Comparative Study on Applicability of Permeability Testing Methods in Shale Reservoirs

Yunqi Shen, Jianzheng Su, Qichao Qin, and Hao Chen*

ABSTRACT: Permeability is one of the important parameters for reservoir evaluation, development, and production prediction. Shale oil and gas being an important unconventional energy source, the characteristics of extremely low porosity and permeability bring great challenges to development. Therefore, it is crucial to accurately describe the permeability of shale reservoirs. Based on the steady-state method, pulse decay method, and nuclear magnetic resonance (NMR) method, this paper systematically analyzed the adaptability of various test methods in shale reservoir permeability characterization and optimized the best test method. On this basis, a new high-precision NMR single parameter $T_w$ of shale reservoirs is established, and the compatibility between the model and the experimental results is verified. The results show that the new model with $T_w$ as the main parameter is the most accurate one to calculate the permeability of shale reservoirs in the range of $10^{-3}$–$10^0$ mD. The correlation coefficient between the weighted average relaxation time and pulse decay method is as high as 96.31%, and the relative error between the permeability predicted by the new model and the pulse permeability is 19.43%. These studies have laid a theoretical foundation for the quantitative characterization of fluid seepage capacity in shale reservoirs.

1. INTRODUCTION

Shale oil and gas have become an important source of energy and play an important strategic role. The physical properties of shale reservoirs are poor, the pore throat has reached the nanometer level, the porosity is about 4~8%, and the matrix permeability is in the range of 0.0001~1 mD. Permeability is one of the key parameters necessary for shale gas reservoir evaluation, productivity calculation, and development plan formulation. A reasonable and accurate permeability testing method plays a crucial guiding role in the development plan formulation and economic analysis of shale reservoirs.

The commonly used methods for testing rock permeability in laboratories can be divided into steady-state methods and unsteady-state methods. A steady-state method is a standard method for indoor measurements of core permeability. A steady-state method is a calculation method based on Darcy’s law. The seepage of the test medium in the rock pores needs to reach a stable state, and when the critical Reynolds number $Re < 7$, Darcy’s law can be applied. The rock permeability measured by a conventional steady-state method in the laboratory is closely related to the inject pressure. Gas is usually used as the inject medium. However, gas often has “gas slippage effect” when the shale reservoir has seepage at low pressure. In order to correct the slippage effect, it is necessary to perform multiple measurements under different displacement pressures. Through Klinkenberg equation fitting, the $K_{infty}$ permeability independent of test pressure can be obtained. However, for tight reservoirs such as shale, it takes a long time for the upstream and downstream flow to reach a stable state, which is susceptible to the influence of ambient temperature during a long period of experiment, resulting in core expansion or contraction, leading to the change of rock permeability.

The pulse decay method is different from the steady state method, the pulse decay permeability does not require recording of sample exit velocity and injection differential pressure, through test sample one-dimensional unsteady seepage pore pressure with time attenuation in the process of data and combined with the corresponding mathematical model and test instrument is limited by the initial conditions and boundary conditions, the seepage equation of accurate solution and suitable simplified error control, so as to obtain the permeability of reservoir parameters. Because the pulse decay method is used to monitor the unsteady flow pressure,
evaluate reservoir parameters. In essence, it re
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NMR method can be used to detect rock permeability rapidly
such as shale.\(^{16}\)

pulse decay method has been well applied in tight reservoirs
and the pulse decay method to test shale permeability, the
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Figure 1.

