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MONITORING MULTISTAGE FRACTURING FLUID FLOWBACK USING TRACERS

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ABSTRACT
Multistage hydraulic fracturing is an important way to stimulate the tight oil and gas reservoir. Due to the number of stages and anisotropy of the long horizontal section, the flowback of each stage is important for the evaluation of the fracturing effect and the next well location deployment. At present, microseismic monitoring technology is a popular evaluation method, it can display the morphology and growth of fractures, but some other technologies are needed to analyze the flowback and production conditions of each stage. In this paper, the tracer monitoring technology is introduced by pumping a variety of tracers with the fracturing fluid into the reservoir, continuous sampling to detect the concentration of tracer concentration, then by accumulating the amount of tracer and the flowback volume to determine the flowback condition, and finally analyzing the fracturing effect of each layer. This method can achieve the qualitative and quantitative analysis of flowback situation, the flowback situation of each layer is also an important information for the future production, it can determine which stage is the main production layer

KEYWORDS: multistage fracturing, production profile, monitoring Tracer, quantitative analysis

I. INTRODUCTION
This paper introduce the isotopic tracer. The tracer has good compatibility with the fracturing fluid, small adsorption to the rock , good stability , high analysis precision and no interference with each other , it is suitable for fracturing monitoring. 12 tracers were screened by a number of experiments. This can monitor the flowback condition of each fracture stage and then help recognizing the fracturing effect of each stage. Through study of the analytical method, a suitable analytical method is found, the detection precision is improved, the labor intensity is reduced, the sample detection work can be done accurately and efficiently, and the continuous monitoring of the fracturing effect can be realized.

II. TRACER SELECTION AND INDOOR EVALUATION
At present, the inorganic salt tracer is hardly used in the field because of its less types, large amount, high cost, low test precision . Fluorobenzoic acid tracer is not used because it is expensive and the process is complicated. So they can not meet the needs of monitoring fracturing fluid's flowback. Therefore, it is necessary to carry out the screening of isotopic tracer and the corresponding analysis method to meet the requirements of real-field monitoring.

1. isotopic tracer ——thermal stability experiment
This test is used to evaluate the thermal stability of the tracer. The 12 tracer elements and the chelating agent are prepared in a molar ratio of 1: 1 to 1: 9, and the concentration of the 12 types of solutions are controlled in 1%. The solutions are heated to 300℃ and then stand for 6 months. The results show that they keep thermally stable when the molar ratio is less than 1:8, but the increase of chelaing agent will lead to the larger amount of the tracer used, so the corresponding costs are higher, so the ratio of elements and the chelating agent 1: 7 is finally decided .

2. Isotopic tracer —PH stability experiment
This test is used to evaluate the stability of the tracer in different pH solutions. 12 kinds of tracer are mixed in water solution with the concentration of 1% , adjusting the solution PH value : 1, 2, 3, 6, 9, 11, 14, after standing

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for 6 months, the experimental results show that the 12 tracers can be stable in the range of pH 3 to 14, this can meet the requirements of the real-field hydraulic fracturing .

3. Isotopic tracer —solubility test

This test is used to evaluate the dissolution characteristics of the tracer, including the solubility and the dissolution time at different temperatures. As the 12 tracers are homologous, the physical and chemical properties are close, so the representative SZJ-1 and SZJ-5 were selected for evaluation experiments.

The solubility of SZJ-1 is shown in Fig. 1, and the solubility of SZJ-5 is shown in Fig. 2. The dissolution time of SZJ-1 is shown in Fig. 3, and the dissolution time of SZJ-5 is shown in Figure 4.

