fc|)^2/(N_{observations}-N_{variables})]^{1/2}$
$w = 1/(\sigma(|Fo|))^2$, $\sigma(|Fo|) = [\sigma^2(I) + (0.04I)^2]^{1/2}/2|Fo|Lp$

## Discussion[1]

The unit cell parameters (see Tab. 1) are in good to excellent agreement with the corresponding data in the literature. The axial ratio a : b : c is 0.3231 : 1 : 0.3335, corresponding to the older goniometric data (0.3237 : 1 : 0.3332) reported by GOLDSCHMIDT (1916): therefore, there is no doubt about the nature of the crystal examined here; this identification is confirmed by the details of the structure (see below).

Atomic coordinates and anisotropic thermal parameters are reported in table 3 and 4; selected interatomic distances and angles are reported in

[1] The complete list of structure factors is available from the authors on request.

*Tab. 3* Fractional atomic coordinates and thermal parameters with e.s.d.'s.

| Atom | x | y | z | $U_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Si | 0.53620(5) | 0.10019(2) | 0.17739(5) | 0.00289(6) |
| Al | 0.95711(5) | 0.44474(2) | 0.24878(5) | 0.00371(8) |
| Be | 0.4565(2) | 0.30095(7) | 0.1741(2) | 0.0058(3) |
| O(1) | 0.7632(1) | 0.03230(4) | 0.3821(1) | 0.0042(2) |
| O(2) | 0.6523(1) | 0.37701(4) | 0.3802(1) | 0.0046(2) |
| O(3) | 0.5250(1) | 0.19980(4) | 0.3432(1) | 0.0046(2) |
| O(4) | 0.2113(1) | 0.05288(4) | 0.1003(1) | 0.0042(2) |
| O(5) | 0.1204(1) | 0.33182(4) | 0.1593(1) | 0.0063(2) |
| H | 0.004(5) | 0.303(1) | 0.070(4) | 0.034(5) |

The hydrogen atom was refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $U_{eq} = (Tr\ \underline{U})/3$

*Tab. 4* Table of general temperature factor expressions – U's.

| Name | U(1,1) | U(2,2) | U(3,3) | U(1,2) | U(1,3) | U(2,3) |
|---|---|---|---|---|---|---|
| Si | 0.00283(6) | 0.00251(7) | 0.00330(6) | –0.00003(6) | 0.00032(5) | 0.00000(6) |
| Al | 0.00366(8) | 0.00371(8) | 0.00375(8) | 0.00017(6) | 0.00043(6) | –0.00026(7) |
| Be | 0.0067(3) | 0.0044(3) | 0.0064(3) | –0.0002(3) | 0.0016(3) | 0.0001(3) |
| O(1) | 0.0041(2) | 0.0045(2) | 0.0039(2) | 0.0013(2) | 0.0000(1) | 0.0001(2) |
| O(2) | 0.0048(2) | 0.0044(2) | 0.0046(2) | –0.0013(2) | 0.0017(1) | –0.0011(2) |
| O(3) | 0.0067(2) | 0.0028(2) | 0.0043(2) | 0.0003(2) | 0.0010(2) | 0.0000(2) |
| O(4) | 0.0034(2) | 0.0047(2) | 0.0046(2) | –0.0011(2) | –0.0002(1) | 0.0004(2) |
| O(5) | 0.0045(2) | 0.0057(2) | 0.0087(2) | 0.0006(2) | 0.0001(2) | –0.0023(2) |

The form of the anisotropic thermal parameter is: $\exp[-2\pi^2\{h^2a^{*2}U(1,1) + k^2b^{*2}U(2,2) + l^2c^{*2}U(3,3) + 2hka^*b^*U(1,2) + 2hla^*c^*U(1,3) + 2klb^*c^*U(2,3)\}]$ where $a^*$,$b^*$, and $c^*$ are reciprocal lattice constants.

table 5 and perspective views are given in figure 2 and 3. There is a close agreement (often within the estimated standard deviation) between our values and HAZEN et al.'s results (1986) at ordinary pressure, which are the most accurate so far reported in the literature. With respect to these authors, our values for the standard deviation are substantially smaller: this corresponds to our considerably lower value for the disagreement index (1.9% versus 4.1%), and to the greater number of independent reflexions considered in our refinement (1636 versus 745). Our bond distances and angles are also in excellent agreement with HAZEN et al. (1986), including the length of the Be–OH bond (1.608[1] Å) which is smaller than the average of the Be–O bonds (1.654[1] Å).

