Existing approaches to tight rock laboratory petrophysics: a critical review

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Abstract. A 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. The following methods were considered: Archimedes mercury immersion; mercury displacement; caliper; helium pycnometry on crushed samples; nuclear magnetic resonance; modified retort method; modified Dean-Stark extraction; pulse decay method; and pressure decay test on crushed samples. The applicability of the pressure decay test on a crushed sample for Bazhenov formation evaluation is checked experimentally with the SMP-200 commercial permeameter. All the above listed methods were combined into five protocols for tight rock petrophysical evaluation. These protocols were analyzed and compared according to the following criteria: accuracy of the results; usage experience; time of measurements; easiness of interpretation; reliability and safety; price. The obtained results revealed that the most effective protocol is the one that includes pressure pulse on a core plug for permeability determination, He pycnometry and modified retort analysis on crushed samples for porosity and saturation determination. As there were cases when the proposed protocol was less effective vs. other protocols, a special scheme was suggested in order to choose the most effective protocol for tight rock petrophysical properties evaluation in definite conditions.

1. Introduction

The demand for oil is constantly rising in the world, whereas traditional oil resources are becoming depleted. These conditions make oil companies take interest in unconventional resource studies. According to the US Department of Energy and Russian researchers, the Bazhenov formation has the highest unconventional resource potential in Russia. Thus, the Bazhenov formation is becoming intensively studied. However, the traditional methods of core analysis are not effective for evaluating the tight rocks of the Bazhenov formation. Therefore, there is an exigency to review the existing approaches to analyzing tight rocks in order to propose the most effective one for Bazhenov formation core evaluation.

2. Lithology and petrophysical properties of the Bazhenov formation

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

Table 1. Average petrophysical properties of the 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

3. Review of published materials on tight rock laboratory testing methods

3.1. Sample preservation

There is much controversy about tight rock sample preservation. Zhou, et al. [3] claimed that a hydrocarbon-bearing shale core should be preserved and core samples are to be stored in desiccators prior to measurements. The main reason for that was the reaction of shale with atmospheric water and changes in core saturation and properties. Unfortunately, Zhou, et al. did not describe the 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 (an experiment was conducted on samples with porosity being 2.2-2.4% and permeability of $1.1 \cdot 10^{-4}$ – $1.8 \cdot 10^{-4}$ mD) the results of the lab analysis for porosity, saturation and permeability measurements made on recently drilled core are equal to the results of the lab analysis for the same core after its 2-year storage in plastic bags without any special storage protocols. Therefore, Handwerger, et al. concluded that the petrophysical properties of tight rock samples (especially important for saturation) change insignificantly due to the irreducible nature of fluid saturation in ambient conditions. It is a very important conclusion, because there are a lot of core samples from the Bazhenov formation that were drilled several years ago and have not been studied yet.

3.2. Porosity determination

The core sample of the Bazhenov formation rock may be represented with the model showed in (figure 1). Three volumes are distinguished according to that model: bulk volume (V_b), grain volume (V_g) and pore volume (V_t). Porosity is the ration of the pore volume to the total volume of the rock and may be determined with the following equation:

$$ \overline{\qquad} \quad \overline{\qquad} \quad \overline{\quad} \tag{1}$$

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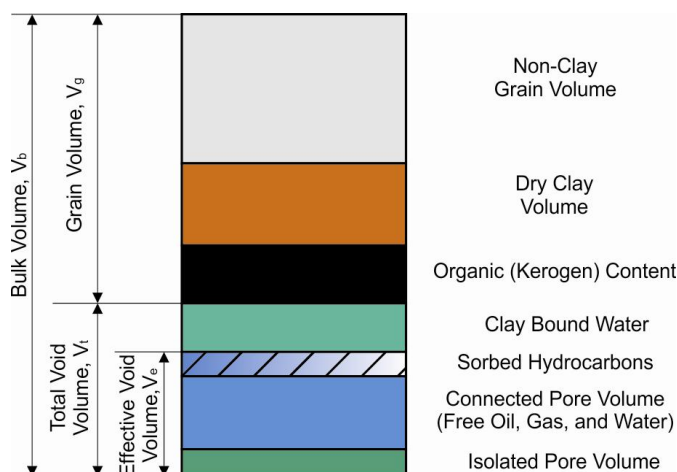

Figure 1. Model of kerogen-rich tight rock based on [5].