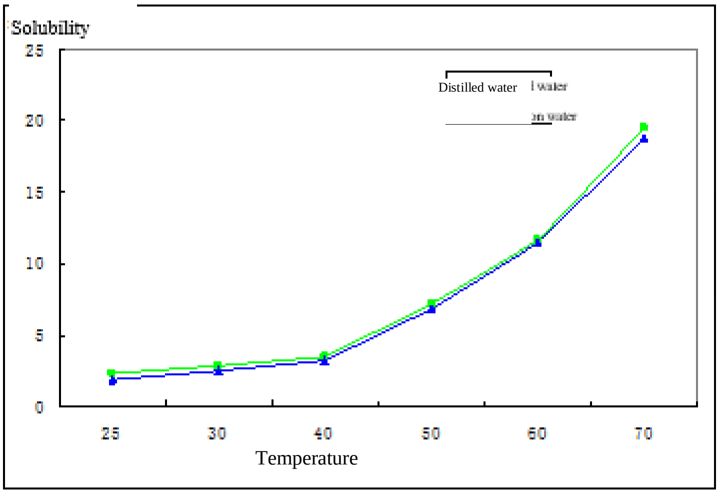

Figure 1 SZJ-1 Solubility vs. Temperature

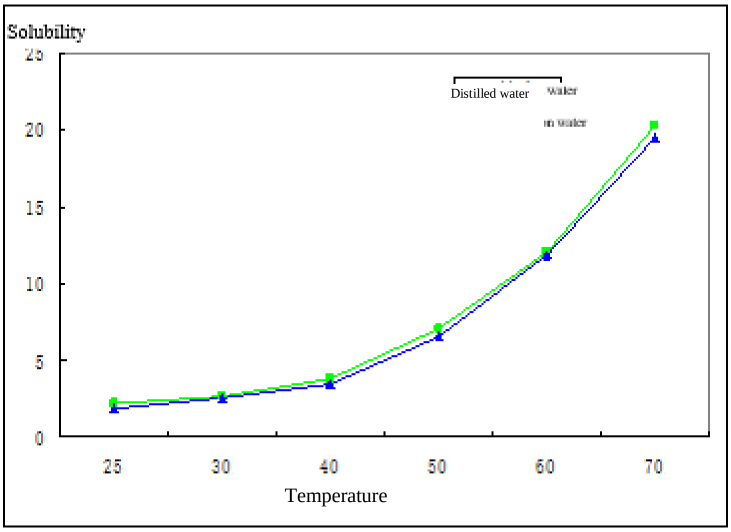

Figure 2 SZJ-5 Solubility vs. Temperature

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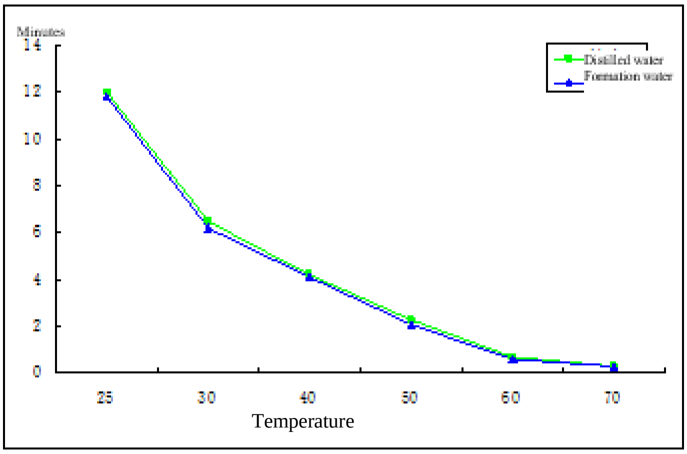

Figure 3 SZJ-1 Dissolution Time vs. Temperature

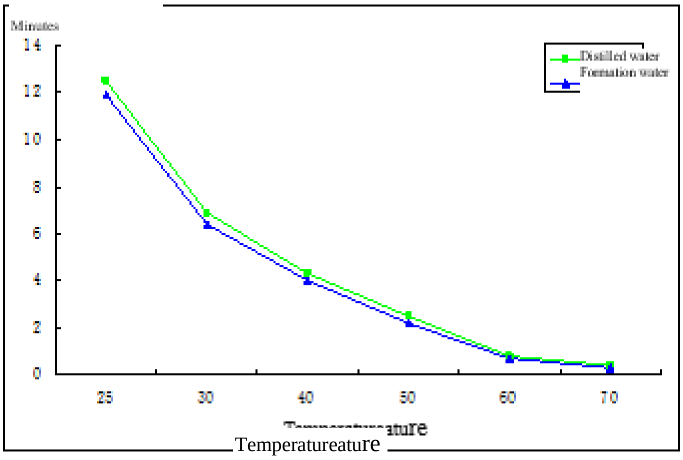

Figure 4 SZJ-5 Dissolution Time vs. Temperature

Conclusion from the experimental data :

1) The solubility and dissolution rate of the tracer accelerate with the increase of temperature.
2) Tracer will be endothermic in the dissolution process, decreasing the water temperature, thereby reducing the solubility of tracer, this problem should be payed attention in winter oilfields;
3) The concentration of the tracer solution should be kept below 1% at the normal temperature (25 ℃) and the dissolution time should be kept above 20 minutes.
4) When the temperature reaches 40-50 ℃, the concentration of the tracer solution should be kept below 3% and the dissolution time should be kept above 10 minutes.

In general, the concentration of the tracer solution and the dissolution time need to be adjusted according to the real-field situation, the tracer needs to be totally dissolved before they are pumped into the reservoir.

4. Isotopic tracer— adsorption test

The purpose of this experiment is to determine the degree of tracer adsorption to formation, as follows:

1) 12 kinds of tracer solutions are prepared with the same concentration by using oilfield formation water and the initial concentration C0 need to be tested.
2) Shredded rock samples are placed in specific containers separately, each rock sample weighting 30g. 12 kinds of tracer solutions are added to 12 containers separately according to 1: 3 (mass), the containers should be stirred evenly, sealed tightly, shocked at room temperature for 5 hours, then standing for 6 months;
3) Remove the 12 tracer solutions from the container, and determine the concentration C of each tracer after pretreatment;

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4) Determine the concentration retention rate C / C0, when the C / C0 is greater than 80%, the tracer meets the experimental requirements.

