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Existing Approaches to the Tight Rock Laboratory Petrophysics: a Critical Review

D V Konoshonkin, S V Parnachev
Petroleum Learning Centre
Tomsk Polytechnic University
Tomsk, Russia

***Abstract*—Review of the existing methods for tight rock porosity, saturation, and permeability determination was performed taking into account that these methods should be applicable for Bazhenov formation evaluation. Following methods are considered: Archimedes mercury immersion; mercury displacement; caliper; helium pycnometry on crushed sample; nuclear magnetic resonance; modified retort method; modified Dean-Stark extraction; pulse decay method; and pressure decay test on crushed sample. Applicability of pressure decay test on crushed sample for the Bazhenov formation evaluation is checked practically using commercial permeameter SMP-200. All the methods were combined into five protocols for tight rock petrophysical evaluation. These protocols were analyzed and compared, according to following criteria: accuracy of the results; usage experience; time for measurements; easiness of interpretation; reliability and safety; price.**

Keywords—tight; rock; mercury; pycnometry; resonance; Bazhenov; porosity; permeability; protocol

I. INTRODUCTION

World oil demands constantly rise while traditional oil resources running out. These conditions make oil companies to take interest in unconventional resources investigation. According to energy agency of the USA and according to Russian researches, in Russia, Bazhenov formation has the highest unconventional resources potential. Thus, Bazhenov formation becomes intensively studied. However, traditional methods of core analysis are not effective for Bazhenov formation tight rocks evaluation. Therefore, there is an exigency to review existing approaches of the tight rock analysis in order to propose the most effective one for Bazhenov formation core evaluation.

II. LITHOLOGY AND PETROPHYSICAL PROPERTIES OF BAZHENOV FORMATION

Bazhenov formation is a geological body composed of shale, silica and carbonate rocks of different proportions and with different amount of organic matter [1,2]. Bazhenov formation consists of tight rocks with porosity less than 16% and permeability usually less than 0.1 mD (TABLE I), but there are "sweet spots" with higher permeability.

III. REVIEW OF THE PUBLISHED MATERIALS ON THE TIGHT ROCK LABORATORY TESTING METHODS

TABLE I. AVERAGE PETROPHYSICAL PROPERTIES OF BAZHENOV FORMATION

Parameter	Value
Porosity	1.4 – 16 %
Permeability	10-6 – 10 mD Predominantly < 0.1 mD
Total organic content (TOC)	5 – 20 %
Thickness	20 – 40 m

A. *Sample preservation*

There is a lot of dissociation about tight rock sample preservation. Zhou, et al. [3] claimed that hydrocarbon bearing shale core should be preserved and core samples are to be stored in desiccators prior to measurements. The main reason for this was the reaction of shale with atmospheric water and changes of core saturation and properties. Unfortunately Zhou, et al. did not describe types and properties of shale for which they recommended preservation.

On the other hand, Handwerger, et al. [4] showed that in case of tight rocks (experiment was conducted on samples with porosity 2.2 – 2.4% and permeability $1.1 \cdot 10^{-4}$ – $1.8 \cdot 10^{-4}$ mD) lab analysis results for porosity, saturation and permeability measurements made on recently drilled core are equal to lab analysis results for the same core after 2 years storage in plastic bags without any special storage protocols. Therefore, Handwerger, et al. concluded that petrophysical properties of tight rock samples (especially important for saturation) change insignificantly due to irreducible nature of saturating fluids in ambient conditions. This is very important conclusion, because concerning Bazhenov formation there is a lot of core which was drilled years ago and was not studied yet.

B. *Porosity determination*

Core sample of Bazhenov formation rock may be represented by the model showed in Fig. 1. Three volumes are distinguished according to that model: bulk volume (V_b), grain volume (V_g) and pore volume (V_p). Porosity is a fraction of the pore volume to the total volume of the rock and may be determined using following equation:

$$\varphi = (V_b - V_g)/V_b = V_t/(V_t + V_g) = V_t / V_b \quad (1)$$

Therefore, in order to determine porosity two of three parameters (V_b, V_t, V_g) should be determined in a laboratory.