3.2.1. Methods of bulk volume determination

There are three main methods for bulk volume determination in tight rock samples (table 2) [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 components (due to its high surface tension and low wettability). Mercury-based methods allow performing fast and accurate bulk volume measurements but cannot be applied to samples with surface fractures and vugs.

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

Table 2. Methods of tight rock bulk volume determination.

Method	Major advantages	Major disadvantages	Accuracy
Archimedes (buoyancy) mercury immersion	Samples can be used for subsequent tests; 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	Rapid measurements; samples are suitable for subsequent tests	Trapping air around the samples; samples with a vugular surface or containing open fractures cannot be used	± 0.01 cm^3 (if the pump was calibrated and is zeroed for each sample)
Caliper	Samples can be used for subsequent tests; rapid measurements	Only for samples of an even shape (e.g. core plugs); higher level of errors for porosity derived from grain volume	± 0.15 cm^3 (in case of ± 0.15 mm for length and ± 0.04 mm for diameter measurements)

3.2.2. Methods of grain volume determination

The grain volume of a tight rock may be determined using the double-cell helium pycnometry method on crushed samples (table 3). 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 the molecular sieving effect should be taken into account (adsorption effect is significant for gases other than helium, for example, methane).

Table 3. Method of tight rock grain volume determination.

Method	Major benefits	Major drawbacks	Accuracy
Helium pycnometry on crushed samples	No damage to the sample; simple and quick; high repeatability	Changes in ambient pressure and/or temperature may induce errors; adsorption and sieving effects	$\pm 0.2\%$ of the true value (for well calibrated system)

3.2.3. Methods of pore volume determination

In order to determine the pore volume of a tight rock sample, nuclear magnetic resonance (NMR) may be used (table 4). The NMR method is based on hydrogen nuclei precession in a magnetic field. As hydrogen is present predominantly in pore fluids, nuclear magnetic resonance may be used to determine the quantity of fluids in pore space and, thus, the pore volume.

Table 4. Method of tight rock pore volume determination.

Method	Major benefits	Major drawbacks	Accuracy
NMR	Sample lithology does not affect measurements; rapid method	Indirect method; samples should be saturated with one fluid for high accuracy	$\pm 20\%$, but depends on measurement conditions

3.3. Saturation determination

The saturation of a tight rock sample may be determined using one of the following methods (table 5):
1) modified retort method at atmospheric pressure;
2) modified Dean-Stark method using toluene extraction;
3) magnetic resonance saturation scan.

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

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

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

Table 5. Methods of tight rock saturation determination.

Method	Major benefits	Major drawbacks	Accuracy
Modified retort method at atmospheric pressure	Direct measurement of fluid volumes; ability to separate free water from bound water; rapid (1 day is required)	Errors for samples with montmorillonite or gypsum; errors for samples with a high kerogen content; samples cannot be used further	For water: ±5% of the measured volume For oil: ±2.5% of the 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 can precipitate inside the sample; errors for samples with montmorillonite or gypsum; oil density should be known; time-taking (a week is required for extraction and a week – for drying)	±50% of the measured volumes (in case of relatively small samples or samples with high gas saturation with residual volumes of oil and water)
Magnetic resonance saturation scan	The only method to determine saturation on a core plug that does not destroy the sample	Indirect method; difficulties with fluid response separation	Depends on differences in the NMR properties of measured fluids

3.4. Permeability determination

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

There are two widely used unsteady state laboratory methods for permeability measurements in tight rocks [13] (table 6):
1) pressure pulse on a core plug;
2) pressure decay on crushed rock.

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

Table 6. Unsteady state methods for permeability measurements of tight rocks.

Method	Permeability range, mD	Major benefits	Major drawbacks	Accuracy
Axial flow, pulse-decay in core plugs	0.00001 – 0.1	Porosity can be determined with the same apparatus; 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 can be affected by micro-cracks.	3% (low/no leaks, adsorption is accounted, low/no temperature fluctuations present)
Pressure-decay on crushed samples	Gas: 0.00001 – 0.01 Liquid: 0.1 – 2000	Porosity can be determined with the same apparatus; 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)

4. Determination of permeability and matrix volume of the Bazhenov formation sample

The applicability of the pressure decay test on a crushed sample for Bazhenov formation evaluation is checked practically with the SMP-200 commercial permeameter.