This apparently unusual behaviour of the OH group in the euclase structure is also shown by the presence of only weak hydrogen bonds, the shortest contacts being 2.933(1) Å.

With respect to HAZEN et al.'s (1986) data at room temperature and pressure, the only relevant difference we have found regards the thermal parameters, which in our case are systematically smaller (by about 30%), with the only exception of the H atom. This exception is very likely to be due to assumption of different scattering factors (reference not given in HAZEN et al.); the overall difference in magnitude between the corresponding B's can be connected with extinction or/and absorption problems. In view of the recent improvement of calculation routines (PILATI et al., 1990), it will be interesting to compare our results with the corresponding lattice-dynamical estimations.

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The courtesy of Mr Giuseppe Raineri in indicating an interesting subject of investigation, and in providing us with adequate crystals is gratefully acknowledged. Financial support from the Italian CNR is also acknowledged. We also thank Miss Suzanne M. Mulley for helpful discussion, and Mr. Roberto Appiani for having kindly provided the photograph in figure 1.

*Tab. 5* Selected interatomic distances (Å), angles (deg.).

| | | | |
|---|---|---|---|
| Tetrahedron around Si | | | |
| Si–O(1) | 1.625(1) | O(1)...O(2)[a] | 2.696(1) |
| Si–O(2)[a] | 1.640(1) | O(1)...O(3) | 2.635(1) |
| Si–O(3) | 1.638(1) | O(1)...O(4) | 2.684(1) |
| Si–O(4) | 1.631(1) | O(2)[a]..O(3) | 2.632(1) |
| | | O(2)[a]..O(4) | 2.657(1) |
| Si-O | 1.634 | O(3)...O(4) | 2.700(1) |
| O(1)–Si–O(2)[a] | 111.35(3) | O(1)–Si–O(3) | 107.67(3) |
| O(1)–Si–O(4) | 110.98(3) | O(2)[a]–Si–O(3) | 106.83(3) |
| O(2)[a]–Si–O(4) | 108.64(3) | O(3)–Si–O(4) | 111.31(3) |
| Tetrahedron around Be | | | |
| Be–O(2) | 1.631(1) | O(2)...O(3) | 2.606(1) |
| Be–O(3) | 1.663(1) | O(2)...O(3)[a] | 2.758(1) |
| Be–O(3)[a] | 1.668(1) | O(2)...O(5) | 2.583(1) |
| Be–O(5) | 1.608(1) | O(3)...O(3)[a] | 2.790(1) |
| | | O(3)...O(5) | 2.697(1) |
| Be–O | 1.643 | O(3)[a]..O(5) | 2.649(1) |
| O(2)–Be–O(3) | 104.60(6) | O(2)–Be–O(3)[a] | 113.42(7) |
| O(2)–Be–O(5) | 105.75(7) | O(3)–Be–O(3)[a] | 113.80(6) |
| O(3)–Be–O(5) | 111.10(7) | O(3)[a]–Be–O(5) | 107.90(7) |
| Octahedron around Al | | | |
| Al–O(1)[a] | 1.850(1) | O(1)[a]..O(1)[b] | 2.461(1) |
| Al–O(1)[b] | 1.984(1) | O(1)[a]..O(2) | 2.839(1) |
| Al–O(2) | 1.910(1) | O(1)[a]..O(4)[c] | 2.743(1) |
| Al–O(4)[c] | 1.871(1) | O(1)[a]..O(5)[e] | 2.739(1) |
| Al–O(4)[d] | 1.932(1) | O(1)[b]..O(4)[c] | 2.686(1) |
| Al–O(5)[e] | 1.868(1) | O(1)[b]..O(4)[d] | 2.631(1) |
| | | O(1)[b]..O(5)[e] | 2.937(1) |
| Al–O | 1.903 | O(2)...O(4)[e] | 2.597(1) |
| | | O(2)...O(4)[d] | 2.801(1) |
| | | O(2)...O(5)[e] | 2.657(1) |
| | | O(4)[c]..O(4)[d] | 2.525(1) |
| | | O(4)[d]..O(5)[e] | 2.653(1) |
| O(1)[a]–Al–O(2) | 98.04(3) | O(1)[a]–Al–O(4)[c] | 165.46(3) |
| O(1)[a]–Al–O(4)[d] | 92.93(3) | O(1)[a]–Al–O(5)[e] | 94.87(3) |
| O(2)–Al–O(4)[c] | 95.57(3) | O(2)–Al–O(4)[d] | 85.02(3) |
| O(2)–Al–O(5)[e] | 89.34(3) | O(4)[c]–Al–O(4)[d] | 83.18(3) |
| O(4)[c]–Al–O(5)[e] | 90.35(3) | O(4)[d]–Al–O(5)[e] | 170.95(3) |
| O(5)–H | 0.75(2) | O(5)...O(3)[f] | 2.933(1) |
| O(3)[f]..H | 2.29(2) | O(5)–H...O(3)[f] | 144(2) |
| Be...H | 2.07(2) | | |

