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\*Y.B. Raiymbekov, P.A. Abdurazova, U.B. Nazarbek

Non-profit Joint Stock Company M. Auezov South Kazakhstan University (Shymkent, Kazakhstan)

## THERMODYNAMIC ANALYSIS OF ENRICHMENT OF LOW-GRADE PHOSPHATE RAW MATERIALS WITH ORGANIC ACID

**Abstract.** The phosphorus industry faces the challenge of developing low-grade phosphate reserves for industrial purposes, requiring technological solutions for enrichment. About 20 million tons of low-grade phosphorites are currently unsuitable for processing. Depleting phosphorite deposits and low phosphorus anhydride levels reduce global demand for domestic ore and necessitate research on enrichment and integrated use. This study aims to conduct a thermodynamic analysis of low-grade phosphate enrichment using acetic acid. Using the HSC Chemistry 9.3 program, developed by Finnish metallurgical company Outokumpu, the thermodynamic interactions of phosphate compounds and acetic acid were analyzed. It reveals that the reactions of phosphate raw materials with acetic acid are thermodynamically feasible, except for fluorapatite, iron (III) oxide, and silicon dioxide. Carbonate, aluminum-, and potassium-containing compounds in low-grade phosphate materials show the highest likelihood of interaction with acetic acid.

**Key words:** thermodynamic analysis, low-grade phosphate, enrichment, organic acid, acetic acid, phosphorite deposits, Gibbs energy, phosphate industry.

### Төменсұрыпты фосфат шикізатын органикалық қышқылмен байытудың термодинамикалық талдауы

**Аңдатпа.** Фосфор өнеркәсібінің алдында байытудың технологиялық шешімдерін қажет ететін өнеркәсіптік мақсаттар үшін төмен сұрыпты фосфаттардың қорларын игеру міндеті тұр. Қазіргі уақытта 20 миллион тоннаға жуық төмен сұрыпты фосфориттер қайта өңдеуге жарамсыз. Фосфорит кен орындарының сарқылуы және фосфор ангидридін төмен деңгейі отандық рудаға жаһандық сұранысты төмендетеді және байыту мен кешенді пайдалану бойынша зерттеулерді қажет етеді. Бұл зерттеу сірке қышқылын қолдана отырып, төмен сұрыпты фосфаттарды байытуға термодинамикалық талдау жүргізуге бағытталған. Финдік Outokumpu металлургиялық компаниясы әзірлеген HSC Chemistry 9.3 бағдарламасының көмегімен фосфат қосылыстары мен сірке қышқылының термодинамикалық өзара әрекеттесуі талданды. Фосфат шикізатының сірке қышқылымен реакциясы фторпатит, темір (III) оксиді және кремний диоксидін қоспағанда, термодинамикалық тұрғыдан мүмкін екендігі анықталды. Төмен сұрыпты фосфат материалдарындағы карбонат, алюминий және калий бар қосылыстар сірке қышқылымен әрекеттесу ықтималдығын көрсетеді.

**Түйінді сөздер:** термодинамикалық талдау, төмен сұрыпты фосфаттар, байыту, органикалық қышқыл, сірке қышқылы, фосфорит кен орындары, Гиббс энергиясы, фосфат өнеркәсібі.

### Термодинамический анализ обогащения низкосортного фосфатного сырья органическими кислотами

**Аннотация.** Перед фосфорной промышленностью стоит задача освоения запасов низкосортных фосфатов для промышленных целей, требующих технологических решений по обогащению. Около 20 млн тонн низкосортных фосфоритов в настоящее время непригодны для переработки. Истощение месторождений фосфоритов и низкий уровень ангидрида фосфора снижают глобальный спрос на отечественную руду и требуют исследований по обогащению и комплексному использованию. Это исследование направлено на проведение термодинамического анализа обогащения низкосортных фосфатов с использованием уксусной кислоты. С помощью программы HSC Chemistry 9.3, разработанной финской металлургической компанией Outokumpu, были проанализированы термодинамические взаимодействия фосфатных соединений и уксусной кислоты. Выявлено, что реакции фосфатного сырья с уксусной кислотой термодинамически осуществимы, за исключением фторпатита, оксида железа (III) и диоксида кремния. Карбонатные, алюминий- и калийсодержащие соединения в низкосортных фосфатных материалах проявляют наибольшую вероятность взаимодействия с уксусной кислотой.

