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LABORATORY MINERAL PROCESSING OF SAMPLES OF THE WEST CARPATHIAN GRANITOID ROCKS

Introduction

Within the framework of geochemical research of the West Carpathian granitoid rocks which was carried out at the Geological Institute of the Slovak Academy of Sciences in Bratislava, concentrates of heavy accessory minerals and monomineral products of K-feldspars, plagioclases, biotite, muscovite and quartz were needful to obtain from these rocks for the following purposes:

1. examination of physical properties of minerals (morphology, optical properties, density, magnetic susceptibility etc.);
2. examination of chemical properties of minerals (fundamental chemical composition, content of trace elements, content of the naturally radioactive elements U, Th, K);
3. estimating geological age of the rocks by radiometric methods.

For these purposes over 100 samples were tested, which in literature are known as the "ZK"-samples and they represent the fundamental types of the granitoid rocks from the West Carpathian region. These samples would be used for the next comparative research. Concerning this fact we consider necessary to describe a basic information on the used laboratory mineral processing method of samples which influences the quality of monomineral product and concentrate of the heavy accessory minerals.

Working process

The laboratory mineral processing of samples can be divided into:

1. sampling, reducing and parting of the sample,
2. gravity concentration on the shaking table,
3. preparing the collective concentrate of heavy accessory minerals and its separation into groups according to the density and magnetic susceptibility,
4. preparing monomineral products of K-feldspars, plagioclases and quartz,
5. preparing monomineral products of biotite and muscovite.

1. Sampling, reducing and parting of the sample

Location of sampling was published by J. Macek et al. (1979), process of collecting, reducing and parting of samples by J. Veselský — M. Žabka (1976). Just mention the fact that every fundamental sample has its weight 15 kg at least and consists of about 100 partial samples.

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2. Gravity separation on the shaking table

A part of sample whose weight is about 10 kg, screened below 0.5 mm is sorted into 2 classes: 0–0.25 mm and 0.25–0.5 mm. Each class is separated individually on laboratory side-slope shaking table at these parameters:

speed	– 400 r. p. m.
angle of the deck slope	– 12°
water consumption	– 0.55 m ³ /hour.

3 control separations are used. The individual final products from the shaking table are the fundamental material for the next separation as follows:

- ST I (heavy product) – of heavy accessory minerals,
- ST II (middle product) – of K-feldspars and quartz,
- ST III (light product) – of biotite and muscovite.

3. Process of obtaining heavy accessory mineral concentrates

The original variant of the method to obtain a concentrate of heavy accessory minerals was described by J. Veselský – M. Žabka (1976). By the further improvement the following was reached:

The fundamental material is the product designated as ST I products for classes less than 0.25 mm and 0.25 mm–0.50 mm. From these products iron filings and ferromagnetic minerals are removed by a. c. hand-magnet under wet conditions which prevent from the oxidation of some minerals (e. g. pyrrhotite) and formation of Fe hydrates, originated especially from crusher and mill filings, would contaminate surfaces of other minerals. Its products contain many mica minerals, they must be removed by flotation in the modified Hallimond tube using the collector ANP – 1 at pH 2.5–3.0 (sulphuric acid). Floated product is washed using the ultra-sound, dried and it is checked by the help of binocular microscope, whether it does not contain pulled heavy accessory minerals. Non-floated product, after washing by use of ultra-sound, is further separated by the sink-and-float method in the organic heavy liquids. This separation consists of two stages. In the first one bromoform is used as distributing medium whose density is nearly 2.8, in the second one (in which feed consists of heavy product from the previous separation) methylene iodide whose density is nearly 3.4. After separation in methylene iodide, both the products are individually separated with the Cook's Isodynamic Magnetic Separator. Side-slope of the vibrating chute is 11°, forward slope 20°. The operating d. c. increases gradually in the range 0.2–2.5 A, while feed of the next separation consists of non-magnetic product from the previous separation. These operating d. c. values were used: 0.2 A; 0.4 A; 0.6 A; 1.0 A; 1.5 A; 2.0 A. By the above mentioned process the accessory minerals are obtained, they are sorted into groups according to their density and according to their magnetic properties.