Table 1. Permeability Prediction Model Based on NMR

| model name       | expression                                                                 | model parameters |
|------------------|---------------------------------------------------------------------------|------------------|
| Coates model     | \[ K = \left( \frac{\phi}{10} \right)^4 \left( \frac{F_{FI}}{BVI} \right)^{12} \] | \( \phi \) is the core porosity; \( F_{FI} \) is the bound fluid saturation, \% and \( BVI \) is the movable fluid saturation, % |
| Coates extension | \[ K = \left( \frac{\phi}{10} \right)^{m} \left( \frac{F_{FI}}{BVI} \right)^{n} \] | \( \phi \) is the core porosity; \( C_{m} \), \( m \), and \( n \) are model parameters; \( F_{FI} \) is the bound fluid saturation, % and \( BVI \) is the movable fluid saturation, % |
| SDR model        | \[ K = C_{g} \phi^{n} T_{1g} \] | \( C_{g} \) is the model parameter; \( \phi \) is the core porosity; \( T_{1g} \) is the geometric mean of relaxation time, ms |
| SDR expansion    | \[ K = C_{g} \phi^{m} T_{1g} \] | \( C_{g} \), \( m \), and \( n \) are model parameters; \( \phi \) is the core porosity; and \( T_{1g} \) is the geometric mean of relaxation time, ms |
| single-parameter model\(^{27}\) | \[ K = C_{g} e^{C_{s} \phi} \] | \( C_{g} \) and \( C_{s} \) are the model parameters; \( T_{1g} \) is the geometric mean of relaxation time, ms |

![Figure 1](https://doi.org/10.1021/acsomega.1c03741)

**Figure 1.** Comparison of permeability between the steady-state method and the pulse decay method: (a) comparison of permeabilities as measured by the two methods; (b) relative error of permeability measured by two methods.

compared with the steady-state method, the test time is greatly
shortened and the error influence such as temperature
fluctuation caused by long-term measurements is reduced.\(^{13\text{-}15}\)

Based on the difference between the steady-state method
and the pulse decay method to test shale permeability, the
pulse decay method has been well applied in tight reservoirs
such as shale.\(^{16\text{-}18}\) In order to be able to better evaluate the
permeability of low-permeability reservoirs, reduce and avoid
conventional penetration experiment error, at the same
time reduce the complexity of conventional permeability
experiments and optimization of experiment operation, and improve
the measurement precision, Seegers\ et \ al., for the
first time, proposed a method for estimating permeability by nuclear
magnetic resonance (NMR) methods.\(^{19\text{-}21}\) Compared with
the steady-state method and the unsteady-state method,
the NMR method can be used to detect rock permeability rapidly
and nondestructively. At the same time, it is one of the
important means to effectively recognize reservoirs and
evaluate reservoir parameters. In essence, it reflects the pore
structure of reservoirs by measuring hydrogen signals in
fluid.\(^{22\text{-}24}\) Scholars have proposed a variety of rock NMR
permeability calculation models; currently, the main models
used are the Coates model, SDR model, the corresponding
extended model, and the single-parameter model. This is
shown in Table 1.\(^{25\text{-}27}\) However, these models are all based on
the analysis and statistics of conventional reservoirs and tight
sandstone reservoirs. There are few studies on the applicability
and prediction effect of shale reservoirs; there is no unified
conclusion on whether the prediction parameters in the
current model are applicable to the calculation of shale
permeability.

Therefore, in this paper, in order to give the recommenda-
tion value for the relatively accurate measurement range,
the steady-state method and the pulse decay method are used to
test the shale core respectively to study the applicability of the
two test methods in the shale permeability test systematically;
based on the pulse decay method and considering the
contribution of different pore structures to permeability, a
new NMR permeability testing model for shale is proposed,
which provides a reference for indoor permeability testing.

## 2. RESULTS AND DISCUSSION

### 2.1. Analysis of Experimental Results

Figure 1 shows the comparison results of the permeability between the steady-
state method and the pulse decay methods in 16 shale samples. In
Figure 1a, the test results of the steady-state method and
pulse attenuation method are set as a limit of \( 1 \times 10^{-1} \) mD.
When the permeability is greater than \( 1 \times 10^{-1} \) mD, the
permeability test results are basically the same. As shown in the
log–log coordinate diagram in Figure 1b, the lower the
permeability of shale, the larger the error between the two
kinds of permeability. For example, the corresponding pulse
permeability of rock samples with the Klinkenberg perme-
abilities of 0.0146 and 0.3587 mD is 0.0005 and 0.3946 mD,
respectively, and the mutual error calculated based on the
Klinkenberg permeability is 96.84 and 10.02%, respectively.