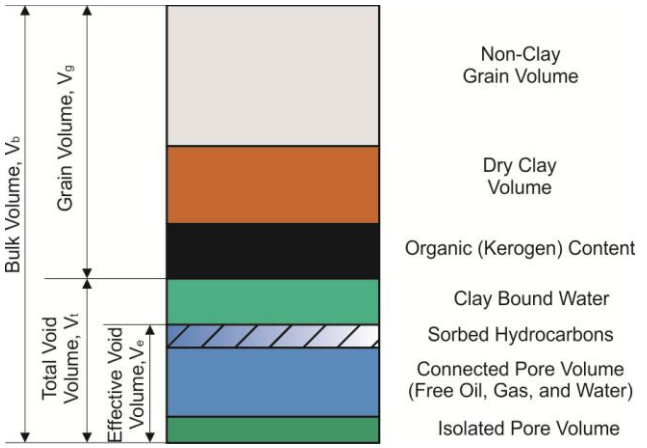

Fig. 1. Model of kerogen rich tight rock [5].

1) Methods of bulk volume determination

There are three main methods for bulk volume determination of tight rock samples (TABLE II) [6]. Two of the methods (Archimedes mercury immersion and mercury displacement) are based on mercury usage. Mercury is used because it does not penetrate into sample pores, it does not change sample saturation and does not react with sample's components (due to its high surface tension and low wettability). Mercury based methods allow fast and accurate bulk volume measurements but cannot be applied for samples with surface fractures and vugs.

Third method (caliper) implies direct sample dimensions measurements. It is very simple and fast but the method cannot be used for samples with irregular shape. Also porosity results have high uncertainty if caliper method is used with grain volume measurement to obtain porosity (1).

2) Methods of grain volume determination

Grain volume of tight rock may be determined using double-cell helium pycnometry method (TABLE II) on crushed sample. The method is relatively simple and gives almost the same results for the same sample (high repeatability). However, cells should be accurately calibrated, temperature fluctuations should be reduced or accounted and adsorption and molecular sieving effect should be taken into account (adsorption effect is significant for gases other than helium, for example, methane).

3) Methods of pore volume determination

In order to determine pore volume of a tight rock sample nuclear magnetic resonance (NMR) may be used (TABLE II).

The NMR method based on hydrogen nuclei precession in magnetic field. As hydrogen presents predominantly in pore fluids, nuclear magnetic resonance may be used to determine quantity of fluids in pore space and, thus, the pore volume.

C. Saturation determination

Saturation of the tight rock sample may be determined using one of the following methods (TABLE III):

- Modified retort method at atmospheric pressure;
- Modified Dean-Stark method using toluene extraction;
- Magnetic resonance saturation scan.

TABLE II. METHODS OF TIGHT ROCK BULK, GRAIN, AND PORE VOLUMES DETERMINATION

Method	Major advantages	Major disadvantages	Accuracy
Archimedes (Buoyancy) Mercury Immersion (bulk volume)	The method is very accurate.	Trapping air around the samples; Samples with a vugular surface or containing open fractures cannot be used.	±0.01 cm^3 (using balance with 0.01 g accuracy).
Mercury Displacement (bulk volume)	Rapid measurements.	Trapping air around the samples; Samples with a vugular surface or containing open fractures cannot be used.	±0.01 cm^3 (if the pump has been calibrated and is zeroed for each sample).
Caliper (bulk volume)	Rapid measurements.	Only for even shape samples (e.g. core plugs); Higher errors for porosity obtained from grain volume.	±0.15 cm^3 (in case of ±0.15 mm for length and ±0.04 mm for diameter measurements)
Helium pycnometry on crushed sample (grain volume)	No damage for sample; Simple and quick; High repeatability.	Changes in ambient pressure and/or temperature may induce errors; Adsorption and sieving effects.	±0.2% of the true value (for well calibrated system).
NMR (pore volume)	Sample lithology does not affect measurements; Rapid method.	Indirect method; Sample should be saturated by one fluid for high accuracy.	±20% but depends on measurement conditions.