**Ключевые слова:** термодинамический анализ, низкосортный фосфат, обогащение, органическая кислота, уксусная кислота, месторождения фосфоритов, энергия Гиббса, фосфатная промышленность.

### Introduction

Phosphorus is one of the most abundant elements on earth. In terms of mineralogy, phosphorites are complex and varied in structure. Phosphorus is an integral part of all biological/physiological processes in plants and animals. In addition, phosphate minerals are a valuable natural resource widely used as mineral fertilizers [1].

Phosphorites occur in various geological environments and in many complex mineralogical structures. Representatives of the apatite group predominate in igneous and metamorphic rocks, in particular phosphorites of the fluorapatite type [2]. Depending on the form of formation, phosphorites are divided into two different genetic groups: marine and terrestrial. Marine phosphorites, in turn, consist of a lithologic classification as follows: fine-grained, granular, concretionary, and shelly. The assignment of such names is not accidental, since each of them depends on the characteristics of the geological formation, location at stratigraphic levels, geographical location.

In world practice, various methods of enrichment of phosphorites are known, such as flotation, separation, combined methods. Among the promising methods of enrichment of phosphorites are calcination and heat treatment, magnetic separation and treatment with various acids.

The mechanical method of enrichment of phosphorites is based on processes where drums of various types, sieves,

classifiers with crushers are used [3-4]. Thus, phosphorites required for industrial purposes by Kazphosphate LLP are subjected to mechanical cleaning and grinding at the Karatau mining complex. In the days of the Kazakh SSR, research work and practical tests were carried out on the basis of this institution on the use of various enrichment methods, including flotation. Flotation separation of carbonate-type phosphorite raw materials in an acidic medium was carried out with phosphoric acid as a reagent-suppressor of phosphate minerals (depressant), while synthetic fatty acids were used to separate carbonate-type compounds into a foamy product [5]. The use of the carbonate flotation method did not allow us to achieve the intended results. This is due to the fact that the flotation of phosphorites and carbonates plays an important role in the enrichment process, i.e. the size of the phosphorite grains.

In recent years, as one of the promising methods for the enrichment of low-grade phosphorites, researchers have proposed treatment with organic acid solutions. At the same time, the efficiency of dissolution of carbonates with these acids is high, they also have a low cost, technologically simple design. The possibilities for recovering used acid demonstrate the advantages of this method. In the course of analytical and patent research, organic acids such as formic, acetic, lactic, resinous, citric and maleic acids have been used in many studies to enrich low-grade phosphate raw materials. The main efficiency

of this method lies in the fact that organic acids have the property of selective decomposition of carbonate compounds in the composition of phosphorite. However, it does not affect the phosphate part.

One of the organic monocarboxylic acids, which is widely used in studies on the decomposition of carbonates, is acetic acid [6]. The availability and low cost of this acid on the market indicate the advantages of its use in the enrichment process.

Based on a systematic analysis and generalization of known data on methods, methods, technologies for enrichment of low-grade raw materials and waste from the phosphorus industry, researchers have proposed the use of organic acids as a promising method. In particular, the effectiveness of acetic acid in the selective leaching of carbonate compounds in phosphate raw materials has been proven. The aim of the study is to conduct a thermodynamic analysis of the process of enrichment of low-grade phosphate raw materials using acetic acid.

### Research Methods

To conduct a thermodynamic analysis of the processes of enrichment of low-grade phosphorites with acetic acid, a modern, multifunctional, software package HSC 9.3 was used, based on the principle of maximum entropy and minimization of the Gibbs energy.

The calculation of changes in thermodynamic functions during a chemical reaction is an urgent and frequent problem, the solution of which allows answering a number of important questions. On the basis of a change in the Gibbs energy, one can assess the possibility of a spontaneous reaction proceeding in a certain direction. The magnitude of the enthalpy change is equal to the thermal effect of the reaction and opposite in sign, which means that the reaction is exo- or endothermic, releasing energy or absorbing it from the environment in the opposite direction. The value of the equilibrium constant determines the composition of the equilibrium with a given initial composition of the system in which the reaction occurs [7].

