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## COMPLEX OF METHODS TO EVALUATE INHIBITING PROPERTIES OF MUDS IN RELATION TO CLAY SWELLING ROCKS (AT EXAMPLE OF "REACTIVE" CLAYS OF MONTMORILLONITE GROUP OF KAZANIAN, TATARIAN AGES IN PERMIAN SYSTEM)

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КОМПЛЕКС МЕТОДОВ ОЦЕНКИ ИНГИБИРУЮЩИХ СВОЙСТВ БУРОВЫХ РАСТВОРОВ ПО ОТНОШЕНИЮ К ГЛИНИСТЫМ НАБУХАЮЩИМ ГОРНЫМ ПОРОДАМ (НА ПРИМЕРЕ «РЕАКТИВНЫХ» ГЛИН МОНТМОРИЛЛОНИТОВОЙ ГРУППЫ КАЗАНСКОГО, ТАТАРСКОГО ЯРУСОВ ПЕРМСКОЙ СИСТЕМЫ)

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inhibiting ability of muds, rocks of Kazanian, Tatarian age, lithomineralogical rock composition, degree of rock swelling, degree of rock erosion, colloidal particles concentration.

One of complication happening during construction of significant amount of wells in deposits of Denisovian basin of Timan-Pechora oil and gas province is loss of wellbore walls stability in intervals of "reactive" clays of Kazanian and Tatarian ages of Permian system. Complex lithological characterization of falling rocks within the named drilling intervals ratarian ages of Perman system. Complex intological characterization of falling focks within the named drilling intervals was performed using X-ray phase, X-ray fluorescence and litho-mineralogical analysis. In result of the study singularities of mineral and chemical composition of rocks and their clay component are determined. The article reviews the results obtained in diversified set of studies of muds inhibiting properties, including the following kinds of tests: study of rock swelling degree; tests to determine erosion of argillaceous slates in mud medium; evaluation of dispersing ability of muds; tests of fracture formation in rock samples in mud medium; study of character and intensity of ion exchange processes behavior in system crock – muds; study of alteration of rocks physical and chemical properties after influence of mud. According to results of the study gradation of different mud types according to their inhibiting ability in relation to clay swelling rocks is proposed.

Ключевые слова: ингибирующая способность буровых растворов, породы казанского, татарского ярусов, литолого-минералогический состав пород, степень набухания пород, степень эрозии пород, концентрация коллоидных частиц.

Одним из видов осложнений, возникающих при строительстве значительного количества скважин месторождений Денисовской впадины Тимано-Печорской нефтегазовой провинции, является потеря устойчивости стенок ствола в интервалах «реактивных» глин казанского и татарского ярусов пермской системы. С применением рентгенофазового, рентгенофлуоресцентного и литолого-минералогического анализов приведена комплексная литологическая характеристика обвальных пород указанных интервалов бурения. В результате изучения определены особенности минерального и химического состава горных пород и их глинистой составляющей. Рассмотрены результаты, полученные в ходе проведения разнопланового комплекса исследований интибирующих свойств буровых растворов, включающего следующие виды испытаний: исследование степени набухания пород; проведение теста на определение эрозии глинистых сланцев в среде буровых растворов; оценка диспергирующей способности буровых растворов; проведение теста на образование трещин в образцах породы в среде буровых растворов; изучение характера и интенсивности протекания ионообменных процессов в системе «порода – буровой раствор»; изучение изменения физико-механических свойств пород после воздействия буровых растворов. По результатам исследований предложена градация различных типов буровых растворов по ингибирующей

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### Introduction

One of most often complications during construction of wells in deposits of Denisovian basin of Timan-Pechora oil and gas province is loss of wellbore stability in intervals of Kazanian and Tatarian ages of Permian system. Well drilling through these intervals using fresh water acrylic muds, and also intended density increase in order to increase wellbore walls stability does not permit to fully resolve this problem.

With purpose to understand reasons of wellbore instability in intervals of Kazanian and Tatarian ages studies were performed to determine lithomineralogical composition and physical and chemical characteristics of rocks, and a complex of methods for evaluation of mud inhibiting properties is proposed, with purpose to establish inhibition mechanism being most effective for the studied rocks.