Retort method [4] is direct method of fluid volume determination which may be performed in sufficiently short time (about 1 day) and may be used to separate mobile (free) and bound water and oil. However, the method destroys the sample and may give erroneous results in case of high amount of montmorillonite, gypsum or kerogen.

Dean-Stark extraction [7] is thought to be applicable method in case of high kerogen content (nitrogen gas blanket flowing through the apparatus should be used [8]). However, Handwerger, et al. [9] showed that some water may be extracted from clay and cause water saturation exaggeration. Also method implies long time for measurements (about two weeks).

Magnetic resonance measurement [10,11,12] is the only method which does not destroy by any means the core plug. Unfortunately, the method is indirect and there are difficulties and uncertainties in fluid response separation.

D. Permeability determination

Permeability may be determined by means of steady state methods and unsteady state methods. However, steady state methods are usually not used for tight rocks because they need too much time for measurements and because of necessity to measure very low flow rates [6,13]. Therefore, predominantly

unsteady state methods are used for permeability measurements in tight rocks.

There are two widely used unsteady state laboratory methods for permeability measurements in tight rocks [13] (TABLE IV):

TABLE III. METHODS OF TIGHT ROCK SATURATION DETERMINATION

Method	Major advantages	Major disadvantages	Accuracy
Modified retort method at atmospheric pressure.	Direct measurement of fluid volumes; Ability to separate free from bound water; Rapid (1 day is required).	Errors for samples with montmorillonite or gypsum; Errors for samples with high kerogen; Sample cannot be used further.	For water: ±5% of measured volume For oil: ±2.5% of measured volume.
Modified Dean-Stark method.	Sample material can be used for further testing; Simple and requires little attention during distillation; Applicable for kerogen rich samples.	Salt may precipitate inside the sample; Errors for samples with montmorillonite or gypsum; Oil density should be known; Long (a week is required for extraction and a week for drying).	±50% of measured volumes (in case of relatively small samples or samples containing high gas saturation with residual volumes of oil and water).
Magnetic resonance saturation scan.	The only method for saturation determination on core plug which does not destroy sample.	Indirect method; Difficulties for fluid response separation.	Depends on differences of NMR properties of measured fluids.

- Pressure pulse on core plug;
- Pressure decay on crushed rock.

Pressure pulse on core plug allows measuring permeability anisotropy at reservoir conditions; however, obtained permeability may be exaggerated because of presence of micro fractures generated during coring. Core crushing (pressure decay on crushed rock method) reduces influence of micro fractures, but measurements are made at ambient temperature and pressure.

IV. DETERMINATION OF PERMEABILITY AND MATRIX VOLUME OF BAZHENOV FORMATION SAMPLE

Applicability of pressure decay test on crushed sample for the Bazhenov formation evaluation is checked practically using commercial permeameter SMP-200.

A. *Sample preparation*

Core sample was not by any means cleaned but was dried in vacuum oven at temperature 60°C. Then it was cut and then crushed using geologic hummer and mortar.

B. *Running of the experiment*

Experiment was run using permeametr SMP-200. Firstly, reference cell calibration and dead volume calibration were made. After that, leak off test was conducted. Next, one of the disks (disk which was almost equal to the volume of crushed sample) was removed from sample chamber and sample was placed into the sample chamber. Then, helium at pressure about 1400 kPa (200 psi) was expanded into sample chamber. Pressure decay curve was recorded using pressure gauge (accuracy 6.9 Pa (0.001 psi)) within 2000 seconds. Permeability was determined by matching simulated and measured curves.

TABLE IV. UNSTEADY STATE METHODS FOR PERMEABILITY DETERMINATION OF TIGHT ROCKS.

