

Optical and Chemical Study of Quartz from Granitic Pegmatites

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Of the nine quartz samples collected from the Contu - Negovanu granitic pegmatites (Southern Carpathians, Romania), three were separated under binocular lenses and turned into fine grained powders, for X-ray fluorescence (XRF) and inductively coupled plasma mass spectrometry (ICP-MS) investigation techniques, whilst the other six samples were prepared in thin slides for electron microprobe analyzer (EMPA) and optical investigations, using a Babinet compensator and a Rayleigh interferometer. The results show that the quartz samples belonging to the Contu-Negovanu granitic pegmatites are chemically very pure, showing an expected high participation of silica and only some remarkable contents of Fe, derived from iron oxide micro-inclusions finely disseminated in the quartz grains. The minor and trace elements distribution suggests that different crystallization conditions influenced the quartz composition, thus resulting some rather high contents of Ba, Rb, Sr and some REE contents (Ce) in the pegmatite quartz crystallized from alkali- and alkaline-earth-rich fluids, while the metamorphic quartz appears to be highly pure, showing no such presence. The analyzed quartz samples present birefringence parameters within the limits of other quartz samples and a decrease of the linear birefringence of quartz with the light wavelength increase is emphasized by the data obtained using the channeled spectrum.

Keywords: quartz; main refractive indices; birefringence; channeled spectrum; major and minor elements

Quartz is the most abundant mineral in the Earth's crust, along with feldspars and occurs as an essential constituent of many acid igneous, sedimentary and metamorphic rocks. Although a silicon oxide (SiO₂), quartz has a three-dimensional framework structure typical for tectosilicates, in which the crystalline edifice is built up of (SiO₄)⁴⁻ tetrahedral linked by sharing each of their corners with other similar ones. Within a tetrahedron, the silicon ion is situated in the center and the oxygen ions are placed in the corners and thus, every silicon atom has four oxygen atoms and every oxygen atom has two silicon atoms as nearest neighbors; the tetrahedron edges represent O-O bonds. Tetrahedra are grouped to form regular trigonal and hexagonal helices which are consistent with the trigonal and the hexagonal symmetry of α -quartz and β -quartz respectively. These two forms represent low and high-temperature modifications: α -quartz is stable at atmospheric temperatures and up to 573°C, whereas β -quartz is stable from 573 to 870°C and can metastably exist above 870°C [1].

The quartz bulk composition is very close to 100% SiO₂, but it may present small amounts of other elements, generally due either to minute inclusions of other minerals, or to the liquid infillings in cavities within the crystals. Usually, Al³⁺ is the main element that penetrates the quartz structure substituting for Si⁴⁺. Therefore, in order to maintain the lattice electro-neutrality, either monovalent alkaline ions are interstitially introduced, such as Li⁺ or Na⁺, or (OH)⁻ anions substituting for oxygen are present; in most of the cases, a combination of the two mechanisms is effective.

Quartz is an uniaxial positive crystal and its enantio-morphism makes it *optically active*: the polarization plane of the light passing along the optic axis is rotated either clockwise or anticlockwise depending on the crystal orientation, right- or left-handed. In some metamorphic and igneous rocks, quartz develops undulatory extinction due to strain; this effect has been produced experimentally at pressures of about 138 kbar, the optic axis being

displaced by 2-7°. In some instances, quartz may show a distinctly biaxial character, with the acute angle $2V$ between the optical axes as high as 8-10°.

Quartz is one of the most stable minerals and in addition to being chemically resistant to most attacking solutions, its hardness and lack of cleavage help it resist many of the other weathering agents.

Properly cut plates of quartz are used for frequency control in radio, TV and other electronic communications equipment, for crystal-controlled clocks and watches and for optical devices. Other uses include gems (its varieties called amethyst-violet, citrine- yellow, rose quartz-pink, smoky quartz-brown), abrasive in sandpaper, sandblasting, millstones, grind stones, etc.

