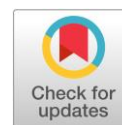# Colloid-chemical properties of surfactant–nitric acid–water systems

Lev Danilin [iD], Tatyana Lugovitskaya *[iD], Elvira Kolmachikhina [iD], Denis Rogozhnikov [iD]

Institute of New Materials and Technologies, Ural Federal University, Ekaterinburg 620009, Russia

* Corresponding author: t.n.lugovitskaia@urfu.ru

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Abstract

The behavior of the surfactants Tween 80, SaS and alkyl betaine in aqueous and nitric acid environments as promising additives for nitrate leaching of hard-to-process ore concentrates of non-ferrous metals was studied. The influence of surfactant concentration (0.04–1.28 g/dm^3), nitric acid concentration (0.1–10 g/dm^3) and temperature (295–343 K) on the surface tension, critical micelle concentration (CMC), pH and optical density of aqueous surfactant solutions and surfactant–HNO_3–H_2O systems was found. The critical micelle concentration of the surfactants used was estimated. A positive effect of nitric acid on the surface activity of surfactants was discovered, which manifests itself in a decrease in both the CMC and the surface tension at the liquid–gas interface. The values of surface activity and Gibbs energy of surfactant micelle formation in aqueous and nitric acid media were calculated. Associative processes in the solutions and compositions were confirmed by measuring the optical density of the systems under study.

Key findings

● The surfactants (Tween 80, SaS, Alkyl betaine) used in our work are suitable and very promising additives for intensifying atmospheric nitric acid leaching of hard-to-process ores.

● The proposed surfactants demonstrate an increase in surface activity in a nitric acid environment.

1. Introduction

Non-ferrous metallurgy remains a leading industry in the economies of advanced countries, as evidenced by the continuously increasing consumption of non-ferrous metals in the power industry, mechanical engineering, civil engineering, and chemical industries [1]. Expanding the range and volumes of manufactured products based on the maximum extraction of value from multi-component raw materials is the primary strategic task of non-ferrous metallurgy, as it meets the interests of individual manufacturers and, in general, the state resource conservation policy guidelines [2]. However, to date, rich and easy-to-dress deposits of non-ferrous metal ores have been almost exhausted. The processing now involves difficult-to-dress ores, and finely dispersed, technogenic raw materials with a low content of valuable components [3]. Double and triple hard-to-process ores are especially important. They contain so-called

carbonaceous matter and highly toxic compounds [4]. Carbonaceous matter in ores and concentrates is represented by various modifications of carbon (graphite, anthracite, and kerogen), has a developed porous surface and, along with humic acids, high adsorption activity towards precious metals [5, 6]. In general, the complexity of the morphological composition of ores and concentrates, as well as the presence of carbonaceous matter, complicates the choice of an effective technology for their processing to achieve a high extraction degree of valuable components using traditional methods. Therefore, there is an urgent need to develop novel methods and approaches for extracting metals from such hard-to-process raw materials. In this regard, we believe that the use of atmospheric nitrate leaching of ores and concentrates in the presence of surfactants could be very efficient. In this case, nitric acid HNO_3 acts as an oxidizer and catalyst for the oxidation reactions of ore concentrate [7, 8]. The introduction of a surfactant will contribute to an adsorption decrease in

the surface energy (Rehbinder's effect) of concentrate particles, leading to their effective wetting, dispersion and, accordingly, faster and more efficient opening of the mineral matrix. In addition, it is assumed that properly selected surfactants will be adsorbed on the surface of carbonaceous matter and eliminate its negative impact at all stages of ore processing.

It should be noted that surfactants are currently not used directly at the stage of nitrate leaching of ores. This is due, on the one hand, to the specifics of the process, in particular to the high oxidative potential of the medium (~700–800 mV), the multi-ionic nature of the pulp, heterogeneity of the process, and, on the other hand, to the lack of targeted research on selecting the surfactants and studying their properties on model and real systems on a laboratory and, especially, an industrial scale.

We are conducting a set of physicochemical studies aimed at selecting surfactants and studying their behavior in nitric acid environments as promising additives for hydrometallurgical processing of hard-to-process ores. The paper presents studies of the behavior of nonionic (Tween 80), amphoteric (alkyl betaine) and anionic (sodium secondary alkyl sulfonate) surfactants in a nitric acid environment.