# Characteristic of "reactive" clays of montmorillonite group of Kazanian, Tatarian ages of Permian system

Rock of Kazanian, Tatarian ages of upper division of Permian system is presented by consolidated bondless clay, according to physical signs being at late diagenesis substage. Presence of psammite loose ingredients in rock composition demonstrates its low hardness. According to results of X-ray phase analysis in rocks of Kazanian, Tatarian ages presence of large concentrations of chlorite, hydromicas and montmorillonite is established, while in pellite rock fraction montmorillonite prevails (78,9 wt. %) [1]. According to results of X-ray fluorescent analysis it is established that rocks of Kazanian, Tatarian ages is characterized by increased contents of silica and reduced contents of magnesium. Sodium and potassium in rock composition are apparently presented by exchangeable ions, which in contact with water medium may cause alteration of composition, structure and stability of rocks [2, 3].

Prevailing content of montmorillonite in clay fraction determines high degree of swelling and rock dispersing – its high water sensitivity (so-called «reactivity») [4–6]. Characteristic feature of montmorillonite is weak attraction between packages in its structure, as only van der Waals forces act between them (weak intermolecular

interactions of dispersion, induction and orientation character). As result, water dipoles are able to freely penetrate into interplanar space of montmorillonite and expand it. In process of water saturation montmorillonite rocks loose strength, obtain plasticity and gradually transit flowing state. Another important factor influencing strength and deformational properties of weakly lithified clay rocks are forces of interaction between mineral particles - structural bonds. The higher is strength of structural bonds between separate elements of clay minerals, the higher is rock stability against hydrodynamic action of mud [7, 8].

## Objects of study

The performed complex of litho-mineralogical studies of rocks in the studied intervals permitted to substantiate methods of study necessary for reliable evaluation of inhibiting ability of muds. Complex methods being proposed provide for subsequent performance of six stages of study: comparative evaluation of inhibiting properties of muds at argillaceous slates linear swelling tester, evaluation of dispersing ability of muds by degree of clay rocks erosion and MBT (Methylene Blue Test) parameter, study of ion exchange processes in system «rock – mud» using inductively coupled plasma mass spectrometry method, evaluation of alteration of strength properties of clay rocks under influence of muds.

 $Table \ 1$  Types of muds studies (mud density  $-1230 \text{ kg/m}^3$ )

Mud #	Mud type	Sources describing peculiarities
		of mud usage
1	Fresh polymer clay mud on base of acrylic polymers $(FP_{0,7MPa} = 4,5 \text{ cm}^3/30 \text{ min}, pH = 8,7)$	[9–12]
2	Fresh polymer clay mud on base of acrylic polymers with addition of organosilicon fluid $(FP_{0.7MPa} = 4.5 \text{ cm}^3/30 \text{ min}, pH = 8.6)$	[13, 14]
3	Fresh biopolymer mud on base of polymer lignite reagent and glycols ( $FP_{0,7MPa} = 6.8 \text{ cm}^3/30 \text{ min}$ , pH = 10,4)	[15–18]
4	Potassium chloride biopolymer mud with inhibitor from polyamine class ( $FP_{0,7MPa} = 4,6 \text{ cm}^3/30 \text{ min}$ , $pH = 8,4$ )	[19–23]
5	Invert-emulsion mud (IEM) using as water phase calcium chloride solution with density 1,28 g/cm $^3$ (FP <sub>HPHT</sub> = 0,3 cm $^3$ /30 min, ES = 860 V, hydrocarbons/water ratio = 77/23)	[12, 24, 25]
6	IEM using as water phase CaCl <sub>2</sub> solution with density 1,39 g/cm <sup>3</sup> (FP <sub>HPHT</sub> = 0,3 cm <sup>3</sup> /30 min, ES = 467 B, hydrocarbons/water ratio = $60/40$ )	[26–28]

During evaluation of inhibiting properties of muds in relation to hydration of clays of Kazanian, Tatarian ages of Permian system following muds being currently used were studied, and also those muds considered promising for usage in well construction at deposits of Denisovian basin (table 1). As basis for comparison during evaluation of inhibiting properties of muds values of rock samples swelling and dispersing in technical and formation waters.