Modern requirements of high-grade quartz for piezoelectric uses has led to an intensive study of its synthetic production, which otherwise is effective since 1900 through a technique based on the greater solubility of quartz in a sodium metasilicate solution at temperatures above 300°C.

The aim of this work is to establish the composition and purity of quartz samples belonging to various types of granitic pegmatites as compared to metamorphic samples and to assess some of their optical characteristics.

Experimental part

Samples and analytical methods

Nine quartz samples (Q-16, Q-60, Q-61, Q-81, Q-81A, Q-95, Q-95A, Q-96 and Q-96A) were collected from the Conțu-Negovanu granitic pegmatites and associated metamorphics (Southern Carpathians, Romania) [2] in order to be chemically and optically investigated. Samples Q-16, Q-81 and Q-81A belong to the feldspar pegmatites and samples Q-61, Q-95, Q-95A, Q-96 and Q-96A are taken from the albite-spodumene pegmatites, while sample Q-60 belong to a metamorphic quartz vein. Three quartz samples (Q-16, Q-60 and Q-61) have been separated

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under binocular lenses and turned into fine grained powders, for XRF and ICP-MS investigation techniques. The

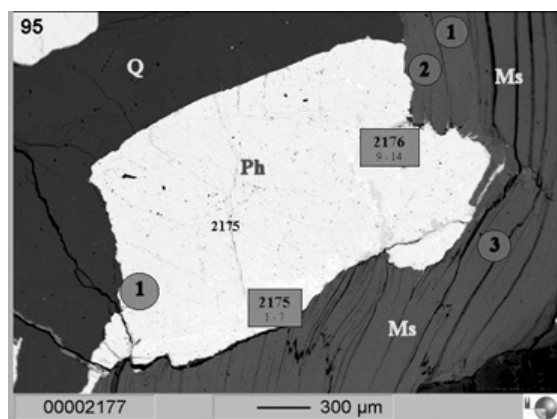


Fig. 1. EMPA image of sample Q-95 (Q - quartz, MS- muscovite, Ph- Fe, Mn phosphates)

other six samples have been prepared in thin slides for optical and EMPA investigations (fig. 1).

In X-ray fluorescence technique, bulk analyses were performed with a Philips PW 2400 X-ray spectrometer, using the analytical procedure called *oxiquant*. Seventy-two natural rocks and clays were used to determine the calibration curves of the pertinent elements.

EMPA were carried out with a JEOL JXA-8900 instrument, using an operating current of 20 nA and accelerating voltage of 20 kV. X-ray intensities of the alkalis, the minor (Ti and Mn) and the major elements were counted for 5 s, 40 and 60 s, respectively. In order to minimize losses of Na and K, the beam diameter was expanded to 10 mm. Components were standardized using natural minerals, glasses of natural rocks [3] and synthetic oxide compounds. The results were corrected using the ZAF procedure [4].

The ICP-MS investigations were performed with a Perkin Elmer / Sciex ELAN 6000 ICP-MS (quadrupole mass spectrometer). Measurements of element concentrations were performed using as internal standards Ru-Re (10 ng/mL) to minimize drift effects and two calibration solutions (high purity chemical reagents). A batch of 5-7 samples was bracketed by two calibrations procedures. Accuracy and precision of determinations were checked with certified reference materials (CRM) [5,6]. All the quantitative investigations were performed in the laboratories of the Geological Institute of the *Albertus Magnus* University of Koln, Germany.

As in our previous researches [7-9], the analyzed specimens were cut parallel to their optical axis. Thus, the flat plate can be investigated perpendicularly to one of the main planes of the crystal, by using a parallel beam of monochromatic light that propagates along a main axis (differing from the optical axis) and having its electric field intensity parallel with the other one. The main refractive indices of anisotropic media can be determined by using refractometers for mineralogy, or interferometers standardized for the monochromatic light in which the instruments will work. The measurements are usually made in linearly polarized light at normal incidence on the layer. The results must be done in the monochromatic light.