For heterogeneous reactions, the specified module Reaction Equations (Fig. 1) calculates the change in the values of enthalpy  $\Delta H$ , entropy  $\Delta S$  and Gibbs free energy  $\Delta G$  in accordance with the following equations (1-3):

$$\Delta H_T = \sum s_i H_i(\text{product}) - \sum s_i H_i(\text{reagent}), \quad (1)$$

$$\Delta S_T = \sum s_i S_i(\text{product}) - \sum s_i S_i(\text{reagent}), \quad (2)$$

$$\Delta G_T = \sum s_i G_i(\text{product}) - \sum s_i G_i(\text{reagent}), \quad (3)$$

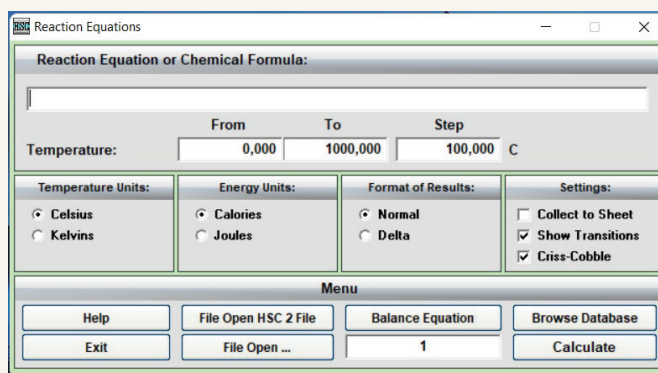
where:  $s_i$  – stoichiometric coefficients.

For a specific heterogeneous reaction, the value of the equilibrium constant ( $K$ ) is closely related to the value of Gibbs free energy  $\Delta G$ , and is calculated by the formula (4):

$$\ln K = -\frac{\Delta G_T}{RT}, \quad (4)$$

where:  $R$  – universal gas constant;

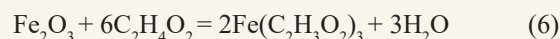
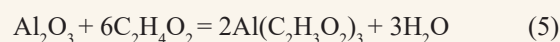
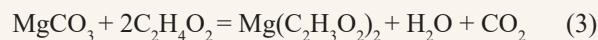
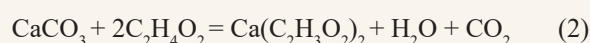
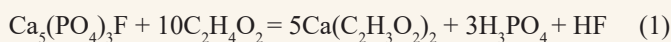
$T$  – temperature.



**Figure 1. Reaction Equations Module.**  
**Сурет 1. Реакция теңдеулерінің модулі.**  
**Рис. 1. Модуль уравнений реакции.**

The database of the program contains the values of the standard enthalpy, entropy, Gibbs energy of more than 17 thousand inorganic and organic compounds. The use of this database allows thermodynamic analyzes to be carried out in the temperature range chosen for a known reaction.

The interaction reactions of phosphate raw materials and the compounds contained in it with acetic acid are carried out according to the following equations:



It is shown that fluorapatite and acetic acid (1) proceed by reaction with the release of calcium acetate, phosphoric acid and hydrogen fluoride, which passes into the gas phase. As a result of the interaction of calcium and magnesium carbonates in phosphate raw materials with acetic acid, a substitution reaction occurs, acetate salts, water and carbon dioxide are released as a product. Since potassium and aluminate compounds are present in the composition of phosphate raw materials in the form of nepheline, their interaction with acetic acid is carried out with the formation of potassium and aluminum acetate products. The proposed chemical reactions are based on known data and, accordingly, are carried out on the basis of the mechanism of interaction of these compounds with acetic acid.

In the process of selective leaching under the action of acetic acid, carbonate, potassium and aluminate compounds contained in phosphate raw materials are involved. They are present in the solution in the form of acetate salts during the exchange reaction, and the phosphate part, compounds of silicon, iron, and calcium fluoride remain in the insoluble part of the pulp. Acetic acid, due to its chemical properties,

is a monocarboxylic acid, which is why it is among the protic polar solvents. This, in turn, is based on the fact that acetic acid contains hydrogen that can be split as the  $H^+$  proton. The  $pK_a$  of acetic acid is  $1.8 \cdot 10^{-5}$ . In other studies [8], these data were substantiated and presented. Below are the results of the thermodynamic analysis of these chemical reactions and the mechanisms of their occurrence.

### Results and Discussion

During the thermodynamic analysis, the temperature range of chemical processes from 0 to 100°C was chosen. The results of the thermodynamic analysis of chemical reactions during the enrichment of phosphorites with acetic acid, depending on the phosphate raw material and the compounds contained in it, are presented in the data of Tables 1-6.