Symmetry codes:
a = x, 1/2–y, –1/2+z
b = 2–x, 1/2+y, 1/2–z
c = 1–x, 1/2+y, 1/2–z
d = 1+x, 1/2–y, 1/2+z
e = 1+x, y, z
f = x–1, 1/2–y, z–1/2

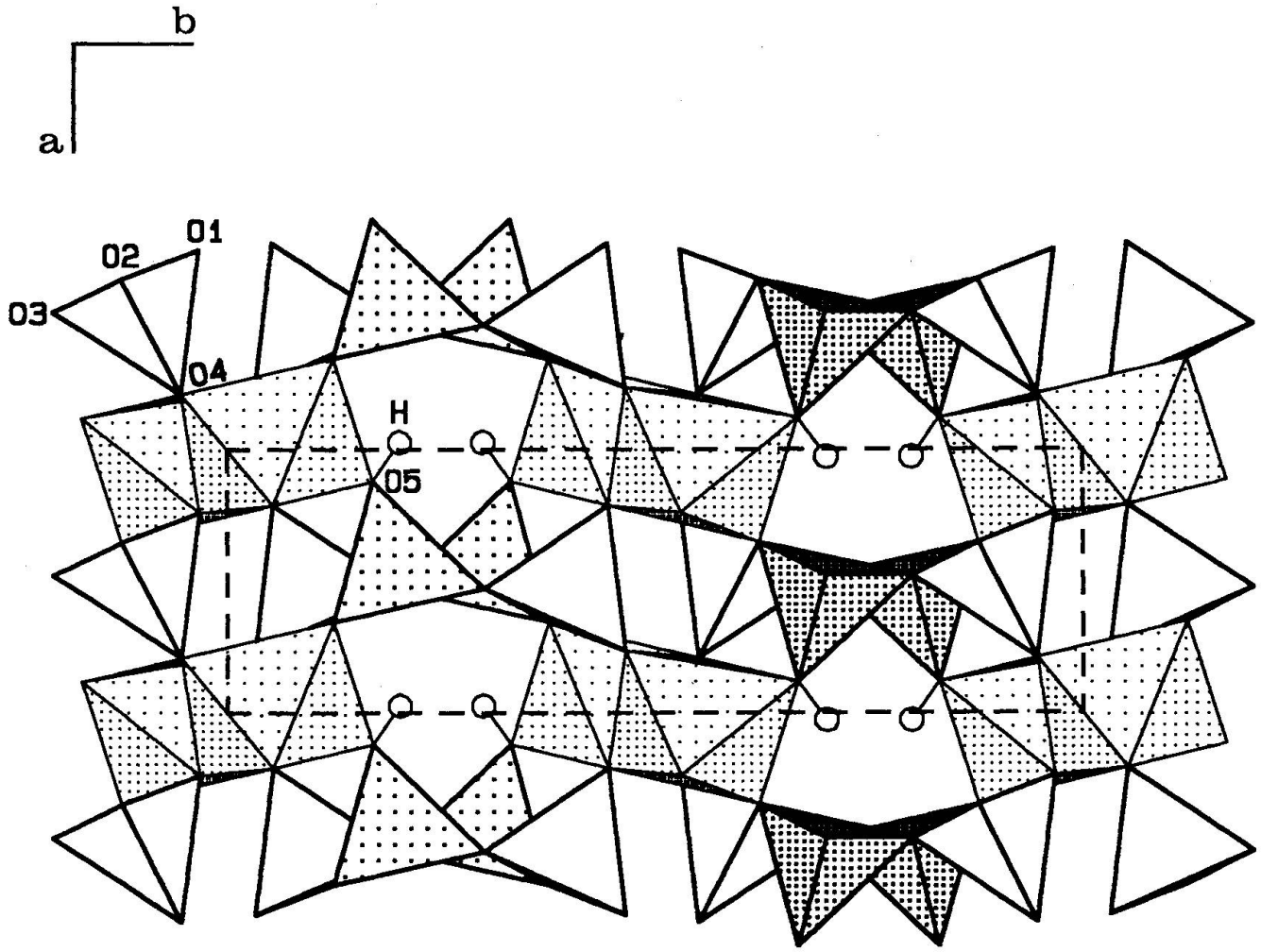


*Fig. 2* A perspective view along the c axis of the crystal structure of euclase, drawn by the program POLIEDRI (PILATI, 1990). The octahedra are centered on Al atoms, the white tetrahedra surround the Si atoms, and