Method	Perm range, mD	Major advantages	Major disadvantages	Accuracy
Axial flow, pulse-decay in core plugs	10^{-5} – 10^{-1}	Confining and pore pressures are applied; Permeability anisotropy can be measured.	Requires high pressure, leak-tight with high quality transducers and data acquisition system – high capital cost; Permeability maybe affected by micro-cracks.	3% (low/no leaks, adsorption is accounted, low/no temperature fluctuations present)
Pressure-decay on crushed sample	Gas: 10^{-5} – 10^{-2} Liquid: 0.1 – 2000	Elimination of micro-cracks.	No confining pressure; Low repeatability of measurements; Difficulties with slip-correction.	±10% (low/no leaks, adsorption is accounted, low/no temperature fluctuations present)

C. *Results*

Results of permeability measurements are presented in (TABLE V). It can be noticed that permeability for sample 1 and sample 2 differs on 10.7%.

Following disadvantages of SMP-200 were determined during experiment:

- Modeled curve has low correlation with measured data. It can be explained by poor quality of theoretical model. It should account for deviation from Darcy's flow due to comparable sizes of pores and helium molecules and also it should account for temperature fluctuations.
- Permeameter SMP-200 has not enough thermal protection. Temperature influence on pressure measurements was determined during leak off test. Negative values of leak off were determined. It may be explained by temperature growth which causes pressure increase in sample chamber.
- Possibility to choose pressure curve part which is then correlated with simulated pressure decay reduces results repeatability. Because of low correlation between theoretical and measured pressure decays, choice of different parts of pressure curve leads to different results of permeability which may differs on 20%.

V. PROTOCOLS OF TIGHT ROCK ANALYSIS

A. *Protocols description*

All of the above methods may be combined into five protocols of porosity, saturation, and permeability determination of tight rocks.

TABLE V. RESULTS OF THE EXPERIMENT.

Parameter	Sample		
	Sample 1	*Sample 2*	*Difference, %*
Particle size, mm	2-5	1-2	160
Sample weight, g	44.8193	30.0360	33
Grain volume, cm^3	17.772	11.813	33.5
Grain density, g/cm^3	2.522	2.543	0.8
Permeability, mD	$1.1662 \cdot 10^{-7}$	$1.0410 \cdot 10^{-7}$	10.7

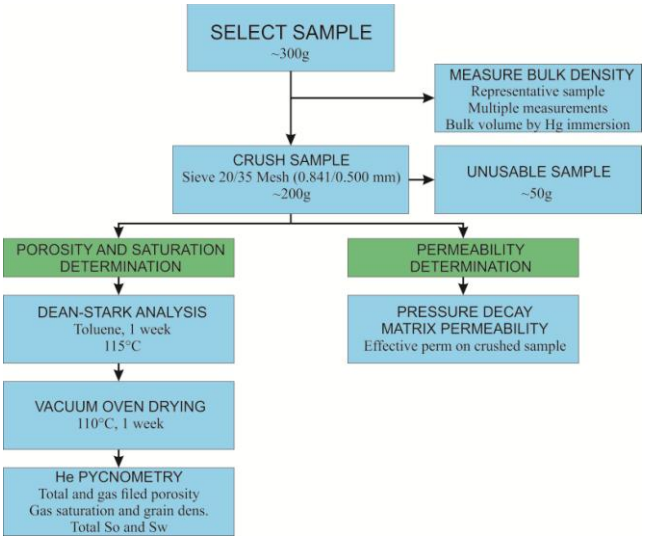

Fig. 2. First protocol of porosity, saturation, and permeability determination of tight rocks (GRI).

First protocol is Gas Research Institute (GRI) which is used for the crushed rock analysis (Fig. 2). In this protocol porosity is determined through bulk and grain volumes obtained from mercury immersion method and helium pycnometry method, respectively. Saturation is found using modified Dean-Stark extraction. Permeability is determined using pressure decay on crushed sample.