2. Materials and methods

The surfactants used were:

– Nonionic surfactant: polyoxyethylated (20) sorbitan monooleate Tween 80 Tw80 (Ferak, France).

– Amphoteric surfactant: alkyl betaine AB (TU 2480-002-04706205-2004) (LLC NPO "NIIPAV", Volgodonsk, Russian Federation).

– Anionic surfactant: secondary sodium alkyl sulfonate SaS (WeylChem, Germany).

The surfactants used were subjected to no additional purification.

Surfactant–HNO_3–H_2O systems were prepared using HNO_3 (ρ = 1.49 g/cm^3) of reagent grade and double-distilled water with a specific electrical conductivity of 2·10^{-5} S/m.

2.1. Preparation of solutions

To prepare an aqueous solution, a weighed portion or an aliquot of the surfactant was dissolved in bidistilled water and stirred on a magnetic stirrer at a speed of 200 rpm for 15 min. The ready solution was filtered on a Millipore filter with pore diameters of ≤0.45 μm. From the resulting solution, a series of solutions with C_{surf} 0.002–1.280 g/dm^3 were prepared by dilution. Solutions with a HNO_3 concentration of 0.1–10 g/dm^3 were prepared by introducing a calculated amount of concentrated acid, taking into account the total volume of the solution.

The solutions were analyzed 24 h after preparation to achieve adsorption equilibrium.

2.2. Surface tension

Surface tension at the liquid–gas interface (σ_{l-g}, J/m^2) was determined by the Rehbinder method. The method is based on measuring the pressure at which the separation of an air bubble blown into the liquid through a capillary occurs. When a bubble is slowly pressed from a capillary into a liquid, an excess internal pressure Δp arises therein, which, according to Laplace's law (1), is determined by the surface tension σ_{l-g} and the curvature of the bubble surface r:

$$\Delta p = \frac{2\sigma_{l-g}}{r}. \tag{1}$$

Since it would be quite difficult to measure r experimentally, to determine σ_{l-g} of the solutions and compositions under study, the differences in the heights of the manometric liquid in water ($\Delta h H_2O$) and in the test solution (Δh) were measured. Surface tension σ_{l-g} was calculated using Equation (2):

$$\sigma_{l-g} = \sigma_{H_2O} \cdot \frac{\Delta h}{\Delta h_{H_2O}}. \tag{2}$$

To find the temperature dependence $\sigma_{l-g} = f(T)$, a thermostatic cell was used. Solutions and compositions were additionally thermostated for 1 h before measurements.

2.3. Critical micelle concentration, optical density and pH

The critical micelle concentration (CMC) of surfactants in aqueous solutions, including those with various HNO_3 content, was determined from surface tension isotherms in semilogarithmic coordinates [9, 10]. Surface activity was calculated as the tangent of the tangential angle to the initial portion of the surface tension isotherm (3):

$$g = \lim_{C \to 0} \frac{\mathrm{d}\sigma_{l-g}}{\mathrm{d}C}, \tag{3}$$

where C is the concentration of the surfactant solution (M/m^3); σ_{l-g} the surface tension of the solution (J/m^2).

From the obtained CMC values, the Gibbs energy $\Delta_m G$ of micelle formation was calculated using Equation (4):

$$\Delta_m G = RT \ln \mathrm{CMC} \tag{4}$$

where R = 8.314 J/mol·K is the universal gas constant; T = 298 K is the absolute temperature; CMC is the critical micelle concentration (mol/m^3).

The optical density (A) of solutions and compositions was measured using Analytik Jena spectrophotometer in the wavelength range 315–400 nm, with cuvette thicknesses from 2 to 5 cm.

The pH value was monitored using a Mettler Toledo Five Easy FE20 pH meter (MTD, Singapore).

All data presented in the work were obtained by averaging the results of at least three measurements.

3. Discussion

At the initial stage of this study, preliminary testing of a number of surfactants from the class of dispersants, stabilizers, and foaming agents (at least 10) was carried out for their compatibility with nitric acid. To do this, a drop of concentrated nitric acid was applied onto a glass substrate, and a drop of an aqueous surfactant solution with a surfactant concentration of 1.28 g/dm³ was placed on its surface.