Combined with Figures 1 and 2, it can be seen that the
relative errors of the two kinds of permeability increase with
the decrease of the steady-state permeability. Statistics show
that the relative errors of steady-state permeability and pulse
permeability of 17 shale cores range from 1.84 to 96.84%, with
an average value of 45.83%. When steady-state permeability is
greater than \( 1 \times 10^{-1} \) mD, the relative error of the two
permeability is greater than 63.21%. When the permeability is
less than \( 1 \times 10^{-1} \) mD, the relative error of the two kinds of
permeability is less than 28.40%.

The experimental results show that the steady-state
permeability and pulse attenuation permeability have obvious
differences in different permeability ranges. In order to further
analyze the reasons for the obvious differences between the
two kinds of permeability, the experimental principles are compared and analyzed respectively.

As shown in Figure 3, during the steady-state method experiment, a certain continuous pressure is applied at one end of the core to make nitrogen form a stable one-dimensional seepage flow in the core, and the permeability of the rock sample is calculated by using Darcy’s formula. As shown in Figure 4, the pulse attenuation rule is that one-dimensional seepage flow is formed in the pore of the core by applying instantaneous pressure pulse in the upstream of the core, and permeability is calculated according to the attenuation curve of the upper and downstream pressure difference over time. The pressure difference through the sample in the steady-state method experiment is usually much higher than that in the pulse attenuation experiment. A large pressure difference will lead to an uneven distribution of effective stress on the sample. For pulse attenuation experiments, data interpretation usually occurs when the upstream and downstream pressures are nearly equal as they approach the equilibrium pressure. As a result, the distribution of pressure and stress across the sample is more uniform.

The precondition of permeability measurement by steady-state method is that the seepage in the pore needs to reach a stable state, for shale rock, with very low permeability, smaller differential pressure to reach a steady state to take a long time, achieve steady state, through greater pressure to speed up and because of the high-pressure difference to produce non-Darcy flow, at this time, Darcy’s law has not been established. Therefore, the steady-state method takes a long time, and the long-term measurement will lead to temperature fluctuations, device leakage, and other problems, which will make the core compress or expand, leading to inaccurate experimental results. The pulse decay method monitors the pressure of unsteady flow, thus shortening the measurement time to a large extent and reducing the error influence caused by too long time. For high permeability cores, the initial instantaneous pressure pulse reaches equilibrium very quickly, which makes the measurement time too short, and the measurement results are greatly affected by the error of the device operation.

For shale samples with permeability greater than 0.1 mD, compared with the pulse decay method, the steady-state method and pulse decay method test results are in the same order of magnitude, and the relative error is small. In addition, the steady-state method has a relatively simple operation method and data processing analysis. Therefore, when the permeability is greater than 0.1 mD, the steady-state method is recommended. When the permeability is less than 0.1 mD, the measurement results of the pulse decay method are more reliable due to the difference of experimental equipment and experimental principles. However, the pulse decay method requires a long period of system equilibrium time before the start of the experiment, and it needs to derive various analytical solutions of permeability from experimental data, which makes the comparison of calculation results more complicated. According to Figure 1a, in the range of 0.1–1 mD, the permeabilities measured by the pulse decay method and steady-state method are in the same order of magnitude, and the results are basically the same. Therefore, cores with a permeability less than 1 mD were selected from existing samples in this paper. Based on the current NMR permeability prediction model, a fast, high precision, and suitable NMR permeability prediction model is established based on the test results of the pulse decay method through NMR experiments.

2.2. Permeability Prediction Based on NMR.

2.2.1. Comparison of Common Models. This is shown in Table 1, for sandstone, carbonate, and other reservoirs, NMR models for predicting rock permeability mainly include the Coates model, SDR model, and extended model. For shale reservoirs, there is a single-parameter model based on the NMR prediction model.