#### Results and discussion

Consolidated data on comparative evaluation of inhibiting properties of above stated types of muds in relation to red "reactive" clay of Kazanian, Tatarian ages of upper division of Permian system are given in table 2. Muds are designated according to table 1.

Main factors determining loss of stability in studied type of rock are processes of wetting, dispersing and weakening structural bonds between particles. In water saturation process montmorillonite rocks loose strength, plasticity and gradually transit to flowing state. One of most demonstrative parameters in evaluation of hydration degree of clay rocks with increased content of smectite (montmorillonite) minerals is degree of their swelling in medium of liquids under study. It is known that volume increase during swelling clay rocks depends on combination of adsorption, osmotic and capillary forces [14, 29-31]. That is why usage of this method permits to evaluate action of the depending on mud component above forces composition.

Table 2
Comparative evaluation of inhibiting properties of muds

Mud	Swelling degree, %	Erosion paramete r, %	Weakenin g coefficient	Rock dispersing degree, kg/m <sup>3</sup>	Current wetting speed, cm/h	
1	29,1	68,2	0,0067	7,6	0,298	
2	27,7	36,8	0,0303	0,8	0,366	
3	31,2	93,0	Complete destruction of sample	4,3	0,385	
4	17,0	10,0	0,550	2,5	0,24	
5	2,5	0	0,655	ı	0,062	
6	1,4	0	1,320	ı	0,054	
Technical water	45,8	Complete destruction of sample		7,7	Complete destruction of sample	
Formation water	32,1	Complete destruction of sample		4,3	Complete destruction of sample	

Results of study of rocks swelling dynamics in medium of muds in dynamic conditions at T = 55 °C (maximal formation temperature of Kazanian, Tatarian ages in deposits of Denisovian basin) confirmed that these rocks are prone to swelling and dispersing under action of water medium (within 72 h of experiment average value of longitudinal swelling of rocks from Kazanian, Tatarian age in technical water medium was 45,8 %). Presence of inorganic salts in water medium permits to significantly reduce rock swelling degree (swelling degree in formation water medium within 72 h of contact was 32,1 %). According to results of study it is established that at other equal conditions least inhibiting ability in relation to red "reactive" clays by swelling parameter is characteristic for fresh muds on base of acrylic polymers and glycols (muds 1 and 3 in table 1), addition of organosilicon compounds to mud recipe permits to somewhat increase inhibiting properties of fresh mud systems. The lowest parameter of swelling degree from the studied waterbased muds is noted for potassium chloride mud (mud 4 in table 1).

Another important factor influencing strength and deformational properties of weakly lithified clay rocks are forces of interaction between mineral particles – structural bonds [32–34]. The higher is strength of structural bonds between separate elements of clay minerals, the higher is rock stability to hydrodynamic action of mud. As prospective method of evaluation of wellbore walls stability in intervals of clay rocks at early and medium lithification stage to hydrodynamic action of specific types of muds may be separated test to determine erosion of argillaceous slates, which is performed at extended action of mud in dynamic conditions and permits to obtain information on mud ability to prevent dispersing, i.e., rock destruction in dynamic conditions [35]. During performing experimental studies with purpose to exclude eroding and dispersing water action during final washing which is made to determine loss of samples weight, washing rock samples from water-based mud rests was performed by saturated potassium chloride solution. It is necessary to note that this method of evaluation of mud inhibiting properties is not informative for hydrocarbon-based muds, as degree of clay rock particles dispersing in them, as a rule, does not exceed measurement error value.

In general, results of studies to determine erosion parameter of studied rocks fully agree with data on

Table 3

their swelling degree in water-based muds medium (see table 2). In process of hydrodynamic action of fresh glycol-based mud (mud 3 in table 1), probably, due to increased system pH value (pH = 10,4), practically complete rock weakening is observed (degree of sample weight loss was 93 %).

It is known that swelling and dispersing process of clay rocks with preemptive contents of montmorillonite to a great extent depends on their specific surface amount, and also on quantity and kind of exchangeable ions [9, 36, 37]. Study of ion exchange processes in system «rock – mud» using inductively coupled plasma mass spectrometry permits to evaluate alterations undergoing in rock, related to destabilization of clay crystal structure: ions desorption or oppositely rock saturation with ions and so-called rock «drying» (loss of bound water).