4.1. Sample preparation

The core sample was not by any means cleaned, but was dried in a vacuum oven at the temperature of 60°C. Then it was cut and crushed using a geologic hammer and mortar.

4.2. Running of the experiment

The experiment was run using the SMP-200 permeameter. Firstly, reference cell calibration and dead volume calibration were made. After that, a leak off test was conducted. Next, one of the disks (the disk that was almost equal to the volume of the crushed sample) was removed from the sample chamber and the sample was placed into the sample chamber. Then, helium at the pressure of about 200 psi (13.61 atm) was expanded into the sample chamber. A pressure decay curve was recorded using a pressure gauge (the accuracy being 0.001 psi or $6.8 \cdot 10^{-5}$ atm) within 2,000 seconds. Permeability was determined by matching simulated and measured curves.

4.3. Results

The results of permeability measurements are presented in (table 7). It can be noticed that permeability for sample 1 and sample 2 differs within 10.7%.

Table 7. Results of the experiment.

Sample	Particle size, mm	Sample weight, g	Grain volume, cm³	Grain density, g/cm³	Permeability, mD
Sample 1	2-5	44.8193	17.772	2.522	$1.1662 \cdot 10^{-7}$
Sample 2	1-2	30.0360	11.813	2.543	$1.0410 \cdot 10^{-7}$
Difference, %	160	33	33.5	0.8	10.7

The experiment has revealed the following drawbacks of SMP-200:
1) The modeled curve has a low correlation with the measured data. This can be explained by the poor quality of the theoretical model. It should account for the deviation from the Darcy's flow due to the comparable sizes of pores and helium molecules and also it should account for temperature fluctuations.
2) The SMP-200 permeameter has insufficient thermal protection. The temperature influence on pressure measurements was determined during the leak off test. The negative values of the leak off were determined. It can be explained by a temperature growth that causes a pressure increase

in the sample chamber.
3) The possibility to choose the part of the pressure curve that is then correlated with simulated pressure decay reduces the repeatability of the results. Because of the low correlation between the theoretical and measured pressure decays, the choice of different parts of the pressure curve leads to different permeability values that can differ within 20%.

5. Protocols of tight rock analysis

5.1. Protocol description

All the above -mentioned methods can be combined into five protocols of porosity, saturation, and permeability determination in tight rocks. *The first protocol is GRI* used for the crushed rock analysis (figure 2). In this protocol, porosity is determined through bulk and grain volumes obtained by the mercury immersion method and the helium pycnometry method, respectively. Saturation is established using modified Dean-Stark extraction. Permeability is determined with pressure decay on a crushed sample.
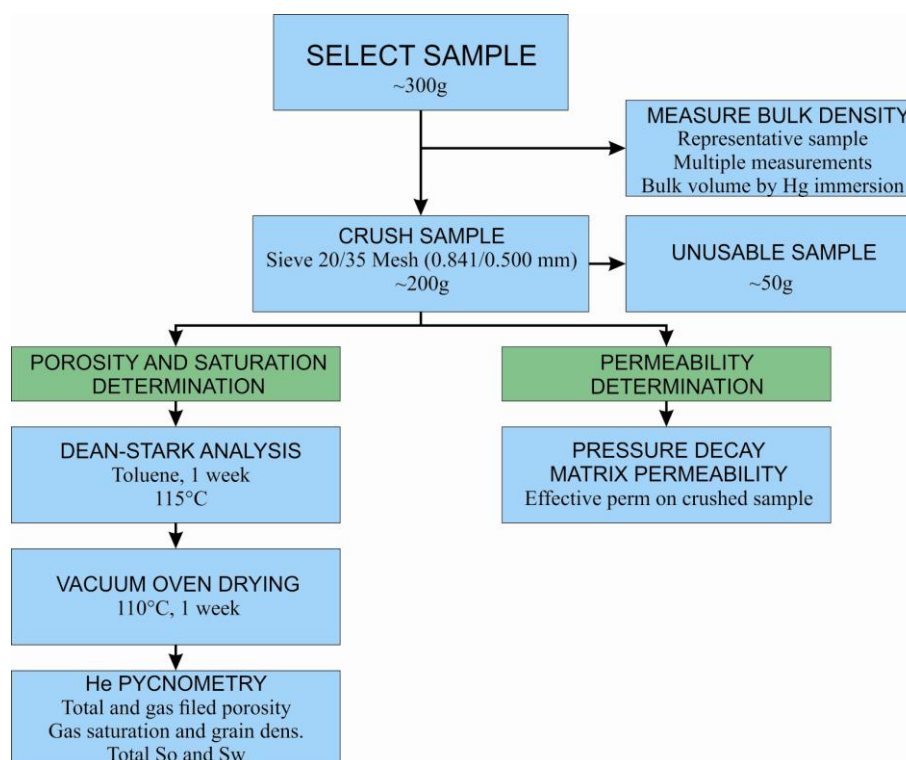