The main refractive indices were determined in thin sections (fig. 2) with both a Babinet compensator [10] and a Rayleigh interferometer [11] standardized in monochromatic yellow radiation of a Na lamp, with $\lambda = 589.3$ nm.

The linear birefringence of the same samples was additionally measured using the method of channeled spectrum [12].

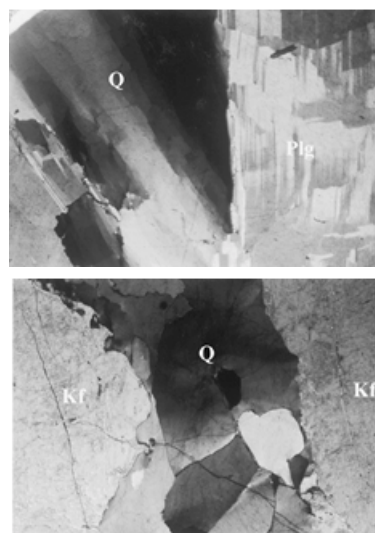


Fig. 2. Polarized light images of quartz associated with plagioclase (Q-95 up) and K-feldspar (Q-16 down), respectively, in granitic pegmatites (N+, x 6.3, Q- quartz, Plg - plagioclase feldspar, Kf-K-feldspar)

The specimen thickness was microscopically measured by the distance on which the microscope tube must be translated in order to obtain good images for the both faces of the crystalline plate [13].

Optical considerations

The crystals with two distinct values of the refractive indices are named uniaxial crystals and those with three distinct values are named biaxial crystals. In uniaxial crystals there is a propagation direction, while in biaxial crystals there are two directions (named optical axes) for which the light velocity does not change with the light polarization state (the light velocity does not depend on the orientation of the electric field intensity of the electromagnetic waves).

The electrical and optical parameters of anisotropic uniaxial crystals are expressed generally by a 3×3 matrix, which in the main system of coordinates has only diagonal elements differing from zero [14].

The light velocity in the anisotropic crystals depends on both the propagation direction and the light polarization state. Within the uniaxial crystals, the main directions coincide with the basic directions of polarization, so in the case of propagation parallel to these directions, light polarization state is not changed.

In order to measure the main refractive indices of a uniaxial crystal, a polarizer with its transmission direction parallel to one of the main axes of the analyzed crystalline layer has been introduced in the measure beam of the interferometer. An identical polarizer and a glass plate with the same thickness as the crystalline layer have been introduced in the comparison beam of the interferometer for compensation reasons.

The displacement of a zero-fringe obtained in white light relative to the zero fringe of a fix fringe-system permits the evaluation of the supplementary pathway introduced by the anisotropic layer beside the glass plate. The relative displacement of fringes is expressed by the order of fringe k , from the mobile fringe-system that superposes with the zero fringe of the fix fringe-system when the plate working as compensator pathway is rotated:

$$(n_i - n_g)L = k\lambda; \quad i = e, o. \quad (1)$$

In eq. (1) n_i is one of the main refractive indices of the anisotropic plate in which light propagates along a main direction, e refers to the extraordinary and o to the ordinary rays, n_g represents the refractive index of the glass plate, k is the interference order that measures the displacement of the fringes, and λ is the light wavelength. The refractive index was estimated by using the formula:

$$n_i = n_g + \frac{k\lambda}{L}; \quad i = e, o. \quad (2)$$

In order to identify the main directions in the anisotropic crystal, two identical crossed polarizers were used. The anisotropic layer has been introduced between the crossed polarizers and it was rotated around the light propagation direction until the emergent flux density became null after the second polarizer. Under these conditions, the transmission directions of the polarizers are parallel to the main directions of the crystal. Subsequently, the second polarizer was eliminated and the system, consisting from one polarizer and the anisotropic layer, (orientated with one from the main directions parallel to the polarizer transmission direction) has been introduced in the measure beam of the Rayleigh interferometer. Light propagates through the polarizer and then, as a linearly polarized beam, it crosses the anisotropic layer. The second polarizer has been introduced in the comparison beam, to compensate the absorption due to the polarizer in the measure beam.