Second protocol is used by TerraTek company and called Tight Rock Analysis (TRA) (Fig. 3). In this protocol instead of modified Dean-Stark extraction (as in first protocol) modified retort analysis is used for saturation determination. Also it is important that helium pycnometry is performed before oil and water volume determination (before extraction or drying core sample). Therefore, in second protocol opposite to GRI protocol after helium pycnometry gas field volume is determined. This excludes mistakes which may be caused in GRI protocol if core sample is not cleaned after drying and salts are precipitated reducing pore space. As for the rest, second protocol (TRA) is similar to first protocol (GRI).

Third and fourth protocols imply permeability determination by pulse decay test on core plug, crushing the core plug and further determination of porosity and saturation using Dean-Stark (*third protocol*) and retort analysis (*fourth protocol*) with helium pycnometry similar to GRI and TRA protocols, respectively (Fig. 4).

Fifth protocol involves nuclear magnetic resonance methods for saturation and porosity determination and pulse test for permeability determination (Fig. 4). This protocol allows measurements without core crushing or destroying.

B. *Protocol selection*

In order to choose the most effective protocol for tight rock analysis (and thus for Bazhenov formation) marketability analysis of all the five protocols was made. The main result of performed analysis is marketability evaluation map (TABLE VI).

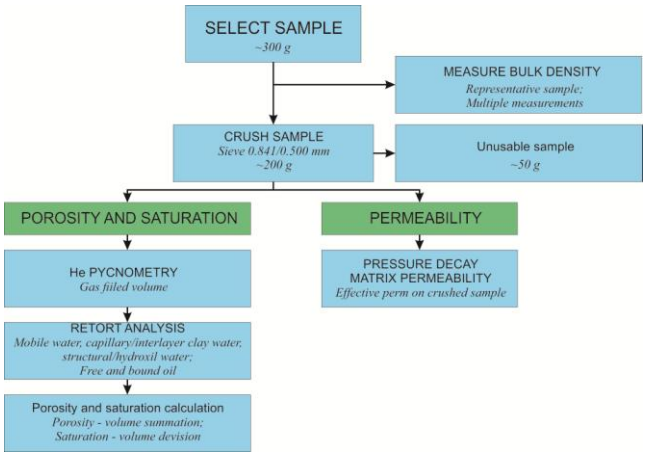

Fig. 3. Second protocol of porosity, saturation, and permeability determination of tight rocks (TRA).

Marketability of every protocol for every criterion was defined according to the expert judgment and using five-point grading scheme: 1 – the weakest position, 5 – the strongest position. Matrixes of quantitative relations were made in order to increase objectiveness of grading (TABLES VII-IX).

In the matrixes of quantitative relations:

- "0.5" means that protocol in the column is inferior of protocol in the raw;
- "1" means that protocol in the column is equal to protocol in the raw;
- "1.5" means that protocol in the column is more effective than protocol in the raw.

For example, in case of porosity and saturation determination usage experience of protocol 1 (GRI) is higher than usage experience of protocol 2 (TRA).

Contribution was calculated using following equation:

$$Contribution = Sum_i / Sum_{total} \quad (2)$$

Contributions for porosity, saturation and permeability were summed and every protocol was graded according to total contribution value.

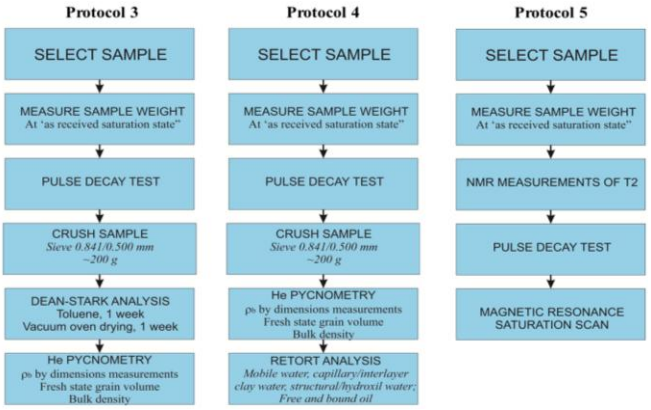

Fig. 4. Third protocol (left) fourth protocol (middle) and fifth protocol (right) of porosity, saturation, and permeability determination of tight rocks.

