DETERMINATION OF WATER CONTENT IN GLASSES AND MINERALS USING THE HIGH TEMPERATURE SETUP BASED ON COULONOMETRIC SENSORS (development of the method of the determination)

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The aim of the study is to develop of the method of determination of water content in natural and experimental glasses and minerals using the high temperature setup based on coulonometric sensors. The apparatus was made earlier by authors, and was described in [1].

The principle of the process is the following. The sample is subjected to the pyrolitic decomposition in the high temperature reactor in the flow of inert gas (Ar). The evolving water enters to the sensitive element of the coulonometric cell. The sensitive element of the cell consists of a pair of platinum electrodes that is coated with phosphorus pentoxide. The gaseous water passing through the cell is continuously absorbed by phosphorus pentoxide with the phosphoric acid formation and electrolyzed into molecular hydrogen and oxygen, accompanied by a transfer of charge from one electrode to another. **C**ollection and treatment of the received information is performed on the data collection device L-154 of the analogy digital converter (ADC) installed in computer. During the experiment the evolving water is registrated by ADC in the form of an amperometric curve (current versus time). The peaks area corresponds to the extracted water quantity. The software (program VAM) [2] allows to calculate the water quantity in micrograms.

There is a problem of adequate standards containing trace amounts of water. Therefore we have to use available reference samples for our preliminary measurements: pyrophillite ($Al_4[Si_8O_{20}]$ $(OH)_4$) with known stechiometric water content (5 wt. % H_2O) and rhyolite with water content nearly 0.19 wt. %. To make tentative estimates of the method possibilities distilled water was used.

The operating sequence is as follows: heating of the setup; injection of the inert gas into the system; attainment of the background water content in the inert gas flow; ignition of the quartz or platinum boat in the reactor; weighing the sample; insertion of the boat containing the sample into the reactor; measurement of the evolving water. At first the boat with the sample is inserted into the 200 ^oC - zone of the reactor to evolve the absorbed water (fig.1, peak 1).

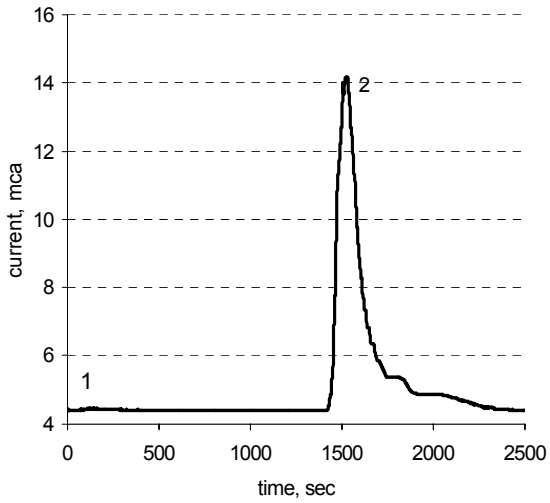

Fig.1. The amperometric curve of the water evolution from pyrophillite
1 – peak of the absorbed water evolution (200 ^oC); 2 – peak of the chemically bonded water evolution (700 ^oC); a weighed sample of pyrophillite = 3,8 mg, $H_2O_{theor.}$=0.19 mg

After the attainment of the background curve the boat containing the sample is moved into the reactor zone with the temperature sufficient for full evolving of water from the sample. For pyrophillite the temperature was 700^oC, and the rhyolite was heated up to 1200^oC. The studies with distilled water were performed at 200^oC. Peak 2 (fig. 1) correlates with the chemically bonded water evolving from pyrophillite. Preliminary results are presented (fig. 2).

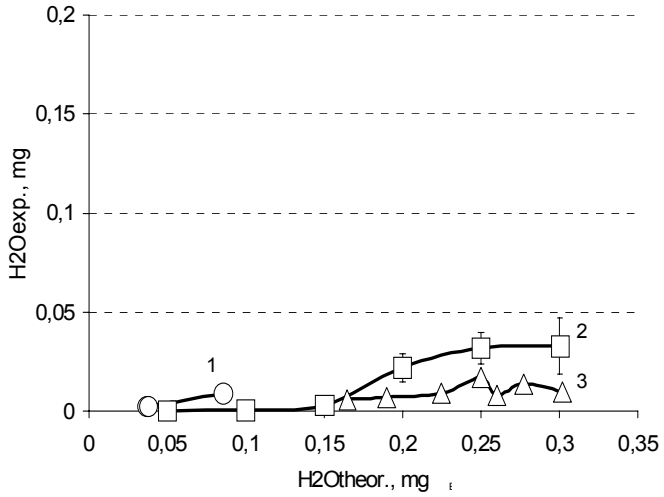

Fig.2. Results of the water content measurements 1 – rhyolite; 2 – distilled water; 3 – pyrophyllite

The results show that experimentally measured water content in the sample within the all water content range (from 0.03 to 0.3 mg) studied is one order of magnitude less than theoretical one for all samples. It also takes place in the water content interval < 0.1 mg, though this interval is considered to be optimum for operation of the coulonometric cell. The reasons of the decrease cell sensitivity in the measured range of the water content are not ascertained.
Thus it is necessary to improve the method, to find different factors which influence on the measurements results (the rate of the sample heating, velocity of the gas flow, adequate standards containing trace amounts of water etc.).

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