If, as a result of the interaction, phase separation was observed at the interface between HNO_3 and the aqueous surfactant solution, this surfactant was not used in further experiments.

Thus, the following surfactants were selected for the study: LS, AB, Tw, SDS, and SaS (see Table 1). The properties of LS and SDDS are presented in [11]. In this work, Tw, AB, and SaS were studied.

Tween 80 (polyoxyethylated (20) sorbitan monooleate) belongs to the class of nonionic surfactants with a molecular weight of 1.308 Da and a hydrophilic–lipophilic balance number of 15 [12]. The structure of the Tw molecule is shown in Figure 1a. Tw is biodegradable, non-toxic, and is used in the pharmaceutical, cosmetic and food industries as a solubilizer for oils and vitamins, an emulsifier, and a stabilizer.

When dissolved in water in the concentration range of C_{Tw} 0.04–1.28 g/dm³, Tw forms colorless solutions with pH 6.2–6.3.

Upon contact with water, the hydrophobic chains of Tw molecules are pushed out of the bulk and associate through bonding among hydrocarbon chains, forming aggregates surrounded on the outside by polar groups facing the solvent [13]. When the surface (boundary) l–g is completely filled, micelles begin to form in the bulk of the solution. Note that CMC is one of the key characteristics of surfactant solutions, which in most cases determines the boundaries of their functional activity [14].

The described associative behavior of Tw can be traced by changes in the surface tension of the corresponding solutions (Figure 2a, curve 1). As can be seen from the $\sigma_{l-g} = f(\lg C_{Tw})$ dependence, with an increase in C_{Tw} from 0.04 to 0.16 g/dm³, σ_{l-g} sharply decreases down to $53 \cdot 10^{-3}$ J/m².

Table 1 Testing of surfactants for compatibility with nitric acid.

No.	Name of surfactant	Phase separation
1	Lignosulfonate (LS)	–
2	Alkyl betaine (AB)	–
3	Didecyldimethylammonium chloride	+
4	Alkyldimethylbenzylammonium chloride	+
5	ScatUs	–
6	Alkyldimethylamine oxide	–
7	Carboxylate of ethoxylated alkylphenols	–
8	Tween 80 (Tw)	–
9	Sodium dodecyl sulfate (SDS)	–
10	SaS (secondary sodium alkyl sulfonate)	–
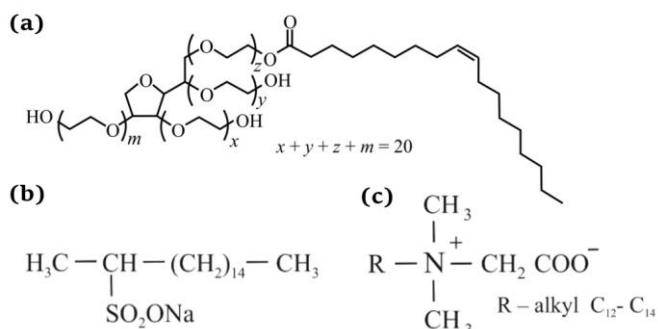
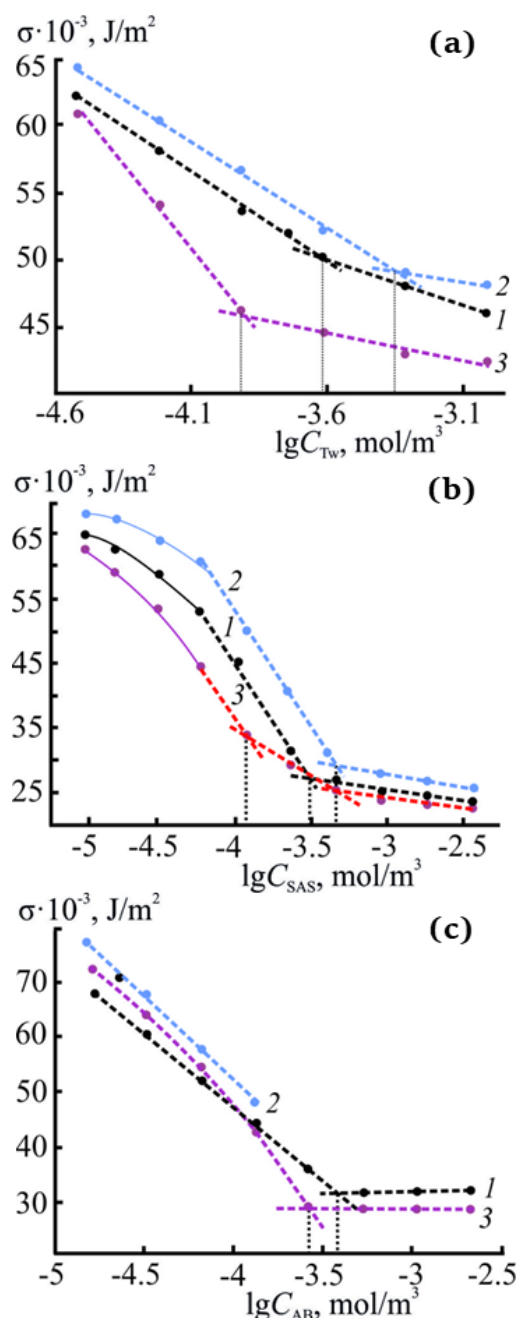**Figure 1** Structural formulae of the surfactants used: Tw (a), SaS (b), and AB (c).