#### Thermodynamic values of reaction (1)

#### (1) реакцияның термодинамикалық көрсеткіштері

#### Термодинамические показатели реакции (1)

Table 1

Кесте 1

Таблица 1

Therm. value	Temperature, °C										
	0	10	20	30	40	50	60	70	80	90	100
delta H, kcal	20.12	16.54	12.63	8.54	4.32	9.83	5.75	1.63	-2.52	-6.70	-10.89
delta S, cal/K	-222.20	-235.08	-248.63	-262.36	-276.05	-258.48	-270.91	-283.11	-295.04	-306.71	-318.11
delta G, kcal	80.82	83.10	85.52	88.08	90.77	93.36	96.01	98.78	101.67	104.68	107.8
K	2.132E-065	7.056E-065	1.713E-064	3.128E-064	4.407E-064	7.098E-064	1.023E-063	1.205E-063	1.184E-063	9.889E-064	7.137E-064
Log(K)	-64.67	-64.15	-63.76	-63.50	-63.35	-63.14	-62.99	-62.91	-62.92	-63.01	-63.14

From the data of table 1 above it follows that the reaction of interaction of fluorapatite with acetic acid thermodynamically does not proceed. As evidence, we can say that the Gibbs free energy ( $\Delta G^0$ ) has a positive value: at 10°C it is 80.82 kcal, increasing the temperature to 60°C it reaches  $\Delta G^0 = 96.01$  kcal. It was shown that at 40°C there was a large change in the values of  $\Delta H$  and  $\Delta S$ , since the indicated reaction practically did not proceed. This situation is explained by the presence of entropy factors. That is, the slowing down of the enthalpy values in

Table 1 with increasing temperature leads to an increase in the values of  $\Delta S$ , which, in turn, is explained by the property of the entropy factor to tend to the destruction and separation of particles.

The interaction of calcium and magnesium carbonates in the composition of fluorapatite with acetic acid indicates the thermodynamic probability of reactions (2)-(3), which have inverse values of  $\Delta G^0$  in the studied temperature zone according to the data in tables 2 and 3.

#### Thermodynamic values of reaction (2)

#### (2) реакцияның термодинамикалық көрсеткіштері

#### Термодинамические показатели реакции (2)

Table 2

Кесте 2

Таблица 2

Therm. value	Temperature, °C										
	0	10	20	30	40	50	60	70	80	90	100
delta H, kcal	-3.63	-2.82	-3.51	-4.25	-5.01	-5.80	-6.60	-7.42	-8.24	-9.07	-9.91
delta S, cal/K	-10.19	-7.18	-9.58	-12.05	-14.54	-17.01	-19.45	-21.86	-24.23	-26.55	-28.84
delta G, kcal	-0.85	-0.79	-0.70	-0.59	-0.46	-0.30	-0.12	0.08	0.31	0.56	0.84
K	4.78E+000	4.070E+000	3.360E+000	2.697E+000	2.110E+000	1.163E+000	1.207E+000	8.866E-001	6.405E-001	4.560E-001	3.205E-001
Log(K)	0.680	0.610	0.526	0.431	0.324	0.208	0.082	-0.052	-0.193	-0.341	-0.494

Table 3

*Thermodynamic values of reaction (3)*

Кесте 3

*(3) реакцияның термодинамикалық көрсеткіштері*

Таблица 3

*Термодинамические показатели реакции (3)*

Therm. value	Temperature, °C										
	0	10	20	30	40	50	60	70	80	90	100
delta H, kcal	-23.25	-19.71	-18.13	-16.88	-15.83	-14.91	-14.10	-13.38	-12.70	-12.08	-11.49
delta S, cal/K	-54.93	-42.07	-36.58	-32.39	-28.99	-26.11	-23.64	-21.49	-19.55	-17.81	-16.21
delta G, kcal	-8.25	-7.79	-7.40	-7.06	-6.75	-6.48	-6.23	-6.00	-5.80	-5.61	-5.44
K	4.024E+006	1.045E+006	3.332E+005	1.234E+005	5.185E+004	2.414E+004	1.225E+004	6.689E+003	3.892E+003	2.393E+003	1.544E+003
Log(K)	6.60	6.01	5.52	5.09	4.71	4.38	4.08	3.82	3.59	3.37	3.31

According to the results of the analysis, the interaction of calcium/magnesium carbonates with acetic acid leads to a decrease in the value of  $\Delta G^0$  as the process temperature increases. From the data in Tables 2 and 3 it follows that the  $\Delta G$  values of magnesium carbonate exceed those of calcium carbonate. The values of enthalpy and entropy effect are also higher for magnesium.