Inductively coupled plasma mass spectrometry is a method based on ionization of sample atoms by inductively coupled plasma with subsequent division of ions formed according to their atomic weight, this method is widely used to determine microquantities of metals and nonmetals in solutions, within this work sample analysis was performed at inductively coupled plasma mass spectrometer Aurora M90 by Bruker (USA).

Evaluation of character and intensity of ion exchange processes in system «rock – mud» was performed according to the following procedure. To equal volume mud samples same amount of milled sludge of falling rocks of Kazanian, Tatarian age was added. After this muds were hot rolled in roller-heart furnace at temperature 55 °C for 16 h. Character and intensity of ion exchange processes were evaluated according to difference in elements content in mud filtrate before and after placement of milled rock samples into muds. As base for comparison data on alteration of elements content in technical and formation waters before and after rock contact were used. Results of studies are given in table 3.

According to results of study it is established that clays of Kazanian, Tatarian age are characterized by undersaturation with main studied cations, which is demonstrated by direction of ion exchange processes between salt-containing liquids (formation water, potassium chloride mud) and studied rock, namely process of preemptive ion consumption by rock. To inhibit hydration of studied rocks promising are muds with increased electrolyte concentration, suppressing montmorillonite swelling processes and aiding to its

transition to less hydrophilic varieties, and also reagents decreasing clay sorption capacity. Usage of the named mud types will aid to transit clay mineral present in rock into less hydrated potassium forms.

Content of cations in mud filtrates before and after contact with rock of Kazanian, Tatarian age

) ( 1	Rock	Content of main cations, mg/dm <sup>3</sup>				
Mud	contact	K <sup>+</sup>	Na <sup>+</sup>	$Mg^{2+}$	Ca <sup>2+</sup>	
Distilled water	Before	Less than 0,5	2,89	0,265	2,51	
	After	52,4	309,0	1,36	20,0	
Formation water	Before	864,0	75170,0	4462,0	17618,0	
roillation water	After	907,0	69625,0	3939,0	16872,0	
	Before	197,0	1017,0	11,8	15,6	
Mud 1	After	280,0	1479,0	12,5	Less than 0,5	
Mud 2	Before	141,0	1435,0	8,5	Less than 0,5	
Mud 2	After	94,3	1610,0	11,2	Less than 0,5	
Mud 3	Before	219,0	2028,0	4,78	Less than 0,5	
	After	604,0	3290,0	13,0	35,6	
Mud 4	Before	55500,0	2318,0	87,5	319,0	
Iviuu 4	After	53510,0	2666,0	124,0	477,0	

According to results of study ion desorption in the maximal degree from clays of Kazanian, Tatarian age is noted for fresh glycol-based mud (mud 3), intensity of desorption of ions present in rock exceeds this parameter even for distilled water. Most probably, this fact is related to increased pH value of this mud recipe (pH = 10,4). Strongly alkaline reaction of environment aids in intensification of process of salts leaching from rock to contacting water medium.

At mud contact with rock alteration of rock and alteration of strain-stress state. consequently reduction of rock strength characteristics. First of all this is related to hydration swelling of clay minerals in composition of rock being drilled. But not less important are other mechanisms of weakening action of mud liquid phase, for example. its wedging action during penetration to surface micro fractures of monolith formation or its separate structural elements [38, 39].

Different methods are used for evaluation of rock strength properties and their alteration under influence of muds, most often measurement of rock sample surface hardness measurement by penetrometer, determination of rock strength by monoaxial compression using mechanical presses [40, 41]. These methods are rather effective for evaluation of mud inhibiting properties in case of availability of unbroken core material samples, but are seldom applicable for evaluation of alteration of strength properties of falling rocks sludge under influence of muds.

We propose the following complex method for evaluation of alteration of physical and chemical properties of rocks of Kazanian, Tatarian age under influence of various types of muds, including evaluation of muds wetting ability, alteration of fracture degree and strength of pressed samples of falling rock sludge. The method presumes pulverizing sludge samples to fraction size less than 160 µm, average sample preparation and pressing «tablets» using compactor included into longitudinal swelling tester set. The obtained «tablets» are placed into equal volumes of studied muds and are held in mud medium for 10 days (time of sample contact with muds should correspond to average time of rocks from problem drilling intervals staying in «uncased» condition). After that pressed samples are extracted from muds and following parameters are evaluated:

- current rock wetting speed (cm/h) in mud medium by alteration of sample weight according to formula stated in RD 39-00147001-773-2004 [42];
- visual test for alteration of degree of fractures in pressed samples after holding in mud medium;
  - mechanical strength of pressed rock samples.