the dotted tetrahedra the Be atoms; the H atoms are represented by small circles. All the corners of these polyhedra are occupied by O atoms.

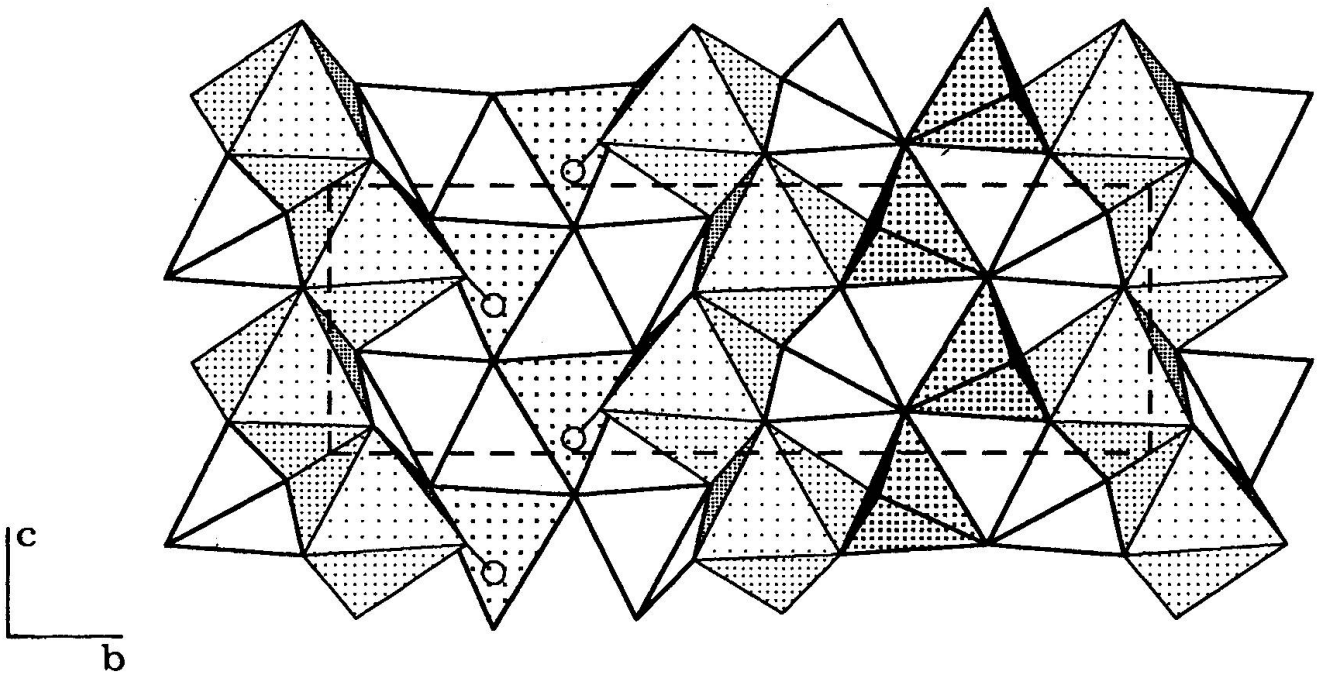


*Fig. 3* A perspective view of the crystal structure of euclase along the a axis. The same drawing program and symbols as for figure 1 have been used.

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