TABLE VI. EVALUATION MAP FOR FIVE PROTOCOLS OF TIGHT ROCK ANALYSIS.

Criteria	Weight of criterion	Marketability				
		Protocol				
		1	*2*	*3*	*4*	*5*
Accuracy of the results	0.35	0.7	1.05	1.05	1.75	1.4
Usage experience	0.25	0.75	0.25	1.25	1	0.5
Time for measurements	0.2	0.4	0.8	0.2	0.8	1
Easiness of interpretation	0.1	0.5	0.5	0.5	0.5	0.3
Reliability and safety	0.05	0.2	0.2	0.15	0.15	0.15
Price	0.05	0.25	0.2	0.15	0.1	0.05
Total	1	2.8	3	3.3	**4.3**	3.4

Marketability of the protocols was determined using following equation:

$$M = \sum(W_i G_i) \quad (3)$$

where M – marketability of the protocol; W_i – weight of criterion; G_i – grade.

Marketability results reviled that **the most effective protocol is protocol 4** (modified retort analysis and helium pycnometry for porosity and saturation determination and pulse decay on core plug for permeability determination). High efficiency of *protocol 4* is caused by high grades of this protocol in following criteria.

1) Accuracy of the results

Protocol 4 gives the most accurate results of porosity, saturation and permeability determination. Application of retort analysis in protocol 4 allows direct measurements of fluid saturating core sample which reduces uncertainties caused by calculations. Also retort analysis was shown to give more accurate results than modified Dean-Stark extraction (protocols 1 and 3) because modified Dean-Stark extraction systematically exaggerates obtained water volume [9]. Also fourth protocol excels protocol 5 because NMR used in protocol 5 is not direct method of porosity and saturation determination and it is exposed to uncertainties caused by longitudinal relaxation time (T_1), transverse relaxation time (T_2), and diffusion coefficient (D) overlapping for different fluids and subsequent uncertainties in saturation determination. Furthermore permeability determined by pulse decay (protocols 3, 4, 5) on core plug is more accurate than permeability determined on crushed sample (protocols 1, 2) in case of confining pressure sufficient for micro fractures closing. Pulse decay is made at reservoir conditions and account flow direction while pressure decay on crushed rock is made at ambient conditions.

TABLE VII. MATRIX OF QUANTITATIVE RELATIONS FOR CRITERION «USAGE EXPERIENCE» FOR FIVE PROTOCOLS IN CASE OF POROSITY AND SATURATION DETERMINATION.

Protocol	Protocol					Sum	Contribution
	1	*2*	*3*	*4*	*5*		
1	1	1.5	1	1.5	1.5	6.5	0.26
2	0.5	1	0.5	1	1.5	4.5	0.18
3	1	1.5	1	1.5	1.5	6.5	0.26
4	0.5	1	0.5	1	1.5	4.5	0.18
5	0.5	0.5	0.5	0.5	1	3	0.12
Total						25	1

TABLE VIII. MATRIX OF QUANTITATIVE RELATIONS FOR CRITERION «USAGE EXPERIENCE» FOR FIVE PROTOCOLS IN CASE OF PERMEABILITY DETERMINATION.

Protocol	Protocol					Sum	Contribution
	1	*2*	*3*	*4*	*5*		
1	1	1	0.5	0.5	0.5	3.5	0.14
2	1	1	0.5	0.5	0.5	3.5	0.14
3	1.5	1.5	1	1	1	6	0.24
4	1.5	1.5	1	1	1	6	0.24
5	1.5	1.5	1	1	1	6	0.24
Total						25	1

TABLE IX. SUM OF CONTRIBUTIONS AND GRADING OF THE PROTOCOLS FOR CRITERION "USAGE EXPERIENCE".

