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## Laboratory modeling and measurement of the electrical resistivity of hydrate-bearing sand samples

M.E. Permyakov<sup>a</sup>, N.A. Manchenko<sup>a</sup>, A.D. Duchkov<sup>a</sup>, A.Yu. Manakov <sup>b,\*</sup>, A.N. Drobchik<sup>a</sup>, A.K. Manshtein<sup>a</sup>

<sup>a</sup>*A.A. Trofimuk Institute of Petroleum Geology and Geophysics, Siberian Branch of the Russian Academy of Sciences,*

*pr. Akademika Koptyuga 3, Novosibirsk, 630090, Russia* <sup>b</sup> *A.V. Nikolaev Institute of Inorganic Chemistry, Siberian Branch of the Russian Academy of Sciences,* 

*pr. Akademika Lavrentieva 3, Novosibirsk, 630090, Russia*

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#### **Abstract**

In this paper, we describe a setup for modeling hydrate-bearing rock samples and measuring their electrical resistivity at different pressures and temperatures using an AMNB cylindrical four-electrode probe. Methods for modeling hydrate-bearing rock samples and measuring their resistivity are considered. The setup was used in a series of experiments to measure the resistivity of sand samples containing water, ice or tetrahydrofuran (THF) hydrate. It is shown that when the rock pores contain hydrates and a partially unfrozen aqueous solution of NaCl and THF, the electrical resistivity is determined by the high resistivity of the solution and increases with the formation of hydrate. The presence of THF hydrate in the experimental samples increased their resistivity by 180–320 Ohm⋅m at a temperature of about 0 °C. After the formation of hydrate and freezing of residual water, the resistivity of the sample is stabilized at 70–80 Ohm⋅m at a temperature of –15 °C. © 2017, V.S. Sobolev IGM, Siberian Branch of the RAS. Published by Elsevier B.V. All rights reserved.

*Keywords:* tetrahydrofuran; tetrahydrofuran hydrates; modeling of hydrate-bearing samples; electrical resistivity

#### **Introduction**

Sub-bottom (submarine) accumulations of natural gas hydrates are common in deep waters and are usually associated with active underwater gas-fluid sources located at depths greater than 300–500 m (Istomin and Yakushev, 1992; Ginsburg and Solov'ev, 1994; Mazurenko and Soloviev, 2003). In the sediments present near such sources, there are necessary conditions for the formation and existence of natural gas hydrates: high pressure, low temperature, the presence of water and free gas (or a sufficient concentration of gas dissolved in water). Note that in the last decade, more than 50 accumulations of methane hydrates were found in sub-bottom sediments of Lake Baikal and the Sea of Okhotsk (Khlystov, 2006; Obzhirov et al., 2012), confined to underwater discharges of gas-containing fluids. There is little doubt that hydrate sediments will be found in the Arctic marginal seas in the coming years.

Interest in accumulations of natural hydrates is primarily associated with their energy potential. According to some estimates, overall they contain up to  $10^{14}$  m<sup>3</sup> methane (Soloviev, 2002). Gas hydrate accumulations have a significant effect on the stability of structures installed on the seabed, subsea wells, etc.

Currently, considerable efforts are being directed to find and explore accumulations and deposits of natural gas hydrates in bottom sediments of water bodies using drilling, bottom sampling with coring tubes, and geophysical (mainly electromagnetic) methods (Schwalenberg et al., 2008; Weitemeyer and Constable, 2010; Weitemeyer et al., 2005). Calibration (verification) of remote geophysical methods of prospecting for gas hydrate accumulations requires investigation of the physical properties of hydrate-bearing rocks. The study of the physical properties of rocks containing hydrates also provides an insight into the mechanisms of formation and decomposition of gas hydrates in the Earth's crust and promotes the development of realistic models of gas hydrate accumulation in various geological settings.

Typically, the physical properties of hydrate-bearing rocks are studied under laboratory conditions using complex setups

Corresponding author.

