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Gravimetric Determination of Silica in Anion Exchanges

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There are many methods for cleaning productive solutions of underground uranium leaching from both organic matter and silica. At the same time, as previously indicated, the organic matter contained in the productive solutions actively sorbs silica, and it is possible that cleaning will have to be carried out from both components in a complex. At the same time, the method of joint purification is not particularly advantageous, since organic matter, due to its intensifying effect on the process of underground leaching of uranium, is most expediently removed from productive solutions before the sorption process and then returned to the recycled sorption mother liquors, while silicon should be removed irreversibly.

It is obvious that in cases where the resulting silicon-containing organic matter is returned to the recycled leaching solutions, the problem of its desiliconization will arise. Taking into account the above, this section first examines the issues of separate purification of productive solutions of underground leaching of uranium from organic substances and silica, and then examines methods of joint purification of productive solutions from organic substances and silica with subsequent desilication of organic matter [1].

Carrying out measurements of the mass fraction of silicon dioxide

Measurement by the gravimetric method in anion exchangers

This measurement procedure applies to in-situ leaching process solutions and establishes a gravimetric method for measuring the mass concentration of silicon dioxide in the range from 2.0 to 1000,0 mg/dm^3.

The method for measuring the mass concentration of silicon dioxide in process solutions is photometric. It is based on measuring the optical density of the reduced silicon-molybdenum complex, colored blue. The measurement process consists of the following operations [2]:

- decomposition of the anionite by "wet" combustion in a mixture of sulfuric, nitric and perchloric acids;
- filtration of the decomposed anionite;
- washing of the silicon oxide precipitate on the filter from sulfate ions;
- calcination of the silicon oxide precipitate;
- distillation of silicon in the form of silicon fluoride;
- calcination of the residue at 900°C taking into account losses during calcination.

At least two parallel determinations are performed for each measurement.

Performing measurements

When measuring the mass fraction of silicon dioxide in anion exchangers, the following operations are performed:

A sample of anionite (hereinafter referred to as resin), weighing 1.0 g, taken with an error of ±0.0002 g, is placed in a dry heat-resistant flask with a capacity of 250 sm^3, (15-20) sm^3 of sulfuric acid with a density of (1.82 ÷ 1.84) g/ sm^3 is added and heated on an electric hotplate until the resin is completely

carbonized for (30-40) min. Then the flask with the sample is cooled to (70-80)°C and 5 sm^3 of nitric acid with a density of (1.34÷1.38) g/sm^3 and 5 sm^3 of perchloric acid are carefully added along the wall of the flask, and heated again until abundant release of sulfuric acid vapors. If the solution turns dark, another (2-5) sm^3 of perchloric acid is added and heated again until abundant release of sulfuric acid vapors and boiled for another (15-20) minutes. Next, the flask with the sample is cooled, the walls of the flask are washed with distilled water from a wash bottle - the total volume of water should not exceed 10 sm^3 and heated again on an electric stove until sulfuric acid vapor is released. After cooling the flask, carefully add (40-50) sm^3 of water, heat to boiling, cool slightly and filter through a “blue ribbon” filter with paper pulp, into a measuring flask with a capacity of 250 sm^3 (if it is necessary to determine calcium, magnesium, iron, aluminum, molybdenum) or into a glass (for draining into the sewer) [3].

The silicon dioxide precipitate on the filter is washed with hot water until there are no sulfate ions (filtrate sample with barium chloride solution). Note - For this, (2-3) sm^3 of the filtrate are collected in a test tube and (2-3) drops of barium chloride solution are added. The absence of turbidity indicates that the precipitate has been completely washed from sulfate ions. The filter is dried on a funnel, placed in a platinum crucible, placed in a cold muffle furnace, the temperature is gradually increased to (900-1000°C) and calcined at this temperature for 1 hour. Then the crucible is removed from the muffle furnace [4].

The precipitate in the crucible is moistened with a solution of sulfuric acid diluted 1:1, (10-20) sm^3 of hydrofluoric acid is added, evaporated to dryness and then placed in a heated muffle furnace. Calcinated at a temperature of 900°C for (15-20) min. Cooled, weighed and the content of silicon dioxide is determined by the difference in weights. At the same time, throughout the entire course of the analysis, a "blank" experiment is carried out.

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