Based on the Coates model, SDR model, extended model, single-parameter model, and pulse permeability, the prediction results of different models are quantitatively evaluated. Comparison between the predicted permeability of each model and the pulse permeability results is shown in Figure 5. Models 1 and 2 predicted permeability according to NMR porosity, movable fluid saturation, and bound fluid saturation parameters. Model 2 changed from a constant property to variable constant on the basis of Model 1. Figure 5a shows the results of comparison between the Coates permeability prediction model and pulse decay method. The correlation between the two is only 32.27%, and the relative error is 208.84%; the NMR permeability of some samples is not in the same order of magnitude as that of the pulse attenuation method. Figure 5b shows the result of comparison between the Coates extended model and pulse attenuation method. Linear regression analysis was conducted based on pulse permeability, and the fitting result was as follows: $C_1 = 0.176$, $m_1 = 0.473$, and $n_1 = 1.015$. The permeability calculated according to

Figure 2. Relationship between relative errors and steady-state permeability.

![Figure 2](https://doi.org/10.1021/acsomega.1c03741)

Figure 3. Schematic diagram of the steady-state method.
Model 2 has a good correlation with the pulse decay method, and the correlation coefficient can reach 91.3%, but the relative error is still large.

The SDR model and the SDR extension model take the geometric mean $T_2$ and porosity as parameters, which are not affected by the movable fluid saturation and irreducible water saturation, which makes the model simpler. Fitting analysis was carried out according to the above method to get $C_2 = 1.806$. In Figure 9c, the correlation between the two is 35.10%, and the relative error is large. Most of the predicted permeabilities and pulse permeabilities are not in the same order of magnitude. For Model 4, linear regression analysis was carried out to obtain $C_3 = 0.143$, $m_2 = 0.987$, and $n_2 = 11.899$. As shown in Figure 5d, the permeability predicted by the SDR extended model has a good correlation with the pulse permeability, with a correlation coefficient of 89.75% and a relative error of 94.64%. Compared with the previous three models, it has a good correlation but a relatively large error.

Model 5 is a single-parameter model, which only takes the geometric mean $T_2$ as a parameter to be considered, and the pulse permeability is used for fitting analysis, $C_4 = 3.078$ and $C_5 = 13.348$. In Figure 5e, the correlation between the predicted
permeability result of Model 5 and the pulse permeability is 90.44%, with a relative error of 64.30%. In summary, Model 5, which only considers the geometric mean value of a single parameter, has a good correlation with the pulse permeability results and can be used to predict permeability more accurately, but the relative error is still large.

According to the comparative analysis of the permeability prediction model based on NMR and the permeability measured by the pulse decay method, the experimental results show that the relative error between the Coates model and pulse decay method is the largest. The Coates model mainly considers the core porosity, movable fluid saturation, and bound fluid saturation parameters, and the permeability of some samples predicted by the Coates model is not in the same order of magnitude as that of the pulse decay method. The extended model also shows the same results. The difference between the SDR model and Coates model is that the saturation of movable fluid is not considered, but the core porosity and geometric mean \( T_2 \) are taken as parameters, and the extended model of the SDR model has a lower relative error than the Coates model. Compared with the Coates and SDR models, Model 5 only considers the geometric mean \( T_2 \) parameter, which is simpler, but the permeability predicted by this model has a better correlation with the pulse attenuation permeability, and the relative error is the smallest.

2.2.2. Correlation Analysis of Main NMR Parameters and Permeability. In order to further analyze the reasons for the error between the predicted permeability results of each model and the pulse permeability, parameters such as NMR porosity, the ratio of movable fluid saturation to irradiated water saturation, and the geometric mean value of \( T_2 \) relaxation time were selected in this paper to analyze the errors respectively with the pulse attenuation method permeability.

A large number of experiments show that porosity and permeability are not correlated to a certain extent. The pores in shale are mainly formed by hydrocarbon generation from organic matter, with a complex pore structure and seepage law. As shown in Figure 6a, the correlation between porosity and permeability is only 26.56%, and the core permeability with high porosity is even relatively low. The permeability is related to the geometric shape of the void, the size of particles, the direction of arrangement, the pore throat connectivity, and other factors in the direction of permeability; the porosity has a poor correlation with permeability.