*E-mail address*: manakov@niic.nsc.ru (A.Yu. Manakov)

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first to simulate hydrate-bearing samples (HBS) in high-pressure chambers and then to measure their characteristics. The physical properties of hydrate-bearing rocks are most extensively studied in countries where national gas hydrate programs are being carried out with the ultimate goal of industrial production of natural gas from gas hydrate accumulations (U.S., Canada, Japan, China). Detailed reviews of foreign studies in these areas are contained in (Gabito and Tsouris, 2010; Riedel et al., 2010; Waite et al., 2009). Judging by the publications, an enormous amount of experimental work has been devoted to the study of the electrical resistivity (ER) of hydrate-bearing marine sediments, i.e., sediments saturated with salt water with a resistivity of about 0.2–0.3 Ohm⋅m. Unconsolidated marine sediments in the upper few hundred meters (porosity of 30–50%) usually have a low electrical resistivity of about 1–2 Ohm⋅m. Hydrate-bearing marine sediments have a higher resistivity since the resistivity of gas hydrates is comparable to that of ice. For example, at a 15–20% concentration of hydrates in pores (7–10% of the sediment), the rock resistivity can increase by a factor of 2–5 or more (Waite et al., 2009). These anomalies are clearly detected by controlled-source electromagnetic (CSEM) sounding (Schwalenberg et al., 2008).

Analysis of the literature shows that the electrical properties of the hydrate-bearing freshwater sediments have been little studied. For Russia, such studies have become relevant after the discovery of hydrate accumulations in the freshwater sediments of Lake Baikal at the end of the last century (Khlystov, 2006). So far, only the coordinates of the accumulations are known. Their structure (lateral spread, thickness) remains to be clarified. Possibly, this can be done using marine modifications of electromagnetic methods. It is obvious that in the initial step, before remote geophysical work, it is reasonable to study the resistivity of the sub-bottom sediments of Lake Baikal, both containing or not containing methane hydrates, given that the resistivity of bottom water in the lake basins is 100–150 Ohm⋅m (Galazii, 1984). The study of the electrical resistivity of fresh-water hydrate-bearing sediments is a new task. To solve it, we developed a setup for modeling hydrate-bearing samples and measuring their resistivity. It is the purpose of this paper to describe the setup and the results of the first, mostly test experiments.

#### **Description of the setup**

The scientific literature presents a large number of setups for modeling HBS and their subsequent study. Equipment of this type is not industrially produced, so that each research team planning a laboratory study of HBS has to design its own equipment. The most common are experimental setups that allow modeling of HBS and measuring only one characteristic. Figure 1 shows a possible configuration of such a setup. Earlier, the same research team designed a setup in the same configuration for measuring the thermal conductivity of hydrate-bearing rocks (Duchkov et al., 2006, 2009).



Fig. 1. Setup for modeling hydrate-bearing samples and measuring their physical properties (Duchkov et al. 2006). 1, high pressure cylindrical chamber; 2, thermostat; 3, measuring system (cylindrical test probe and recording system); 4, PC; 5, discharge valve; 6, pressure gauge; 7, gas cylinder.

We used this configuration to design a setup for modeling hydrate-bearing sediments and measuring their electrical resistivity. The appearance of individual parts of the setup is shown in Fig. 2. The main unit is a cylindrical steel high-pressure chamber, in which a HBS is formed and the probe is placed (Fig. 2*a*)*.* The operating pressure (up to 10 MPa) in the chamber is produced by gas. The chamber has an inner diameter of 100 mm, a length of 250 mm, and a volume of about  $4 \text{ dm}^3$ . The top and bottom of the chamber are closed with shutters, which are sealed with rubber and fluoroplastic rings. The chamber is pin-jointed to a support and can be rotated about a horizontal axis for ease of washing and removal of samples. Gas (methane or  $CO<sub>2</sub>$ ) from a gas cylinder is fed to the chamber through a thin capillary tube soldered into the upper shutter using a pressure gauge and a vent valve. The shutters have drilled holes through which various gauges can be introduced into the chamber. The chamber is mounted on a textolite substrate for the purpose of electrical insulation.

Electrical resistivity was measured using an AMNB cylindrical four-electrode probe permanently located at the center of the chamber (sample) and a measuring system, which will be described below. To provide stable temperature conditions, the chamber is enclosed in a steel thin-walled jacket through which a thermostating liquid is circulated during the experiment. The thermostating liquid is polymethylsiloxane—a liquid with high electrical resistivity. Temperature control is achieved using a liquid thermostat consisting of a HAAKE EK20 refrigerator and a control unit—a HAAKE DC30 immersion circulator placed in a HAAKE B3 bath of stainless steel (Fig. 2*b*)*.*Temperature (*T*) in the chamber (on its outer wall and in the upper part of the sample) was measured by DS18B20 digital sensors with an accuracy of  $\pm$  0.1 °C.

As mentioned above, the experiment usually includes two separate operations: modeling hydrate-bearing (or frozen) samples and measuring their characteristics (in our case, electrical resistivity). Modeling artificial HBS corresponding to real geological objects is the most difficult of these operations. A large number of modeling techniques have been proposed, but a common approach has not yet been developed.

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