One of the main refractive indices has been measured by counting the displacement of the fringes relatively to the central fringe of the fixed fringe system. The second main refractive index has been estimated after a rotation of the polarizer in the measure beam with 90 degrees around light propagation direction.

Then the orientation of the anisotropic layer has been modified in such a way that light propagates along the other main direction. In the case of uniaxial crystals crossed by light on a main direction differing from the optical axis, a common value of the ordinary index is obtained in the two measurements.

Recently, a new method based on a channeled spectrum has been developed [15,16] and it can be applied to crystals or to liquid crystalline samples. By applying this method, the sample can be investigated using a parallel beam of light that propagates along a main axis (different from the optical axis). In order to register a channeled spectrum, the sample between two crossed polarizers is introduced in the measure beam of one spectrophotometer. Two identical polarizing filters having parallel transmission directions are introduced in the compensatory beam of the spectrophotometer in order to compensate absorption in the polarizing filters from the measure beam (fig. 3).

A channeled spectrum consists of successive maxima and minima corresponding to the emerging light from the device [15]. The highest maxima are obtained in the

channeled spectrum when the main axes of the crystalline plate bisect the angle between the transmission directions of the polarizers. The radiations keeping their polarization

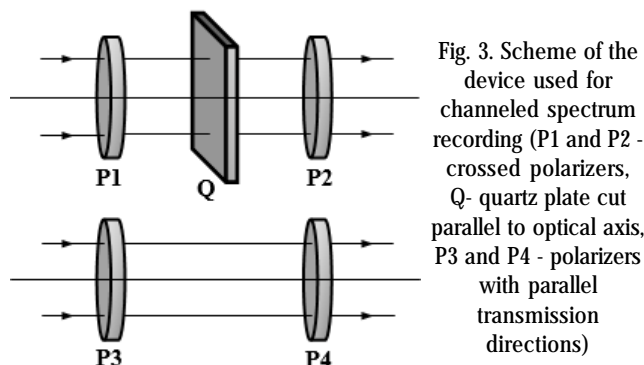


Fig. 3. Scheme of the device used for channeled spectrum recording (P1 and P2 - crossed polarizers, Q- quartz plate cut parallel to optical axis, P3 and P4 - polarizers with parallel transmission directions)

state do not pass through the device, while the radiations which change their azimuth angle from θ in $2\pi-\theta$ do cross the analyzer with maximum luminous intensity [16].

The wavelengths corresponding to two consecutive minima (λ_{ok} and λ_{ok+1}) and those corresponding to the maximum between them can be used to determine the birefringence Δn in the visible range (in which the polarizers are transparent) [17]:

$$\Delta n = \frac{1}{2L} \frac{\lambda_{0k+1/2}(\lambda_{0k} - \lambda_{0k+1})}{\lambda_{0k+1} - 2\lambda_{0k+1/2} + \lambda_{0k}} \quad (3)$$

In eq. (3), Δn is the birefringence ($\Delta n = n_e - n_o$), L is the thickness of the layer, λ_{ok} and λ_{ok+1} are the wavelengths corresponding to two consecutive minima of k and $k + 1$ orders, while $\lambda_{ok+1/2}$ is the wavelength of the maximum between them.

Results and discussions

The bulk chemical analyses of the investigated samples (table 1) revealed a constantly high participation of silica (99.01-100.00% SiO_2). Among the other oxides, iron oxide is the only one with remarkable contents (0.142% FeO), derived from iron oxide micro-inclusions finely disseminated in the quartz grains, which are visible under binocular lenses.