Figure 2. First protocol of porosity, saturation, and permeability determination of tight rocks (GRI).

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

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

The fifth protocol involves nuclear magnetic resonance method for saturation and porosity determination and a pulse test for permeability determination (figure 4). This protocol allows performing measurements without core crushing or destroying.
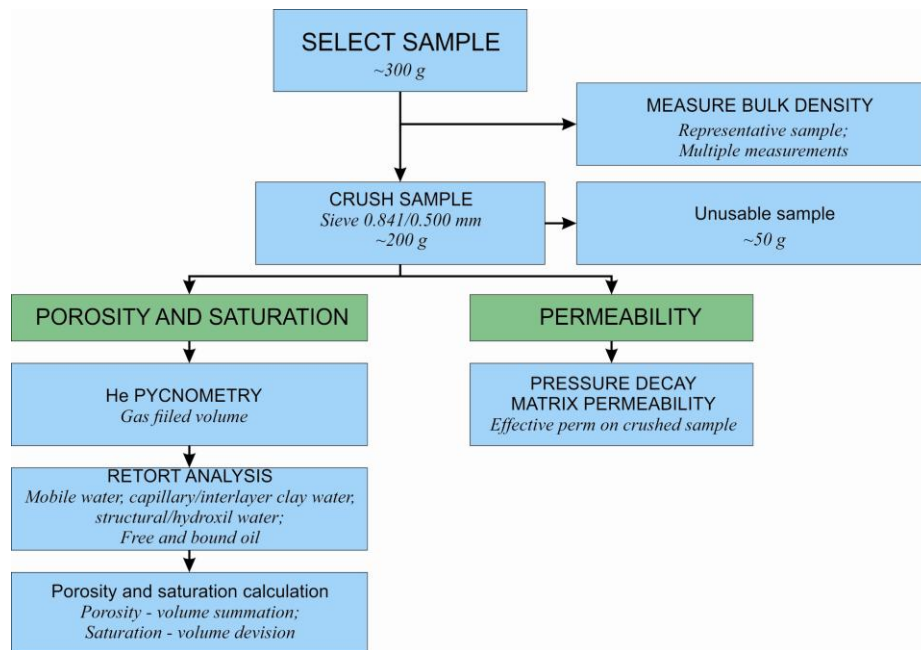

