

and the muds from its leaching. The considerable range of reflections shows that it is well crystallised. The Mg-Fe-chlorite is characterised by a small number of diffractions, which indicates poor crystallisation.

On the DTA and DTG curves (figs. 3 and 4) there is an endothermic effect at 600-650°C, corresponding to release of the first portion of structural water from the brucite-like layer, and a small endothermic effect at 750-800°C, which characterises release of the second portion of structural water from the mica-like layer. The endothermic effect passes directly into a well defined exothermic effect with a maximum at 820°C (fig. 3) corresponding to recrystallisation of an olive structure.

The derivatogram of the red mud from the leaching of the Belgorod bauxites (fig. 5) as a whole is characterised by the presence of the same effects as the derivatograms of the muds given in figs. 3 and 4. Consequently, the muds from the leaching of Fe and Mg-Fe-chlorites are represented by undecomposed chlorites. Hydrated sodium aluminosilicate was not found in the muds, and this confirmed the thermodynamic calculations.

Thus, the chlorites are not decomposed during the autoclave leaching, and their presence in the bauxites from some deposits is reflected in the technology of the extraction of alumina by the Bayer method. For example, the incomplete extraction of the  $Al_2O_3$  from the hydrargillite bauxites of the Belgorod KMA deposit in the Bayer process is due to the fact that about 10% of the alumina

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#### Nitric acid autoclave recovery of products from concentration of high-silica bauxites

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Recently researches into the mechanical concentration of bauxites have been widely developed in the USSR. Usually, the concentration of low-grade bauxites leads to the production of two products i. e., a concentrate distinguished from the initial rock by an increased silica ratio and a second product, which contains a considerable amount of alumina in the form of kaolinite or other aluminosilicates and is not waste material. The mechanical concentration could become most effective if the bauxite concentrate were treated by the Bayer process and the low-ratio product, which contains an increased amount of silica, were treated by the acid method.

Investigations carried out in the USSR and abroad show that it is possible to use autoclave treatment for the extraction of alumina from clays and kaolins. According to calculations made in recent years<sup>1)</sup>, the nitric acid method is the most economical of the acid methods for the production of alumina from clays. It seemed expedient to investigate the use of this method for the treatment of high-silica bauxites and kaolinite products from their mechanical concentration, which differ from clays by the presence of bauxite minerals and a higher content of alumina.

Earlier<sup>2)</sup> we studied the behaviour of the principal alumina-containing minerals in the breakdown of the products from concentration with nitric acid. The conditions for transfer of aluminium into solution and separation of aluminium from iron directly during nitric acid treatment of the boehmite-kaolinite product isolated during the concentration of high-silica bauxites of the North-Omega deposit are examined in the present work.

The starting material is characterised by a particle size of  $-44\mu$  and by the following contents of the principal components (wt.%): 47.5  $Al_2O_3$ , 22.5  $SiO_2$ , 7.6  $Fe_2O_3$ , and 16.1 calcination loss. The alumina-containing minerals are represented by kaolinite, boehmite, and hydrargillite and, in smaller amounts, diaspore and hydromica; the iron-containing minerals are goethite and haematite.

contained in the bauxites and practically all the silica are present in the composition of high-iron chlorite, which does not decompose during autoclave leaching of bauxites.

#### Conclusions

1. The decomposition reactions of various types of chlorites in alkaline aluminate solutions are thermodynamically impossible.
2. The results from the thermodynamic calculations were confirmed by various methods of physico-chemical analysis of the muds after the leaching of chlorites and KMA bauxites.

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Treatment of the boehmite-kaolinite product with nitric acid under atmospheric pressure conditions does not secure satisfactory extraction of alumina. Thus, treatment of the raw product with 40% nitric acid only extracts about 5% of the alumina into solution, while treatment of the roasted product does not extract more than 50%. Treatment at increased temperatures in an autoclave possesses advantages, among which the high degree of decomposition, the reduction in the acid consumption, and the production of solutions less contaminated with iron should be noted primarily.

The experimental part of the work was carried out in autoclave bombs made of Kh18N9T steel. Samples of the initial product were roasted in a muffle furnace at 600°C for 2h (the optimum roasting conditions for the given product) and loaded into bombs with a specified amount of 40%  $HNO_3$ , with filling factors of 0.6-0.7. The bombs were placed in an air thermostat with an electric heater and automatic temperature control. Agitation of the pulp was realised by rotation of the bombs through the "head". The degree of breakdown of the initial product was determined from the  $Al_2O_3$  and  $Fe_2O_3$  contents in the insoluble residue, and in addition the solutions were analysed for iron and aluminium content.

The effect of the acid consumption and the length and temperature of the process on the breakdown of the previously roasted boehmite-kaolinite product was investigated. In the first stage of the investigations a 100% consumption of nitric acid on the stoichiometric amount for the formation of  $Al(NO_3)_3$  was used. The initial concentration of the acid amounted to 40%; it is not advisable to use stronger acid, since 50%  $HNO_3$  gives a thick pulp, which must then be diluted considerably with water.