4. Process of obtaining monomineral product of K-feldspars and plagioclases

The material, designated as ST II is appropriate for preparing monomineral products both, the K-feldspars and plagioclases. This material (in class less than

0.25 mm) is separated under dry conditions in the high intensity magnetic separator of any type. If the Kaldrovitš type is used, appropriate supply is 40 A. Non-magnetic product is distributed into classes:

less than 0.073 mm, 0.073–0.10 mm, 0.10–0.16 mm, 0.16–0.25 mm. For separation of plagioclases of the West Carpathian granitoid rocks the appropriate class seems to be in the range 0.10–0.16 mm. For removing the mica impurities nearly 150 g of the non-magnetic product of this class, flotation is used. Flotation parameters are as follows:

collector — ANP-1

frother — Dow-froth (1 drop/1 liter),

pH — 2.5–4.0 (sulphuric acid).

Flotation tailing have to be perfectly washed by water and after drying are further separated in the Cook's Isodynamic Magnetic Separator whose side-slope of the chute is 11°, its forward-slope of 25°, operating current gradually in the range 0.5–3.0 A.

In this way obtained finished non-magnetic product is washed by water using the ultra-sound 2-times for 2 minutes. After the slime class decantation it is sedimented and after drying at 40 °C it is an appropriate material for study of secondary minerals from the feldspars by the help of electron microscope and X-ray analyzer. After decantation, the sediment consists of feldspars and quartz especially.

The next operation is cleaning of this product in HF solution (pH = 2–2.5) removing the slime and drying the sediment.

For separation of K-feldspars and their intergrowths from quartz and plagioclases, the sink-and-float method is carried out in the heavy organic liquids with density 2.61 (diluted bromoform) in the used separating funnels, in which the heavy product is first removed after finished sinking of minerals (so called lower removal). This separation is efficient in a set of separatory funnels with common removals, both for heavy and washing liquids from all funnels respectively.

4 a) Preparing monomineral product of K-feldspars

From the light product (density less than 2.61) after drying, the monomineral product of K-feldspars is obtained by further sink-and-float method in the special separatory flasks described by J. Macek — M. Žabka — L. Viliňová (1980), from which the light product is separated at first (so called upper removal) in the separatory medium, the density is 2.584 and when cleaning separation of the light fraction, the density is 2.580. Providing these operations, the most important is to adhere to the respective values of densities of heavy liquid during the whole separation process.

The described methods of preparing monomineral product of K-feldspars enables to obtain sufficient quantity of the K-feldspar monomineral product also among samples which are relatively poor in K-feldspars.

4 b) Preparing monomineral product of plagioclases

Plagioclases are separated from the product whose specific gravity is more than 2.61 from the previous separation by flotation. As a collector dodecylamine

or octadecylamine in the acetate form are appropriate, as the frother the Dow-froth. Flotation is carried out in the acid medium ($\text{pH} = 2.5\text{--}3.5$) caused by the HF acid. It is necessary to condition the feed with the water solution of HF ($\text{pH} = 1.8\text{--}2.5$) for about 4 min. before the collector is added. Collector at flotation cannot be overdosed. Flotation time-factor depends on yield of the froth product quantity according to possibilities it is usually short. Separation effect can be vanished even by a little contamination of the complex-forming reagents (e. g. natrium metaphosphate), by presence of the dust class, substantially lowered by kaolinization or sericitization of plagioclases (Ney P., 1973).

Froth product is thoroughly washed in HF solution ($\text{pH} = 2.5$) and 1–2 cleaning flotations are carried out under the same conditions. In this process it is necessary to avoid that the grains of quartz would be in contact with collector without the HF for in that case also quartz would be floating.

The reached flotation concentrate of plagioclases is further separated in heavy liquids whose density is 2.62. Separatory funnels can be used which enable the "lower removal" of the heavy product. The light product (density less than 2.62) consists of alkalic plagioclases and their intergrowths, the heavy product (density more than 2.62) consists of plagioclases, and impurities of micas. By following separation in the heavy liquid (density = 2.68) in flasks, enabling the "upper removal" light product (specific gravity less than 2.68) is obtained, which consists of plagioclases.

5 a) Preparing monomineral product of biotite

The fundamental material for obtaining monomineral product of biotite is the material, designated as ST III in the class 0.—0.25 mm or 0.25–0.5 mm. This product is separated in the respective class in the high-intensite magnetic field separator of any type. In the case of using the Kaldrovitš type, the operating d. c. 10 A is appropriate. The magnetic product contain about 85 % of biotite.