Figure 3. Second protocol of porosity, saturation, and permeability determination of tight rocks (TRA).
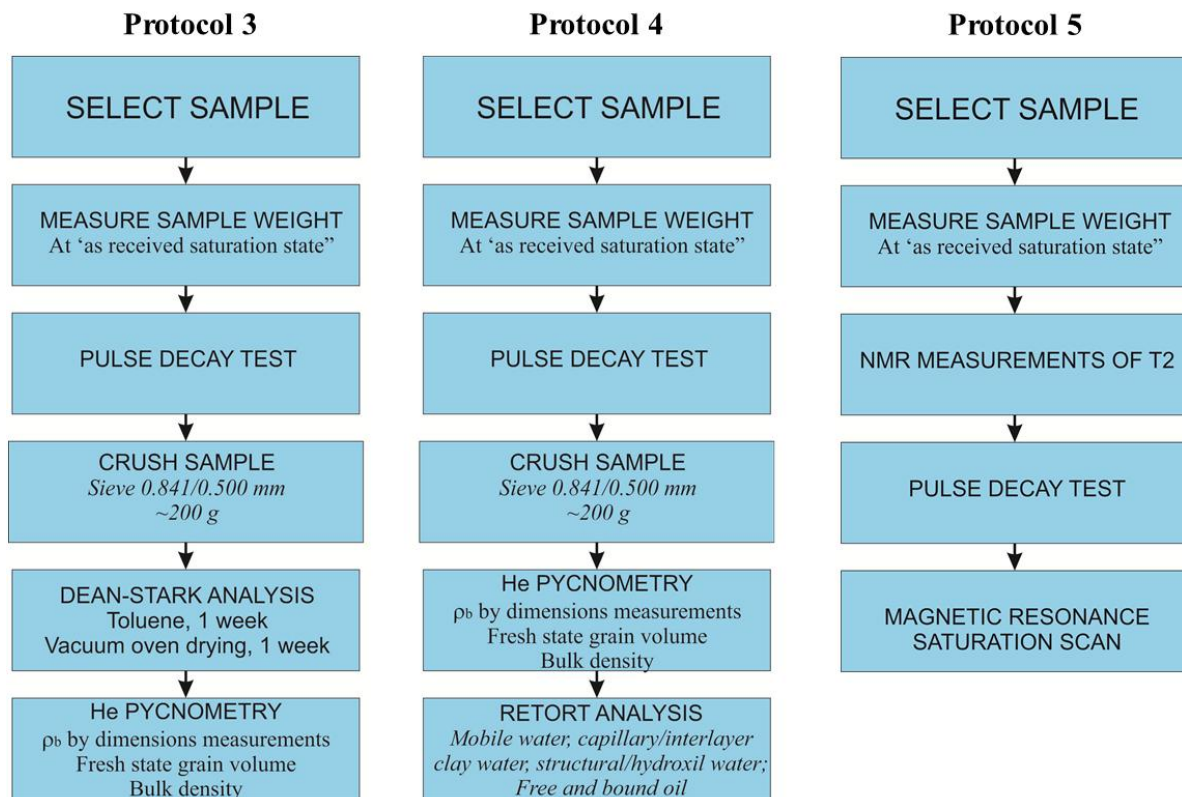

Figure 4. The third protocol (left), the fourth protocol (middle) and the fifth protocol (right) of porosity, saturation, and permeability determination of tight rocks.

5.2. Protocol selection

In order to choose the most effective protocol for tight rock analysis (and thus for the Bazhenov formation), the marketability analysis of all the five protocols was made. The main result of the analysis performed is a marketability evaluation map (table 8).

Table 8. Evaluation map for five protocols of tight rock analysis.

Criteria	Weight of criterion	Marketability				
		Protocol 1	Protocol 2	Protocol 3	**Protocol 4**	Protocol 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 of 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

The marketability of every protocol for every criterion was defined according to the expert judgment and using a five-point grading scheme: 1 – the weakest position, 5 – the strongest position. The matrixes of quantitative relations were made in order to increase grading objectiveness (tables 9, 10 and 11).

Table 9. Matrix of quantitative relations for the "usage experience" criterion for five protocols in case of porosity and saturation determination.

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

Table 10. Matrix of quantitative relations for the "usage experience" criterion for five protocols in case of permeability determination.

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

Table 11. Sum of contributions and grading of the protocols to the "usage experience" criterion.

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

In the matrixes of quantitative relations:
1) "0.5" means that the protocol in the column is inferior to the protocol in the raw;
2) "1" means that the protocol in the column is equal to the protocol in the raw;

3) "1.5" means that the protocol in the column is more effective than the protocol in the raw.

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

The contribution was calculated with the following equation (tables 9, 10 and 11):

$$\overline{} \tag{2}$$

Contributions to porosity, saturation and permeability were summed up and every protocol was graded according to the total contribution value. The marketability of the protocols was determined using the following equation:

$$, \tag{3}$$

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

The marketability results presented in table 8 have revealed that **the most effective protocol is protocol 4** (modified retort analysis and helium pycnometry for porosity and saturation determination and pulse decay on a core plug for permeability determination). The high efficiency of *protocol 4* is reasoned by the high grades of this protocol in the following criteria.

5.2.1. Accuracy of the results
Protocol 4 gives the most accurate results of porosity, saturation and permeability determination. The application of the retort analysis in protocol 4 allows the direct measurements of a fluid volumes saturating core sample that reduces uncertainties caused by calculations. Also, the 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 the water volume obtained [9]. Besides, the fourth protocol excels protocol 5, because NMR used in protocol 5 is not the direct method of porosity and saturation determination and it is prone to uncertainties caused by the longitudinal relaxation time (T_1), transverse relaxation time (T_2), and diffusion coefficient (D) overlapping for different fluids and subsequent uncertainties in saturation determination. In addition, the permeability determined with the pulse decay (protocols 3, 4 and 5) on a core plug is more accurate than the permeability determined on a crushed sample (protocols 1 and 2) in case of confining pressure sufficient for micro fractures closing. Pulse decay is made at reservoir conditions and account for the flow direction, while the pressure decay on a crushed rock is made at ambient conditions.