The minor element distribution (table 2) within the quartz samples indicates the presence of some rather important contents of Ba (13.8 - 14 $\mu\text{g}\cdot\text{g}^{-1}$ Ba), Rb (3.8 - 6.4 $\mu\text{g}\cdot\text{g}^{-1}$ Rb) and Sr (1.3 - 2.5 $\mu\text{g}\cdot\text{g}^{-1}$ Sr) which suggests the crystallisation of the respective samples (Q-16 and Q-61) from an alkali- and alkaline-earth-rich fluid. Comparatively, the

Oxides (%)	Samples						
	(Q-16) ¹	(Q-81) ²	(Q-81A) ²	(Q-95) ²	(Q-95A) ²	(Q-96) ²	(Q-96A) ²
SiO_2	99.88	100.00	99.49	99.01	100.00	99.43	99.96
TiO_2	0.01	0	0.021	0	0	0	0
Al_2O_3	0.03	0	0.026	0.017	0.011	0.012	0.019
FeO	0.116*	0.007*	0.033*	0.142*	0	0.054*	0.089*
MgO	0	0	0	0	0	0	0
MnO	0	0.021	0.003	0.066	0	0.019	0.029
CaO	0.03	0.003	0	0	0.002	0.009	0
Na_2O	0.016	0	0.022	0	0.006	0	0
K_2O	0.019	0.015	0.006	0.003	0	0	0.003
P_2O_5	0	0	0	0.03	0.029	0	0.022

* FeO as total iron; ¹ XRF analysis; ² EMP analysis; (Q-16) - feldspar pegmatite with muscovite and biotite; (Q-81), (Q-81A) - feldspar pegmatite with muscovite; (Q-95), (Q-95A), (Q-96), (Q-96A) - albite-spodumene pegmatite with phosphates

Table 1
CHEMICAL ANALYSES (wt. %) OF
QUARTZ SAMPLES

Table 2
MINOR AND TRACE ELEMENTS CONTENTS ($\mu\text{g}\cdot\text{g}^{-1}$) OF QUARTZ SAMPLES

Minor elements ($\mu\text{g}\cdot\text{g}^{-1}$)	Samples		
	(Q-16)	(Q-60)	(Q-61)
Co ¹	0	-	-
Ni ¹	0	-	-
Cr ¹	0.51	-	-
V ¹	0.13	-	-
Zn ¹	0	-	-
Li	3.2	2.5	2.2
Sc	0.3	0	0
Rb	6.4	0.2	3.8
Sr	2.5	1.1	1.3
Y	0.013	0.0	0.1
Zr	0.266	0.2	0.2
Nb	0.49	0.0	0.5
Cs	0.6	0.0	0.3
Ba	13.8	1.0	14
La	0.014	0.019	0.757
Ce	0.019	0.037	1.558
Pr	0.003	0.004	0.175
Nd	0.035	0.017	0.691
Sm	0.006	0.003	0.127
Eu	0.004	0.002	0.015
Gd	0.004	0.003	0.099
Tb	0.001	0	0.011
Dy	0.011	0.003	0.045
Ho	0.001	0.001	0.006
Er	0.002	0.001	0.009
Tm	0.001	0	0.001
Yb	0.006	0.002	0.005
Lu	0.001	0	0.001
Hf	0.015	0.005	0.007
Ta	0.03	0.005	0.029
Pb	0.4	0.3	0.4
Th	0.192	0.009	0.337
U	0.067	0.002	0.025

¹ -undetermined; ¹ XRF analysis; all the other analyses are ICP-MS;
(Q-16) - quartz from feldspar pegmatite with muscovite and biotite;
(Q-60) - quartz associated with black tourmaline in metamorphic vein;
(Q-61) - quartz from albite-spodumene pegmatite from the marginal zone.

metamorphic quartz sample (Q-60) has a much more pure composition, displaying only a low Li content ($2.5 \mu\text{g}\cdot\text{g}^{-1}$ Li).