Figure 2 Surface tension isotherms of aqueous solutions (a, b, c, curve 1) and aqueous acid compositions of surfactants (a, b, c, curves 2 and 3): T_W (a), SaS (b) and AB (c); $C_{HNO_3} = 0$ (1); 0.6 (2); 5.0 g/dm³ (3), $T = 298$ K.

This decrease in $\sigma_{l\text{-}g}$ values reflects the process of filling the surface layer with surfactant molecules and reaching the limiting value of adsorption. Then, the process of micellization is observed in the bulk of the solution and the $\sigma_{l\text{-}g} = f(\lg C_{Tw})$ dependence reaches a plateau. The CMC of Tw in pure water is 0.24 mmol/m³. These data are in good agreement with those given in the literature [12–14]. Some adsorption parameters calculated based on tensiometric measurements are given in Table 2. The value of surface activity g of Tw in water is 0.15 J·m/mol, and the Gibbs energy of micellization $\Delta_m G$ is –3 kJ/mol. Negative $\Delta_m G$ values indicate the spontaneous occurrence of micellization.

Micelle formation in the studied compositions is also confirmed by a change in their optical density (Table 3).

Similar patterns of changes in the surface properties of Tw–H₂O solutions and Tw–HNO₃–H₂O compositions were also observed for other analyzed surfactants (SAS and AB).

SaS, or secondary sodium alkyl sulfonate (Figure 1b) is an anionic surfactant with an average carbon atom content of 15.9 and a molecular weight of 328 Da [15]. It has foaming, solubilizing, emulsifying properties and low sensitivity to the presence of electrolytes. SaS is chemically stable in strongly alkaline and strongly acidic media, and in the presence of oxidizing agents. It is thermally stable in aqueous solutions at their boiling temperatures [16].

Table 2 Surface properties of surfactant solutions and surfactant–HNO₃–water systems.

Surfactant	g, J·m/mol		
	0*	0.6**	5.0***
Tw	0.23	0.20	0.28
SAS	0.19	0.18	0.32
AB	0.15	0.18	0.23
	CMC, mmol/m³		
	0.6	5.0	0
Tw	0.40	0.12	0.24
SAS	0.47	0.12	0.32
AB	–	0.26	0.36
	$\Delta_m G$, kJ/mol		
	0.6	5.0	0
Tw	–1.77	–4.70	–3.87
SAS	–1.87	–5.47	–1.98
AB	–	–3.27	–2.26

*, **, *** C_{HNO_3}, g/dm³

Table 3 Changes in the optical density of surfactant–HNO₃–H₂O systems depending on their composition ($\lambda = 315$ nm, $l = 3$ cm).

Composition	Tw		SaS	AB
C_{HNO_3}, g/dm³	0.6	5	5	5
C_{surf}, g/dm³		D		
0.04	0.220	0.938	–	0.914
0.08	0.218	0.926	0.534	1.013
0.16	0.217	0.944	0.535	1.003
0.32	0.221	0.932	0.537	0.999
0.64	0.245	0.924	0.595	1.007
1.28	0.271	–	0.505	1.020

An aqueous SaS solution in the studied concentration range has a pH within 5.20–6.48. The introduction of SaS (Figure 2b, curve 1) leads to a sharp decrease in the surface tension of water. The surface activity of SaS is 0.19 J·m/mol, and the CMC is 0.32 mmol/m³ (Table 2).