Evaluation of mechanical strength of pressed rock samples after contact with studied muds under action of external load on samples is proposed to perform using texture analyzer CT3 made by Brookfield (fig. 1).

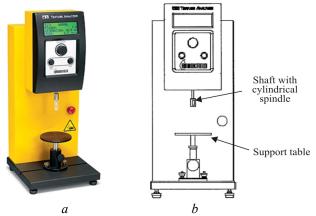


Fig. 1. Appearance (a) and schematic layout (b) of texture analyzer CT3 made by Brookfield

Study of mechanical strength of rock samples at texture analyzer CT3 provides for performance of the following: sample being studied is placed to analyzer support table; cylindrical spindle with certain geometric parameters is placed to the shaft (for all samples identical spindles should be used); experiment conditions set in instrument software; tests are performed in two sample compression cycles, with load measurement by dynamometric sensor, result of the experiment is dependency of load on sample/spindle in grams to experiment duration or to spindle travel (depth of penetration into rock sample), on basis of which using software following parameters are calculated: sample hardness (maximal load in first compression cycle) before and after mud influence, and peak stress corresponding to maximal load exerted per sample area unit in moment of its destruction. The latter parameter reflects compression strength of the studied sample. Influence of studied muds on strength of rock samples is proposed to be evaluated by value of weakening coefficient  $(K_p)$ , being ratio of peak stress values for pressed rock sample after influence of studied mud and initial dry sample.

Instrument software permits to build diagrams of alteration of load on rock samples depending on spindle travel (depth of penetration into rock sample), which assist not only to evaluate character of sample integrity disturbance under external load action visually, but also to characterize additionally adhesion properties of sample softened by action of muds being studied (fig. 2).

For example, from the given diagrams it is seen that rock sample held in mud 1 medium was momentarily destroyed at application of small load (80 g), meanwhile travel of load graph below zero mark demonstrates occurrence of adhesive properties of softened surface of sample. In rock sample held in mud 5 medium, other destruction character under load is observed, accompanied with appearance of internal fractures and gradual sample destruction only after application of rather significant load (over 10 kg).

Results of study regarding evaluation of physical and mechanical properties of rocks of Kazanian, Tatarian ages under influence of studied muds are given in table 2 and fig. 3.

It is established that studied type of clay rocks is characterized with high sorption capacity in relation to mud water phase: increase of rock sample weight in medium of studied water-based

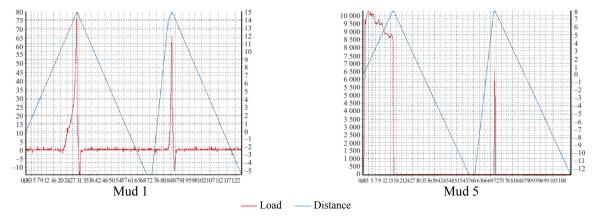


Fig. 2. Diagrams of alteration of load on rock samples depending on penetration depth

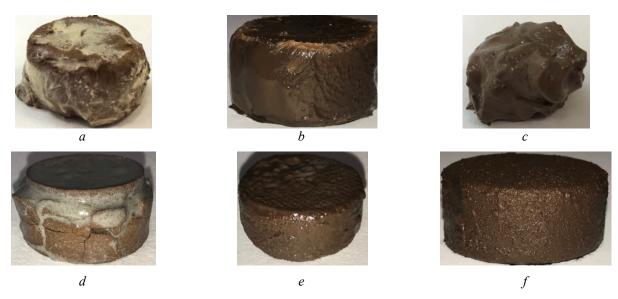


Fig. 3. Rock samples before and after mud influence: a - mud 1: significant sample softening with loss of initial form; b - mud 2: insignificant volume increase and sample softening; c - mud 3: significant sample softening with loss of initial form; d - mud 4: significant sample volume increase (swelling), appearance of several large fractures; e - mud 5: insignificant volume increase and sample surface softening; f - mud 6: no alteration of sample form and size is observed

muds is in range from 57 to 92 %. The lowest wetting ability from water system muds in relation to the studied rocks is with polyamine-based potassium chloride mud. This testifies that usage of this mud will aid to reduction of wetting intensity of near-wellbore area, and, as result, well walls stabilization. The most wetting ability is characteristic for fresh glycol-based mud. This permits to conclude that usage of glycols as clay hydration inhibitor will not help to preserve mechanical strength of studied rocks in near-wellbore area.