Models 1 and 2 considered the bound fluid saturation/movable fluid saturation (FFI/BVI) parameters for shale reservoirs with poor fluid mobility and usually by centrifugation measure the irreducible water saturation and movable fluid saturation, but as a result of shale samples having a lower permeability, there is very little displacement of water by centrifugual experiments, and the experimental results are influenced by environmental factors and large experimental operation. In Figure 6b, the correlation between FFI/BVI and the pulse decay method is only 27.14%. Therefore, the permeability of the prediction model, which takes permeability and FFI/BVI as parameters, has a large relative error with pulse permeability, and this method is not suitable for the prediction of shale reservoir permeability.

In Figure 6c, there is a good correlation between the geometric mean of relaxation time and pulse permeability, and the correlation coefficient is 72.70%. The geometric mean of relaxation time represents the distribution state of fluid in shale pores. In Model 5, the geometric mean of relaxation time is considered as the only parameter that greatly improves the accuracy of permeability prediction results. Therefore, among the main parameters considered by the above five models, the geometric mean of relaxation time and pulse permeability have a good correlation and can be used as the main parameters of the permeability prediction model.

2.2.3. Establishment and Evaluation of the NMR Permeability Prediction Model. On studying the geometric mean of relaxation time of \( T_{2g} \) in NMR, it is found that the geometric mean of relaxation time is greatly affected by the extreme marker value, and each marker value must be a positive number greater than 0 to determine the geometric mean. However, the porosity component of shale samples in small pores is less than \( 10^{-5} \), and the porosity components of the beginning relaxation time and the end relaxation time are significantly different. Therefore, \( T_{2g} \) as a parameter to predict permeability has a certain error. In order to further reduce the prediction error of the NMR permeability model, on the basis of Model 5, the porosity component is taken as the weight coefficient to calculate the \( T_2 \) weighted average, as shown in eq 1.

\[
T_w = \frac{\sum_{i=1}^{n} (T_{2g} \times \phi_i)}{\sum_{i=1}^{n} \phi_i}
\]

(1)

There is a good correlation between the weighted average of \( T_w \) relaxation time and pulse permeability, with a correlation coefficient of 96.31%, as shown in Figure 7. Using \( T_w \) as a parameter of the permeability prediction model, it can better reflect the diversity of fluid space in shale reservoirs. The new model can be expressed as follows (eq 2):

\[
K = 3 \times 10^{-6} \exp(6.3416 T_w)
\]

(2)

As shown in Figure 8, the relative error of the two kinds of permeability is 19.43% when the permeability is calculated by
models, the new model to predict the permeability of the samples and the pulse attenuation method showed that the permeability value has a smaller relative error; therefore, the new model is more suitable for predicting the permeability of low-permeability shale. However, this new model in the prediction of permeability at present also has certain limitations; \( T_w \) permeability parameters and the permeability of the pulse decay model have good correlation, and the model is based on the permeability of the pulse attenuation method for fitting, and this experiment adopts the pulse attenuation device, due to the upstream and downstream chamber volume and accuracy sensors. Therefore, the current permeability test range of this model is in the order of \( 10^{-3} \) to \( 10^{-4} \) mD. To determine whether this model is suitable for the prediction of samples with permeability lower than the order of \( 10^{-3} \) mD, further research work needs to be carried out.

3. CONCLUSIONS

Shale reservoirs have extremely complex pore connectivity, and different test methods provide test results that have great uncertainty. Based on three different permeability testing methods, this paper conducts permeability testing and prediction respectively. By comparing and analyzing the results of different permeability calculation methods, a more accurate permeability prediction model is proposed. The conclusions are as follows:

(1) The study shows that compared with the steady-state method, the pulse decay method is more suitable for the measurement of shale permeability when the permeability is less than 0.1 mD. The lower the permeability, the greater the error of the experimental results of the two methods, and the maximum value can reach 96.84%. When the permeability is greater than 0.1 mD, the relative error of the two permeability testing methods is less than 28.40%, and when the permeability is less than 0.1 mD, the relative error is more than 63.21%.

(2) Taking the pulse decay method permeability as the standard, the correlation between the parameters of the current NMR permeability prediction model and the pulse permeability is deeply explored. The research shows that the shale core porosity and irreducible water saturation have poor correlation with the permeability and are not suitable as the prediction parameters of shale permeability.