In the presence of small amounts of HNO₃ (C_{HNO_3} 0.1–1.0 g/dm³), the surface tension of the SaS–HNO₃–H₂O solutions increases slightly, their surface activity naturally decreases, and the CMC increases accordingly.

The introduction of nitric acid in concentrations of more than 1 g/dm³ leads to a significant decrease in $\sigma_{l\text{-}g}$, compared with an aqueous SaS solution, and an increase in its micelle-forming ability, expressed in a decrease in CMC values down to 0.12 mmol/m³. We also note that two inflection points appear on the $\sigma_{l\text{-}g} = f(\lg C_{SaS})$ curve (Figure 1b, curve 3), which may indicate the formation of premicellar structures in the region of lower SaS concentrations. The established decrease in CMC values at increased HNO₃ content may be associated with a number of electrostatic interactions. HNO₃, as a strong electrolyte, is ionized in aqueous solutions. H⁺ interacts both with the hydrophilic groups of SaS, which are in equilibrium with micelles, and with water molecules from the aqueous pseudophase. The interactions of H⁺ ions with the negatively charged functional group of SaS micelles are also energetically favorable, leading to a decrease in electrostatic repulsion, as a result of which the stability of the micelles increases and, consequently, the CMC decreases. As C_{HNO_3} increases, the concentration of H⁺ in solution increases as well. The replacement of Na⁺ by H⁺ ions in the micellar phase indicates the possible protonation of the anionic head groups of SaS in the presence of HNO₃. Thus, in this case, the SaS micellar solution contains Na⁺ and H⁺ counterions, which tend to bind to the ionic micelle and form ion pairs [17, 18]. In addition, it should be noted that the presence of nitric acid in a SaS solution, in addition to electrostatic interactions, could also lead to chemical ones, in particular to SaS hydrolysis. However, this requires additional research.

SaS association processes both in aqueous solution and in SaS–HNO₃–H₂O systems proceed spontaneously ($\Delta_m G$, 1.98–5.47 kJ/mol) and are consistent with changes in the optical density of the compositions (Table 2).

Alkyl betaine (Figure 1c) is an amphoteric surfactant with a molecular weight of 308 g/mol. It is used as a thickener and foaming agent, being compatible with all types of surfactants.

In an aqueous solution, the AB molecule, having carboxyl and amino groups (Figure 1c), is in the zwitterionic form [19]. The pH of this solution varies in the range of 5.9–6.3. When HNO₃ is introduced into AB–HNO₃–H₂O systems, the carboxyl group of the molecule is protonated and AB will exhibit the properties of a cationic surfactant.

The surface tension isotherm of the AB solution (Figure 1c), just like that of the other surfactants considered, has a classical form, that is, with increasing C_{AB}, $\sigma_{l\text{-}g}$ first decreases, and when the CMC is reached, it takes a constant

value. The CMC of AB is 0.36 mmol/m^3. The introduction of HNO$_3$ into AB–HNO$_3$–H$_2$O system has a positive effect, leading to an increase in the surface activity of the solution from 0.15 (aqueous solution) to 0.23 J·m/mol and a decrease in CMC from 0.36 down to 0.26 mmol/m^3.

Thus, we can conclude that nitric acid has a positive effect on the surface activity of all surfactants under study, manifested in a decrease in surface tension at the liquid–gas interface and in CMC.

Since atmospheric leaching of ore concentrates is most often carried out at temperatures of 333–353 K, in the next series of experiments we studied the effect of temperature on changes in the surface properties of surfactant–HNO$_3$–H$_2$O systems (Figure 3).