The lanthanide rare earth elements (REE) are generally in the 3⁺ valence state over a wide range of oxygen fugacity (f_{O_2}), except for Ce that can be partly or entirely in the 4⁺ state at the oxygen fugacity of the Earth's surface and Eu which can be partly in the 2⁺ state at the low oxygen

fugacity of the Earth's interior. Because of their charge and large radii, the rare earths are incompatible elements, showing different degrees of incompatibility. Light rare earth elements (LREE) have greater ionic radii and therefore are highly incompatible with the crystalline lattice of almost all minerals - that is why they generally accumulate in the residual liquid phase of the fluid systems, concentrating within late minerals such as quartz, feldspars or micas. Heavy rare earth elements (HREE) have ionic radii small enough to be accepted to a certain degree within the structure of ferromagnesian minerals- they generally concentrate in the early solid phase, the residual liquid being thus depleted in these elements. In the investigated samples, scarce REE amounts are present, but still differentiated according to the rock type, showing that even the generally very pure quartz is influenced by different crystallization conditions.

In the REE chondrite normalized plot (fig. 4) it can be seen a clear differentiation of the quartz belonging to the albite-spodumene pegmatites (sample Q-61), which is more enriched in REE due to the crystallization from a late alkaline-earth-rich fluid, known to have had generated the

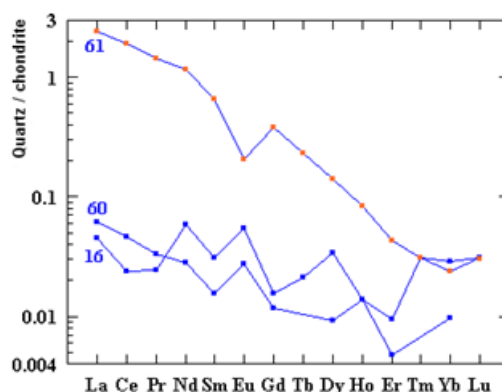


Fig. 4. REE chondrite-normalized plot of quartz samples (samples description as in table 2)

fertile pegmatites, *i.e.*, the spodumene association pegmatites from Contu-Negovanu, unique in the Romanian geologic record.

The birefringence at $\lambda_0 = 589.3 \text{ nm}$ was measured using both a Babinet compensator and a Rayleigh interferometer. The measured refractive indices are shown in table 3 for four samples.

From table 3 it results that the investigated quartz samples are uniaxial positive with linear birefringence of a mean value $\Delta n = 0.0091$ at $\lambda_0 = 589.3 \text{ nm}$. The obtained

Table 3
MAIN REFRACTIVE INDICES AND BIREFRINGENCE OF SOME QUARTZ SAMPLES ($\lambda_0 = 589.3 \text{ nm}$) MEASURED WITH RAYLEIGH INTERFEROMETER AND BABINET COMPENSATOR

Sample (Method)	n_o	n_e	Δn
Q-81 (Rayleigh)	1.5442	1.5534	0.0092
Q-81 A (Rayleigh)	1.5441	1.5532	0.0091
Q-95 (Rayleigh)	1.5441	1.5531	0.0090
Q-95 A (Rayleigh)	1.5442	1.5531	0.0089
Q-81 (Babinet)	1.5443	1.5533	0.0090
Q-81 A (Babinet)	1.5442	1.5530	0.0088
Q-95 (Babinet)	1.5442	1.5533	0.0091
Q-95 A (Babinet)	1.5441	1.5531	0.0090

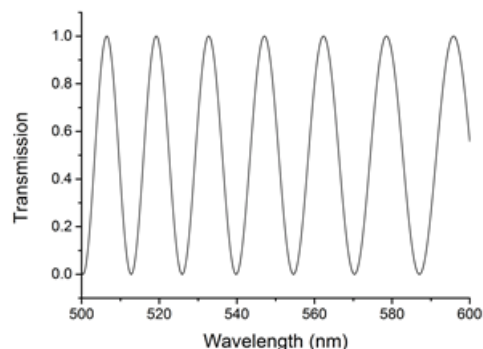


Fig. 5. Channeled spectrum obtained with quartz sample plate being cut parallel to the optical axis of thickness $L = 1$ mm

results are consistent with those from literature ($n_e = 1.553$, $n_o = 1.544$ and $\Delta n = 0.0090$ at $\lambda_0 = 589.3$ nm) [18].