(3) On the basis of considering the correlation between the main parameters of NMR and the permeability of the pulse decay method, the single-parameter "\( T_w \)" model of shale NMR permeability is established; the \( T_w \) parameter has a good correlation with pulse permeability, which can better reflect the diversity of fluid space in shale reservoirs. The relative error of permeability between this model and the pulse decay method is 19.43%, indicating that this model can accurately predict shale permeability.

4. EXPERIMENTAL MATERIALS AND METHODS

4.1. Experimental Materials. The experimental materials were seven ultralow permeability interstitial shale cores in a block of Jianghan Oilfield, China, three ultralow permeability continental shale cores in a block of Shengli Oilfield, China, and six ultralow permeability marine shale cores in a block of Longmaxi Formation, Fuling shale gas field, China.
samples were cut 2.5 cm and a length of about 5 cm. Both ends of the plunger were selected to drill plunger samples with a diameter of about 0.01 mL/min. For low-permeability cores, Malkovsky showed with a measuring range of 0–600 mL/min and an accuracy of 0.01 mL/min. For low-permeability cores, Malkovsky showed through experimental studies that constant pressure displacement was more accurate than constant velocity measurement data.9

Test steps are as follows:
(1) Obtain a core of standard size and weigh it.

shows the conventional physical properties of 16 rock samples. The permeability distribution range of the 16 rock samples covers the reservoirs with low–ultralow permeability and also contains three different lithologies of inter salt shale, marine shale, and continental shale, which are well representative.

In this paper, shale cores with different permeability levels were selected to drill plunger samples with a diameter of about 2.5 cm and a length of about 5 cm. Both ends of the plunger samples were cut flat, and the rock samples with obvious defects on the surface of the plunger were removed. Part of the rock sample is shown in Figure 9.

**Table 3. Petrophysical Properties of Sandstone Samples**

| core lithology  | \( K_\infty \) distribution range/mD | quantity/piece | porosity/% | Klinkenberg permeability/mD |
|----------------|-------------------------------------|----------------|------------|-----------------------------|
| intersalt shale| >1.0                                 | 3              | 5.804–6.836| 1.651–7.6104 |
|                | 0.1–1.0                              | 1              | 6.225      | 0.105                      |
|                | 0.01–0.1                             | 3              | 4.514–8.995| 0.033–0.068               |
| continental shale | 0.1–1.0                              | 3              | 6.245–7.724| 0.154–0.369               |
|                | 0.01–0.1                             | 3              | 5.323–7.485| 0.015–0.056               |
| marine shale   | >1.0                                 | 1              | 3.533      | 1.016                      |
|                | 0.1–1.0                              | 1              | 3.951      | 0.359                      |
|                | <0.01                                | 1              | 3.381      | 0.004                      |

(2) Add the foaming liquid in the soap film flowmeter to an appropriate liquid level, and connect the soap film flowmeter with the outlet end of the core gripper.

(3) After the rock sample is put into the holder, the confining pressure is increased to 2 MPa. Gas (nitrogen) of different pressures is applied respectively to the upstream of the rock sample, and the flow rate is measured by the downstream soap film flowmeter. The outlet end of the flowmeter is connected with the atmosphere.

(4) After the gas flow is stable, the gas flow rate is tested to calculate the apparent permeability, and the core’s Klinkenberg permeability is finally obtained by fitting according to the Klinkenberg equation (eq 3).

\[
K_g = K_\infty \left(1 + \frac{b}{\bar{p}} \right)
\]

In the formula, \( K_g \) and \( K_\infty \) are, respectively, the gas permeability and Klinkenberg permeability of rock samples, mD; \( b \) is the slippage factor, representing the strength of slippage effect in porous media, MPa; \( \bar{p} \) is the average gas pressure, MPa.

**4.2. Experimental Method.**

**4.2.1. Steady-State Method.**

Figure 10 shows the experimental flow chart of the steady-state method, which is mainly divided into three parts. The upstream is connected with a nitrogen bottle and constant pressure valve, the middle is a core holder, and the downstream is connected with a high-precision flowmeter with a measuring range of 0–600 mL/min and an accuracy of 0.01 mL/min. For low-permeability cores, Malkovsky showed through experimental studies that constant pressure displacement was more accurate than constant velocity measurement data.9

Test steps are as follows:
(1) Obtain a core of standard size and weigh it.