Usage of organosilicon compounds in the fresh mud recipe, seemingly, due to their hydrophobizing effect, permits to somewhat reduce degree of weakening pressed rock samples in mud medium.

According to results of study it is established that the proposed complex method permits to substantiate component composition not only for water system muds used to drill-in clay sediments being studied, but for invert-emulsion muds also. Despite the fact that majority of methods confirmed practically complete IEM inertness in relation to clays of Kazanian, Tatarian age, it is discovered that component composition of mud water phase effects significant influence on its inhibiting ability in relation to hydration of clay rocks (see table 2). Most prominently differences between inhibiting ability of IEM on base of saturated (mud 6) and non-saturated calcium chloride solution (mud 5) showed themselves during study of alteration of physical and mechanical rock properties in mud medium.

Fresh mud on base of polymer-lignite reagent and glycols reagent and glycols Fresh polymers with organosilicon liquid

Fresh polymer-clay mud on base of acryl polymers with organosilicon liquid

Potassium chloride biopolymer mud with inhibitor from polyamine class

Potassium chloride biopolymer mud with inhibitor from polyamine class

Fig. 4. Scale of inhibiting ability of studied types of muds in relation to red "reactive" clay of Kazanian, Tatarian ages of upper division of Permian system (arrow direction points increase of inhibiting properties of muds)

In particular, strengthening of rock samples is observed in medium of IEM based on saturated calcium chloride solution. This result, most probably, is related to partial filling of voids in samples by oil phase of mud, which due to its low compressibility takes a part of load during compression and permits more even distribution of skeletal stresses on the whole area of rock [43]. Usage solution saturated with calcium ions as water phase permits to obtain IEM with low water phase activity ( $a_w = 0.39$ ) and to minimize osmotic processes in system «mud – rock». According to results of study it is established that in order to drill a well through clay sediments of Kazanian, Tatarian ages in deposits with difficult geological drilling conditions, where most severe complications are registered, related to loss of stability of wellbore walls, it is possible to recommend usage of water-based invert-emulsion muds, saturated with calcium ions.

### Conclusions

Consequently, complex of methods for evaluation of inhibiting properties of muds,

presented in the article, permits to perform allaround analysis of physical and chemical interaction in system «rock – mud» with regard to lithological composition and properties of clay rocks in problem drilling intervals. Complex of methods most closely resembles well conditions due to modeling temperature, exposure time, and usage of natural core material in studies.

IEM on base of

calcium chloride

solution with density

 $1,39 \text{ g/cm}^3$ 

Method of evaluation of rock strength parameters using texture analyzer CT3 may be used as indicator of alterations in physical and chemical rock properties under influence of studied liquids and is recommended for usage as one of advanced methods for evaluation of inhibiting properties of muds when it is possible to use only desegregated sludge of falling rocks as core material.

By aggregate of data received using the discussed complex method, gradation of muds per their inhibiting ability in relation to red "reactive" clays of Kazanian, Tatarian ages is proposed, which will be further considered during selection of mud type for drilling wells through the studied clay rocks (fig. 4).

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Комплекс методов оценки ингибирующих свойств буровых растворов по отношению к глинистым набухающим горным породам (на примере «реактивных» глин монтмориллонитовой группы казанского, татарского ярусов пермской системы) / И.Л. Некрасова, П.А. Хвощин, Д.А. Казаков, О.В. Гаршина, Г.В. Окромелидзе, Д.В. Тирон // Вестник Пермского национального исследовательского политехнического университета. Геология. Нефтегазовое и горное дело. − 2019. − Т.19, №2. − С.150−161. DOI: 10.15593/2224-9923/2019.2.5