In addition to carbonate compounds, the results of the analysis of the thermodynamic probability of the interaction of potassium and aluminum compounds in phosphate raw materials with acetic acid are shown in Tables 4, 5.

Table 4

*Thermodynamic values of reaction (4)*

Кесте 4

*(4) реакцияның термодинамикалық көрсеткіштері*

Таблица 4

*Термодинамические показатели реакции (4)*

Therm. value	Temperature, °C										
	0	10	20	30	40	50	60	70	80	90	100
delta H, kcal	-102.81	-100.52	-99.95	-99.57	-99.30	-99.10	-98.96	-98.86	-98.80	-98.76	-98.75
delta S, cal/K	-6.99	1.34	3.30	4.58	5.46	6.09	6.52	6.80	6.98	7.09	7.13
delta G, kcal	-100.90	-100.90	-100.92	-100.96	-101.01	-101.07	-101.13	-101.20	-101.27	-101.34	-101.41
K	5.481E+080	7.706E+077	1.769E+075	6.227E+072	3.199E+070	2.304E+068	2.249E+066	2.889E+064	4.766E+062	9.877E+060	2.522E+059
Log(K)	80.73	77.88	75.24	72.24	70.50	68.36	66.35	64.46	62.67	60.99	59.40

Table 5

*Thermodynamic values of reaction (5)*

Кесте 5

*(5) реакцияның термодинамикалық көрсеткіштері*

Таблица 5

*Термодинамические показатели реакции (5)*

Therm. value	Temperature, °C										
	0	10	20	30	40	50	60	70	80	90	100
delta H, kcal	-119.66	-109.97	-106.02	-102.95	-100.43	-98.26	-96.37	-94.70	-93.18	-91.79	-90.49
delta S, cal/K	-386.62	-351.49	-337.76	-327.46	-319.27	-312.44	-306.69	-301.75	-297.38	-293.49	-289.98
delta G, kcal	-14.05	-10.45	-7.01	-3.68	-0.45	2.70	5.79	8.84	11.83	14.79	17.70
K	1.774E+011	1.170E+008	1.683E+005	4.542E+002	2.072E+000	1.482E-002	1.568E-004	2.337E-006	4.725E-008	1.254E-009	4.247E-011
Log(K)	11.24	8.06	5.22	2.65	0.31	-1.82	-3.80	-5.63	-7.32	-8.90	-10.37

According to the above data, the fact that potassium and aluminum oxides can actively interact with acetic acid can be seen in the thermodynamic probability of reactions (4)-(5). Unlike alumina, potassium oxide reacts very actively because its  $\Delta G^0$  value is -100.90 kcal, and as the temperature increases, this value growth. However, since the value of the Gibbs free energy decreases between 10-40°C according to reaction (5), this value begins to acquire a positive value from 50°C.

The decomposition of potassium and aluminum oxides in acetic acid can be explained by the occurrence of these compounds in the composition of phosphate raw materials in the

form of a nepheline mineral. Nepheline is a rock-forming mineral, sodium/potassium aluminosilicate (Na,K)AlSiO<sub>4</sub> [9]. Dorfman M. D., Bussen I. V., Dudkin O. B. in studies conducted by the Academy of Sciences of the USSR, revealed some dissolution of nepheline in acetic acid [10]. Obviously, this information complements the data on the thermodynamic probability of these reactions.

The interaction of Fe<sub>2</sub>O<sub>3</sub>, another compound contained in phosphate raw materials with acetic acid, in the data of Table 6 shows that thermodynamic processes do not occur. The value of  $\Delta G^0$  in the temperature range of the subject has a positive value.