The biotite pre-concentrate obtained this way is further separated by flotation under following conditions:

collector — penthylamine or hexylamine,

frother — the Dow-froth

pH — 9–11 (NaOH or KOH).

The froth product at non-altered rocks includes about 95 % of biotite. After the cleaning flotation of froth product under the same conditions but with lower dosing of collector and frother, the content of biotite is increased in the froth product usually to 98 %.

After washing of the product by using the ultra-sound and after drying, this biotite pre-concentrate is cleaned in the Cook's Isodynamic Magnetic Separator under these conditions:

sides-slope of the chute — 11°

forward-slope of the chute — 25°

operating d. c. — 1st separ.: 0.4 A,
2nd separ.: 0.45 A.

During the first separation biotite is directing in the non-magnetic product, during the second one — in the magnetic product. The values of operating d. c. can be somewhat different according to the type of samples.

5 b) Preparing monomineral product of muscovite

The fundamental material for separation of muscovite is the non-magnetic product from the high-intensity magnetic field separator (Kaldrovitš — 10 A). This product can be separated again in the respective class in the same separator but under higher operating d. c. so that muscovite would direct in the magnetic product. When the Kaldrovitš type is used, the 45 A of operating d. c. is appropriate. The obtained muscovite pre-concentrate is further separated by flotation under these conditions:

collector: ANP 1 (or other amines with the open long chain in the form of acetates, about 50 mg/l),

frother: the Dow-froth or pine oil (2 drops/l),

pH: 2.0—2.5 (sulphuric acid),

activator: lead nitrate (2 drops/l).

The froth product needs to be washed well using the ultra-sound, after decantation and drying it is cleaned with the Cook's Isodynamic Magnetic Separator with operating d. c.:

first separation — 1.6 A,

second separation — 2.2—2.5 A (the side-slope and the forward slope of the chute as at separation of biotite). Magnetic separation is repeated according to need 2—3-times. At these repeated separations the difference of operating d. c. values is decreasing. In the case that in the muscovite concentrate still feldspars or other minerals are found, whose susceptibility is owing to impurities the same as the muscovite susceptibility in the given sample, they can be removed by the sink-and-float method in heavy liquid (diluted methylene iodide).

Discussion and conclusion

Purity of concentrate of the heavy accessory minerals was controlled optically by the help of binocular microscope. Impurities are formed by dark rock-forming minerals, especially biotite. The reached purity is sufficient for further mineralogical tests. The loss of heavy accessory minerals with using the shaking table is lower than when using the gold pan or batea. When calculating weight of the individual heavy minerals to a ton of feed, we can count with about 30 % loss.

Preparing the monomineral products of some heavy minerals is beyond the framework of this paper. A detailed description of obtaining the monomineral products of zircon is mentioned in M. Žabka — N. Ju. Levkovskaya — G. Ja. Terec (1977).

Purity of monomineral product of K-feldspars was controlled optically with binocular microscope after previous colouring by sodium hexanitrocobaltitanate and by X-ray diffraction method. In some cases it reached 97 %, in majority of cases 98 or 99 %.

Purity of monomineral product of biotite and muscovite reached up to 99 %, checked with binocular microscope. Impurities at biotite are mostly formed by chlorites, at muscovite by inclusions of different minerals.

The obtained clean monomineral products were used for geochemical study (classical chemical analysis, study of trace and rare elements) and for other

reasons. Monomineral products of biotites for estimating of the geological age by the K/Ar method need to be prepared from the higher classes (0.5–1.0 mm).

Complex geochemical examination of the West Carpathian granitoid rocks largely uses monomineral products of rock-forming minerals and concentrates of heavy accessory minerals from these rocks. Within the framework of this research more than 100 samples were treated, which could be useful for further comparative research.

For obtaining any comparative results it is necessary to use the uniform laboratory mineral processing methods of preparing the monomineral products and concentrate of the heavy accessory minerals, because this influence their quality.

Conclusion

In the paper the results of the experimental works are summarized, which the author carried out for elaboration of processes for obtaining the monomineral products of rock-forming minerals (K-feldspars, plagioclases, biotite and muscovite) and concentrate of the heavy accessory minerals from the West Carpathian granitoid rocks. By the described mineral processing methods more than 100 samples were treated, in literature known as the "ZK" samples, the monomineral products were of a high purity.

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