Figure 9. Part of experimental rock samples.

Figure 10. Flow chart of the steady-state permeability measurement experiment.

**4.2.2. Pulse Decay Method.**

In this paper, the main shale pore size distribution used for the pulse decay method is in the range of 2–50 nm, and the average pore size is 35 nm, which belongs to the mesopore core. In the process of shale permeability testing and analysis, because the pores in shale are complex and diverse and most of them are nanosized, the gas flow process is controlled by slippage effect and solid surface force. The pulse decay method is not designed to fully capture the apparent diffusion of adsorbed gas, and it has some limitations in measuring shale permeability. The current experimental study shows that the slippage effect can be ignored when the pore size is greater than 32 nm and the gas pressure is greater than 7 MPa when the pulse attenuation method is used to measure the permeability of shale. Meanwhile, due to the weak adsorption of nitrogen, nitrogen was used as the high-pressure gas source in the test, and the change of upstream and downstream pressure was controlled within 5% of the initial pore pressure, so that the influence of gas desorption on permeability calculation could be ignored.28

Figure 11 shows the experimental flow chart of the pulse decay method. The core permeability can be obtained through the upstream and downstream pressure attenuation curves with the theory of gas unsteady flow.

The specific experimental steps are as follows:
(1) Open all valves and inject gas at constant pressure. After the pressure is balanced for a few minutes, close the...
charging valve to ensure that the precision pressure gauge counts zero.

(2) Close valve 2. When valve 7 is closed, open valve 6 to empty the pressure downstream of rooms 2 and 3, so that the pressure gauge can reach the maximum range.

(3) Close valve 6, and the confining pressure and displacement pressure gradually increase to the target pressure with step length $\Delta P = 0.5$ MPa (confining pressure, 9 MPa; inject pressure, 7 MPa).

(4) After the pore pressure of upper and lower chambers and the rock sample reaches equilibrium, the pressure of the downstream chamber is manually reduced by about 70 kPa to form an initial attenuation pressure pulse to monitor $\Delta P$ and pressure of chamber 2, with the calculated average pore pressure $P_m$. Close valves 3 and 4 for a constant value.

The mathematical model for calculating the pulse permeability of rock samples based on the experimental data is shown in eq 4.14

$$K = \frac{c\mu p L^2}{f(a, b)}$$

In the formula, $k$ is the permeability of the pulse decay method; $c$ is the compressibility of pore fluid; $\varphi$ is the core porosity; $\mu$ is the fluid viscosity; $L$ is the length of the rock sample; and $S$ is the slope of pressure difference $p$ and time $t$ of the upper and downstream chambers of the pulse permeameter tested in the monologarithmic coordinate graph; $a$ and $b$ are respectively the ratio of pore volume of the rock sample to upper and downstream chamber volume of the pulse permeameter. When $a = b = 1$, $f(a, b) = 1.71$.

4.2.3. NMR Method. The research instrument used in this paper is the RecCore 2500 low-field nuclear magnetic resonance instrument from the Institute of Percolation and Fluid Mechanics, Chinese Academy of Sciences, Langfang, China. Figure 12 shows the flow chart of the NMR test experiment; the main experimental steps are as follows:

(1) First, drill one or two representative standard cores with a diameter of 2.5 cm from the full-diameter core, wash the oil-bearing core, and dry it.

(2) Then, the core is vacuumed at 80 °C for 12 h.

(3) The core is pressurized with saturated kerosene for 24 h.

(4) The saturated core was detected by the NMR $T_2$ attenuation spectrum, and the data were processed.

(5) A high-speed centrifugal test was carried out with 3 MPa centrifugal force, and the BVI was calculated by weighing and the NMR test again.

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**Figure 11.** Flow chart of the permeability measurement experiment by the pulse decay method.

**Figure 12.** Flow chart of the NMR test experiment.
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Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.1c03741

Notes
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