Table 6

*Thermodynamic values of reaction (6)*

Кесте 6

*(6) реакцияның термодинамикалық көрсеткіштері*

Таблица 6

*Термодинамические показатели реакции (6)*

Therm. value	Temperature, °C										
	0	10	20	30	40	50	60	70	80	90	100
delta H, kcal	-31.39	-28.89	-30.89	-33.01	-35.22	-37.49	-39.80	-42.14	-44.52	-46.91	-49.33
delta S, cal/K	-252.60	-243.31	-250.26	-257.38	-264.55	-271.67	-278.71	-285.65	-292.47	-299.16	-305.72
delta G, kcal	37.60	40.04	42.47	45.01	47.62	50.30	53.05	55.87	58.76	61.72	64.74
K	8.136E-031	1.319E-031	2.157E-032	3.535E-033	5.797E-034	9.511E-035	1.563E-035	2.575E-036	4.261E-037	7.088E-038	1.187E-038
Log(K)	-30.09	-30.88	-31.66	-32.45	-33.23	-34.02	-34.80	-35.58	-36.37	-37.14	-37.92

**Conclusion**

Thus, the determination of the thermodynamic probability of the interaction of phosphorite and the basic compounds contained in it with acetic acid allows us to draw the following conclusions:

- reactions of interaction of fluorapatite and iron oxide (III) with acetic acid are not carried out thermodynamically due to the positive value of the Gibbs free energy;
- thermodynamic probability of interaction of calcium and magnesium carbonates, potassium and aluminum oxides with

acetic acid in the composition of phosphate raw materials is carried out according to the following scheme:  $K_2O > MgCO_3 > CaCO_3 > Al_2O_3$ . In addition, since the value of  $\Delta H^0$  in these reactions has negative values, these reactions are exothermic.

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#### Information about authors:

**Raiymbekov Y.B.**, PhD, Postdoctoral Fellow of the Department of Chemistry and Pharmaceutical Engineering, M. Auezov South Kazakhstan University (Shymkent, Kazakhstan), [eplysr@bk.ru](mailto:eplysr@bk.ru); <https://orcid.org/0000-0002-2119-2406>

**Abdurazova P.A.**, PhD, Associate Professor of the Department of Chemistry and Pharmaceutical Engineering, M. Auezov South Kazakhstan University (Shymkent, Kazakhstan), [abdurazova.perizat@okmpu.kz](mailto:abdurazova.perizat@okmpu.kz); <https://orcid.org/0000-0002-5244-7678>

**Nazarbek U.B.**, PhD, Associate Professor of the Department of Chemistry and Pharmaceutical Engineering, M. Auezov South Kazakhstan University (Shymkent, Kazakhstan), [unazarbek@mail.ru](mailto:unazarbek@mail.ru); <https://orcid.org/0000-0001-8890-8926>

#### Авторлар туралы мәлімет:

**Райымбеков Е.Б.**, PhD, М.Әуезов атындағы Оңтүстік Қазақстан университетінің, «Химия және фармацевтикалық инженерия» кафедрасының постдокторанты (Шымкент қ., Қазақстан)

**Абдуразава П.А.**, PhD, М.Әуезов атындағы Оңтүстік Қазақстан университетінің, «Химия және фармацевтикалық инженерия» кафедрасының қауымдастырылған профессоры (Шымкент қ., Қазақстан)

**Назарбек У.Б.**, PhD, М.Әуезов атындағы Оңтүстік Қазақстан университетінің, «Химия және фармацевтикалық инженерия» кафедрасының қауымдастырылған профессоры (Шымкент қ., Қазақстан)

#### Сведения об авторах:

**Райымбеков Е.Б.**, PhD, постдокторант кафедры «Химия и фармацевтическая инженерия» Южно-Казахстанского университета имени М.Ауэзова (г. Шымкент, Казахстан).

**Абдуразава П.А.**, PhD, ассоциированный профессор кафедры «Химия и фармацевтическая инженерия» Южно-Казахстанского университета имени М. Ауэзова (г. Шымкент, Казахстан)

**Назарбек У.Б.**, PhD, ассоциированный профессор кафедры «Химия и фармацевтическая инженерия» Южно-Казахстанского университета имени М.Ауэзова (г. Шымкент, Казахстан)

### ВСЕРОССИЙСКАЯ НАУЧНО-ТЕХНИЧЕСКАЯ КОНФЕРЕНЦИЯ С УЧАСТИЕМ ИНОСТРАННЫХ СПЕЦИАЛИСТОВ «ЦИФРОВЫЕ ТЕХНОЛОГИИ В ГОРНОМ ДЕЛЕ»



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Телефоны: (81555) 79567, (81555) 79268, факс: (81555) 74625