Fig.1 shows the effect of the length of autoclave treatment of the boehmite-kaolinite product with nitric acid at 140, 160, and 180°C on the extraction of aluminium (curves 1-3)

and iron (curves 4-6) into solution. With short treatment (1h) the increase in temperature has a significant effect on the degree of extraction of aluminium. However, the longer the process, the smaller the effect of temperature. Maximum extraction of aluminium, corresponding to 94-95%, was obtained after 1h at 180°C, 1.5h at 160°C, and 2h at 140°C. The strong effect of temperature on the decomposition rate of the alumina-containing minerals makes it possible to suppose that the process is controlled by a kinetic stage under the conditions adopted.

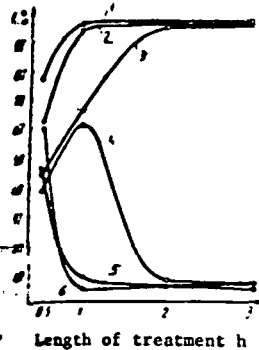


Fig. 1  
Extraction of aluminium (1-3) and iron (4-6) into solution as a function of the length of the process of breakdown of the initial product at various temperatures (°C): 180 (1,6), 160 (2,5); 140 (3,4).

The insoluble residues after nitric acid treatment, filtration, and washing (satisfactory filtration) were analysed by IR spectroscopy. In those cases where extraction of aluminium close to the maximum value was obtained the  $Al_2O_3$  was detected in the residue mainly in the form of hydromica. The content of this mineral is clearly explained by the incomplete extraction of aluminium into solution.

The behaviour of iron during nitric acid treatment of the product investigated is interesting. Curves 4-6 (fig. 1) show that two processes take place simultaneously during treatment of the product in the autoclave, i. e., dissolution of iron and its precipitation as a result of hydrolysis, and the curves for the extraction of iron with time therefore have a maximum. At 140°C (curve 4) this maximum is clearly defined, and at higher temperatures (curves 5 and 6) it lies to the left of the first experimental points. As a result of hydrolysis the concentration of iron in the solution decreases to 0.9-1.1 g/l, and the iron ratio of the solution ( $\mu_{Fe} = Al_2O_3/Fe_2O_3$ ) increases to 100.

The hydrolysis of aluminium under the adopted conditions does not go further than the formation of soluble basic salts since the concentration of aluminium in the solution does not decrease.

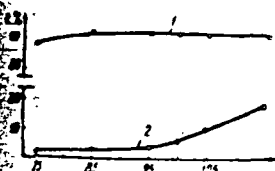


Fig. 2  
Extraction of aluminium (1) and iron (2) into solution as a function of the acid consumption.

Consumption of acid on stoichiometric amount for the formation of  $Al(NO_3)_3$ , Z

#### Diffusion and activity of hydrogen in the aluminium-copper system

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The interest in the study of the liquid state in metals is due to successes in the theory of the liquid state and the increasing possibilities for practical utilisation of laboratory experimental data. The study of the diffusion of hydrogen in liquid metals is of theoretical and practical interest.

The effect of the acid consumption on the extraction of aluminium and iron into solution is shown in fig. 2. The extraction of alumina does not increase when the acid consumption exceeds the stoichiometric amount for the formation of  $Al(NO_3)_3$ . The hydromicas still remain unopened. Even with an acid consumption of 85-95% of the stoichiometric perfectly acceptable extraction of alumina (93-94%) is obtained, and 0.4-0.5 g/l of  $Fe_2O_3$  is retained in the main solution, which has pH = 1.2-1.4, and this amounts to ~2.5% of the amount of iron present in the raw material. The solutions contain about 0.02 g/l of  $SiO_2$  and less than 0.01 g/l of  $TiO_2$ . The aluminium content in these solutions corresponds to 100-110 g/l, and after combination with the wash waters it decreases to 85 g/l.

The obtained solutions have an iron ratio approaching 300. The insoluble residues contain (wt. %): 5.5-6.0  $Al_2O_3$ , 53-54  $SiO_2$ , 19-20  $Fe_2O_3$ .

Consequently, considerable removal of iron from the solution of aluminium nitrate is achieved directly during nitric acid autoclave treatment of the boehmite-kaolinite product containing 7.6%  $Fe_2O_3$ . However, this degree of removal of iron is not the maximum; further higher purification of the solution can be realised after removal of the solid phase. This is demonstrated by investigations of the high-temperature hydrolysis of iron from nitrate solutions, realised both on synthetic solutions and on solutions obtained during the bleaching of clay<sup>3,4</sup>).

#### Conclusions

1. The low-ratio kaolinite products obtained during concentration of bauxites can be subjected to a nitric acid autoclave treatment in order to transfer the aluminium selectively into solution.
2. For the case of a boehmite-kaolinite product containing iron it was shown that under conditions of nitric acid autoclave treatment it is possible almost completely to transfer the aluminium into solution and to separate it from the bulk of the iron, silicon, and titanium.

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Of the three main links in the interaction of gases with liquid metals (absorption, diffusion, dissolution) the least investigated is diffusion. The setting up of experiments on the determination of the diffusion of hydrogen in molten metals involves procedural difficulties, i. e., convective flows in the melt and the difficulty of their suppression