Mesoporous aluminosilicates form three different phases: a hexagonal phase, a cubic phase, and an unstable lamellar phase. Among them, mesoporous aluminosilicates with a cubic phase are widespread, while the other two are of limited use in catalysis [12, 13]. The synthesis of mesoporous aluminosilicates is based on the use of templates and their further removal by calcining at 450-600 °C. The use of nonionic surfactants as templates is of greatest interest for petrochemistry and oil refining. Materials synthesized on the basis of hexadecylamine possess the highest specific surface area (up to 1030 m²/g) compared with other widely used templates, for example, triblock copolymers of polyethylene and polypropylene oxide (only up to 750 m²/g) [14]. Another important issue in the synthesis of mesostructured aluminosilicates is the silicon to aluminum ratio (Si/Al). Karakhanov et al. [15] found that the most optimal ratio Si/Al = 10. Thus, mesoporous aluminosilicates with a high aluminum content possess higher specific surface area of 1030 m²/g, respectively), and also provide higher selectivity of n-dodecane hydroisomerization.

In this work, we present the results of a study of the effect of the mesoporous aluminosilicates' synthesis method on their physicochemical characteristics.

Experimental part. Samples of mesoporous aluminosilicates for the synthesis of which hexadecylamine was used as a structure-forming template were prepared by the authors. The synthesis of mesostructured aluminosilicate was based on the method of copolycondensation of tetraethylorthosilicate Si(OC_2H_5)₄ and secondary aluminum butoxide (sec-BuO)₃Al/aluminum triisopropoxide Al(i-OPr)₃ in the presence of alcohol. For this, the sources of aluminum were dissolved in alcohol. Then a solution of tetraethylorthosilicate was added dropwise to them under intensive stirring. In parallel, while heating a solution of hexadecylamine with alcohol was prepared. After complete dissolution of the template, distilled water was added to the solution. A solution of silicon and aluminum compounds mixture was added portionwise to the resulting viscous homogeneous mass containing the template solution. Further, after adding water dropwise and with stirring for 10 minutes, the resulting mixture was stirred for 6 hours at room temperature. Then it was kept without stirring at room temperature for a day. Next, the precipitate was filtered on a Buchner funnel, dried at room temperature for 24 hours, and then slowly heated in an oven to 110 °C during 3 hours. The resulting solid-phase substance was placed in a tubular furnace and heated to 600 °C at a heating rate of 1 °C/min, after which the sample was calcined at this temperature for 6 hours in an oxygen flow.

The porous structure of the synthesized samples and the values of the BET surface area were studied using the standard nitrogen adsorption/desorption method on a Quanta Chrome Autosorb-6 sorptometer.

The ordering of the porous structure of the synthesized materials was analyzed using the method of small-angle X-ray scattering. X-ray diffraction patterns were recorded on a Rigaku D/MAX 2200 diffractometer with a Cu K radiation source.

Determination of Lewis/Brønsted acid sites in the synthesized catalysts was carried out using experiments of diffuse reflectance infrared Fourier transform spectroscopy (DRIFT) of adsorbed pyridine using a PerkinElmer Spectrum One FTIR-spectrometer. Before these tests, the samples were pretreated at 110 °C to remove water adsorbed on acid sites. After adsorption of pyridine, the samples were dried at 40 °C before DRIFT analysis. The background spectrum was recorded using KBr. The spectra obtained after pyridine desorption were subtracted from those measured before pyridine adsorption (fresh samples) in order to determine the bands relative to the Lewis and Brønsted acid sites.

The IR spectrum of the samples was obtained on a Thermo Nicolet 8700 FT-IR spectrometer with a spectral resolution of 4 cm⁻¹.

Results and discussion. The nitrogen adsorption/desorption isotherms in both studied samples belong to type IV according to the classification of Brunauer, Emmett and Teller; the presence of a hysteresis loop in them is correlated with capillary condensation occurring in the mesopores. However, they differ in the type of hysteresis loop (figure 1). If on the isotherm of the MAS-1 sample the hysteresis loop belongs to the H1 type, indicating a narrow pore size distribution and ordered structure, then on the isotherm of the MAS-2 sample - to H4 type and indicates narrow slit-shaped pores [16]. The synthesized materials also sharply differ in specific surface area (MAS-1 – 511 m²/g, MAS-2 – 1170.0 m²/g), the average pore diameter and pore volume vary from 3.82 nm to 2.19 nm and 1.47 cm³/g to 0.875 cm³/g (table 1). According to the data shown in figure 2, for both samples the maximum on the pore size distribution curve is observed at 3.5-4.2 nm. The average pore size for all three samples is due to the presence of a small minority of macropores.