Protocol	Contribution		Sum	Grade
	for saturation	*for permeability*		
1	0.26	0.14	0.4	3
2	0.18	0.14	0.32	1
3	0.26	0.24	0.5	5
4	0.18	0.24	0.42	4
5	0.12	0.24	0.36	2

2) Usage experience

Usage experience of protocol 4 is only lower than usage experience of protocol 3 (modified Dean-Stark extraction, helium pycnometry and pressure decay on crushed rock). High usage experience of protocol 4 is caused by numerous researches made for permeability determination of tight rocks using pulse decay on core plug. Application of pressure pulse on core plug began in 1968 [14] while application of pressure decay on crushed sample began in 1993 [15]. On the other hand, protocols 2, 4 have lower usage experience than protocols 1, 3 for porosity and saturation determination. It is caused by the fact that modified retort analysis for tight rock evaluation began in 2011 when Handwerger, et al. [4] proved its applicability, while modified Dean-Stark extraction is used from 1992 [7]. Application of NMR for tight rock analysis (protocol 5) began only recently [10,11,12].

3) Time for measurements

In time criterion protocol 4 is only inferior of protocol 5 to obtain porosity, saturation and permeability results. This caused by relatively long time necessary for modified retort analysis. On the other hand, this drawback of protocol 4 is reduced by possibility to perform modified retort analysis for several samples at a time.

4) Easiness of interpretation

All of the five protocols except protocol 5 are comparable in interpretation complexity. Protocol 5 includes NMR method which implies mathematical inversion process, creation and analysis of D-T_2 maps and, thus, implicates high quality specialist and complex interpretation process.

5) Reliability and safety

Reliability and safety of protocols were evaluated according to the complexity of used apparatuses. Apparatus for porosity and saturation determination are assumed to have the same complexity. Apparatus for permeability determination using pressure pulse includes additional tools for confining and pore pressure application in comparison to pressure decay apparatus. Therefore, protocols 3, 4, 5 are less reliable than protocols 1, 2.

6) Price

Low grade of protocol 4 caused by higher price of modified retort apparatus in comparison to modified Dean-Stark apparatus and higher price of pulse decay apparatus in comparison to pressure decay apparatus.

7) Scheme for determination of the most effective protocol for tight rock petrophysical properties evaluation

There are cases when proposed protocol 4 is less effective than other protocols. Therefore, in order to choose the most effective protocol for tight rock petrophysical properties evaluation in definite conditions, scheme (Fig. 5) is suggested.

VI. CONCLUSION

The most effective protocol for porosity, saturation, and permeability determination of tight rocks of Bazhenov formation is **protocol 4** that includes pressure pulse on core plug for permeability determination; helium pycnometry and modified retort analysis on crushed sample for porosity and saturation determination.

If NMR is used in logging or if core is crushed during coring process or if retort cannot be applied for analysis, scheme is suggested to choose the most effective protocol for tight rock petrophysical properties evaluation.

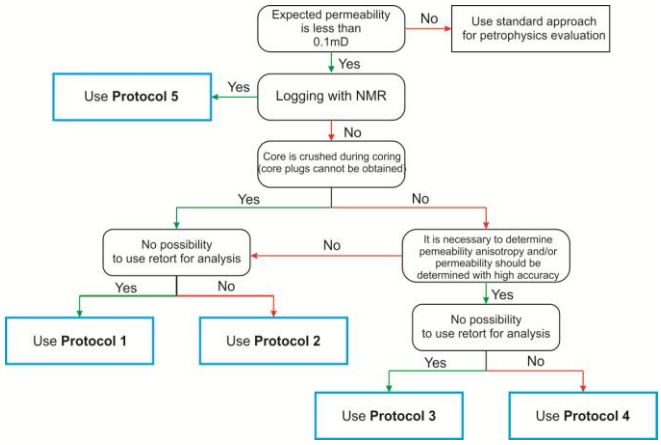

Fig. 5. Scheme for determination of the most effective protocol for tight rock petrophysical properties evaluation in definite conditions.

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