The results obtained by the new method [17] using the channeled spectrum of a quartz plate being cut parallel to optical axis are given below (fig. 5).

In figure 5 a channeled spectrum obtained for a quartz plate being cut parallel to its optical axis and having the thickness $L = 1$ mm is illustrated. From this spectrum the wavelengths which appear in relations (3) and (4) were measured and then, the birefringence and its dispersion were evaluated. The dependence of Δn on the wavenumber is given in figure 6. As it results from figure 6, the linear birefringence of the studied quartz plate decreases when the visible wavelength increases.

From figure 3 and table 4 it results a good correlation between the literature and the measurements obtained in this study.

The method of channeled spectrum has the advantage to offer information about the linear birefringence and its dispersion in the entire visible range.

The knowledge on the quartz birefringence in visible range can be used in designing optical devices such as interferometric filters [19,20].

Conclusions

The quartz samples belonging to the Contu-Negovanu granitic pegmatites are chemically very pure, showing an expected high participation of silica (99.01 - 100.00 % SiO_2) and only some remarkable contents of Fe (0.142 % FeO), derived from iron oxide micro-inclusions finely disseminated in the quartz grains. Still, the minor and trace elements distribution reveals that different crystallization conditions influenced the quartz composition, *i.e.* from alkali- and alkaline-earth-rich fluids resulted some rather high contents of Ba (13.8 - 14 $\mu\text{g}\cdot\text{g}^{-1}$), Rb (3.8 - 6.4 $\mu\text{g}\cdot\text{g}^{-1}$) and Sr (1.3 - 2.5 $\mu\text{g}\cdot\text{g}^{-1}$) in the pegmatite quartz (samples Q-16 and Q-61), whilst the metamorphic quartz is highly pure, showing no such presence in the respective sample (Q-60). The distinctly higher REE contents (1.558 $\mu\text{g}\cdot\text{g}^{-1}$ Ce) in the Q-61 sample belonging to the albite-spodumene pegmatites confirm the *fertile* character of the initial fluid, which generated the presence of the spodumene mineral association in these pegmatites, unique in Romania.

The analyzed quartz samples present birefringence parameters within the limits of other quartz samples and a decrease of the linear birefringence of quartz with the light wavelength increase is emphasized by the data obtained using the channeled spectrum.

When the main refractive indices and birefringence of anisotropic layers such as those of quartz are known, some optical devices working in polarized light can be designed.

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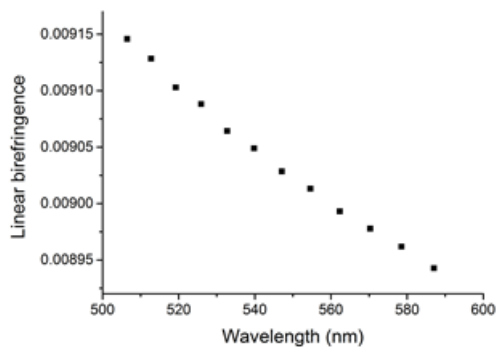


Fig. 6. Linear birefringence of quartz from granitic rocks

Table 4
LINEAR BIREFRINGENCE OF QUARTZ FROM GRANITIC ROCKS, FROM LITERATURE AND EXPERIMENTALLY DETERMINED IN THIS PAPER, RESPECTIVELY

Nr. crt.	Wavelength [nm]	Δn from literature [18]	Δn experimentally obtained by channeled spectra method
1	508	0.00924	0.00912
2	598.3	0.00911	0.00890

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