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

5.2.3. Time of measurements
In the time criterion, protocol 4 is only inferior to protocol 5 in terms of obtaining porosity, saturation and permeability results due to the relatively long time necessary for a modified retort analysis. On the other hand, this drawback of protocol 4 is neutralized by the possibility to perform a modified retort analysis for several samples at a time.

5.2.4. Easiness of interpretation

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

5.2.5. Reliability and safety

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

5.2.6. Price

The low grade of protocol 4 is caused by the higher price for the modified retort apparatus in comparison with the modified Dean-Stark apparatus and the higher price for the pulse decay apparatus in comparison with the pressure decay apparatus.

5.2.7. Scheme for determining 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 (figure 5) is suggested.
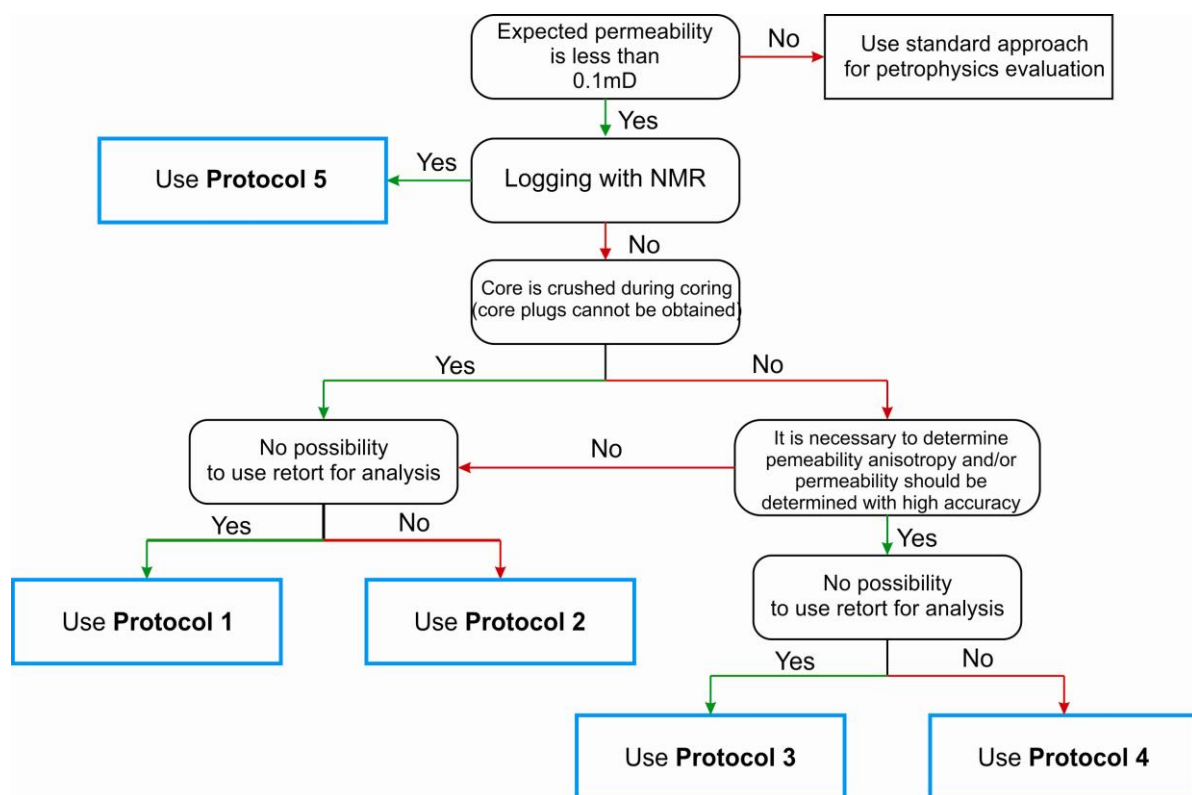

Figure 5. Scheme for determining the most effective protocol for tight rock petrophysical properties evaluation in definite conditions.

6. Conclusions

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

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

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