An increase in temperature from 298 to 343 K has virtually no effect on the surface properties of the Tw/AB–HNO$_3$–H$_2$O systems (Figure 3a). At premicellar concentrations of $C_{Tw/AB} \leq 0.18$ g/dm^3 and in the range of C_{HNO3} 1–10 g/dm^3, σ_{l-g} almost remains constant. At higher concentrations of $C_{Tw/AB} > 0.18$ g/dm^3, σ_{l-g} tends to decrease with increasing temperature.
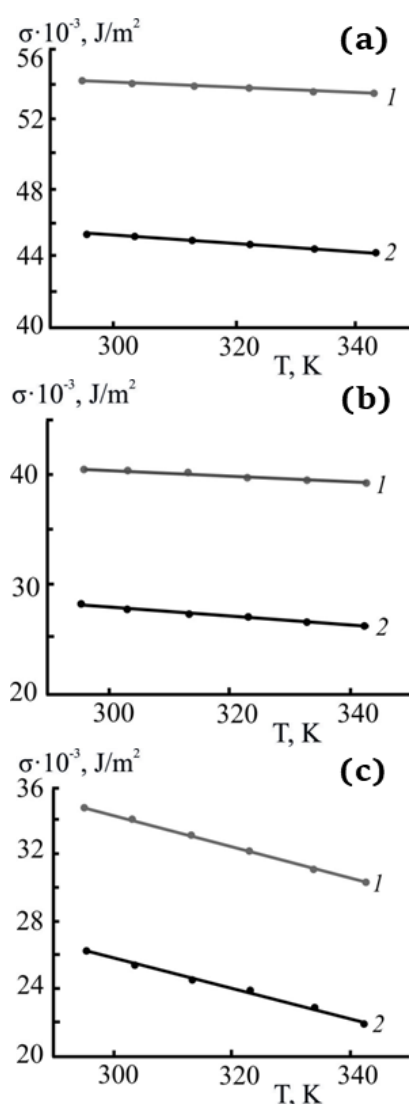

Figure 3 Temperature influence on the surface tension of surfactant–HNO$_3$–H$_2$O systems: Tw (a), AB (b) and SaS (c); C_{surf} = 0.4 (1) and 0.64 g/dm^3 (2); C_{HNO3} = 7 g/dm^3.

An increase in temperature in the SaS–HNO$_3$–H$_2$O system in the range of C_{SaS} 0.04–1.28 g/dm^3 and C_{HNO3} 0.6–10.0 g/dm^3 leads to a decrease in their surface tension by an average of $\sim$3.5–5·10^{-3} J/m^2. This change in $\sigma_{l-g} = f(T)$ is due to a decrease in the proportion of free counterions. An increase in temperature causes gradual dehydration of Na$^+$ and H$^+$ counterions, leading to intensification of their condensation on the R–SO$_2$O$^-$ anion due to the increase in electrostatic attraction between dehydrated ionic particles with a higher surface charge density. The described processes generally contribute to the compaction of associates (micelles and mixed micelles), an increase in hydrophobicity and their more intense movement into the surface layer [20].

Thus, taking into account the conducted studies of the behavior of surfactants depending on the composition of the solution (C_{surf} 0.40–1.28 g/dm^3) or surfactant–HNO$_3$–H$_2$O compositions (C_{HNO3} 0.1–10.0 g/dm^3) and external conditions (T 295–343 K), they can be arranged in the following series by the increase in surface activity: AB $\leq$ Tw < SaS.

4. Limitations

Research on the selection of surfactants for metallurgical processes is usually carried out using model systems. The preparation of model systems is complicated by the need to take into account a larger number of factors: the concentration of acid and solid phase, the size of ore/concentrate particles, the appearance of ions of variable valence during the dissolution of ore, and high temperatures. This requires a huge number of experiments, which do not always lead to positive results.

5. Conclusions

In general, our results allow us to conclude that the surfactants used in our work are suitable and very promising additives for intensifying atmospheric nitric acid leaching of hard-to-process ores. The study of leaching processes of hard-to-process ores in the presence of the proposed surfactants on a laboratory and semi-industrial scale will be the subject of further reports.

● Supplementary materials

No supplementary materials are available.

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Conceptualization and Methodology: T.N.L., D.A.R.
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Validation: T.N.L., D.A.R.

● Conflict of interest

The authors declare no conflict of interest.

● Additional information

Authors IDs:

Tatyana Lugovitskaya, Scopus ID 36656117800;
Elvira Kolmachikhina, Scopus ID 57204526484;
Denis Rogozhnikov, Scopus ID 55736095600.

Website:

Ural Federal University, https://urfu.ru/en/.

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