The experimental results are shown in Table 1.

Table 1 Adsorption Test Results

Tracer	Initial centrationC_0(ug/L)	After adding core	6 months C	Concentration ntion(%)
SZJ-1	100	98.76	98.15	＞97
SZJ-2	100	98.12	97.65	＞97
SZJ-3	100	97.78	97.21	＞97
SZJ-4	100	98.24	97.66	＞97
SZJ-5	100	98.37	97.57	＞97
SZJ-6	100	98.09	97.41	＞97
SZJ-7	100	98.11	97.48	＞97
SZJ-8	100	98.37	98.11	＞97
SZJ-9	100	98.73	98.15	＞97
SZJ-10	100	97.28	97.03	＞97
SZJ-11	100	98.55	98.02	＞97
SZJ-12	100	98.85	98.14	＞97

Experiments show that the 12 kinds of tracer adsorption capacity are less than 3%, meet the requirements of relevant standards, the standards needs the adsorption less than 20%

5. Isotopic tracer— stability test

Multi-stage fracturing usually pumps large amount of fracturing fluid, so it needs longer flowback time. In order to ensure the accuracy and continuity of the monitoring results, the tracer is required to maintain stable for a long time in the reservoir temperature and fracturing-fluid environment. At the same time, for the multistage fracuturing, fracturing fluid are pumped into each stage separately but flowed back at the same time, so different kinds of tracers are detected at the same time , requiring the interference between different tracers should be small. The purpose of this experiment is to determine the stability of the tracer in the simulated real-oilfield environment and the interference between each tracer, the experimental method is as follows: 12 tracers' solutions with the same concentration are mixed together, then stirred with conventional water-based fracturing fluid at a volume ratio of 1: 1000, standing for 6 months in water baths with different temperatures. The concentrations of various tracers are finally measured. Tracer concentration retention rate should be greater than 90% to be qualified.

Table 2 Compatibility Test Results

Tracer	Initial concentration ug/L	Tested concentration ug/L	Concentration Retention rate(%)
SZJ-1	10.0	9.95	＞97
SZJ-2	10.0	9.86	＞97
SZJ-3	10.0	9.92	＞97
SZJ-4	10.0	9.90	＞97
SZJ-5	10.0	9.84	＞97
SZJ-6	10.0	9.87	＞97
SZJ-7	10.0	9.75	＞97
SZJ-8	10.0	9.82	＞97
SZJ-9	10.0	9.79	＞97
SZJ-10	10.0	9.82	＞97
SZJ-11	10.0	9.81	＞97
SZJ-12	10.0	9.84	＞97

As can be seen from Table 2, the concentration retention rate of each tracer is more than 97%, in line with the industry standard, which requires the concentration retention rate greater than 90% , indicating that tracer is compatible with the fracturing fluid , and the interference between tracers is small.

A number of indoor experiments have proved that the screened 12 tracers can meet the technical requirements of real-oilfield hydraulic fracturing; they have advantages of good stability, less adsorption to formation and easy detection.

III. STUDY ON ANALYSIS METHOD OF MULTI - LEVEL FRACTURING TRACER

1. Study on Automatic Separation and Enrichment of Trace Elements

The common methods include precipitating method, solvent extraction method, ion exchange method, chromatography and extraction chromatography medthod. The ion exchange method uses a solid ion exchanger to react with the trace elements in the water, allowing the trace element ions in the solution to selectively enter the solid exchanger while the other elements are separated by not entering the solid ion exchanger. And then use the appropriate reagent to leach the trace elements into the liquid phase, achieving the purpose of enrichment and separation.

By comparison, we believe that this method is more suitable. Because this method can reduce the amount of solvent used compared with other methods, while the stationary phase is not easy to lose, the operation is also easier than the other methods. We use SPE-03 multi-channel automatic solid-phase extraction instrument to achieve the automatic separation and enrichment. The instrument can automatically complete the column activation, balance, pre-leaching, extraction, drying, elution, component collection, and pipe cleaning operations, improving the efficiency and quality of the analysis and also significantly reduce the impact of chemicals on laboratory operators.

A special solid phase extraction column is used as the stationary phase, and 3mol / L HNO3 is used as the eluent. 3mol / L HNO3, distilled water, pH adjuster, water samples to be processed are put into the corresponding reagent bottles, The SPE-03 automatic solid phase extraction instrument is used, cleaning the activated column with 3mol / L HNO3 solution, then cleaning it with water and pH adjusters, then the impurities and other interference elements will be separated from water sample through the column, trace elements would be adsorbed on the column to achieve enrichment, and finally leaching with 3mol / L HNO3, the elements will be washed into the shower Lotion. The whole process is completed by the instrument automatically; this separation and enrichment method is fast, simple and cheap.

Experimental study

Drugs and materials are as follows: HNO3: 3mol / L, pH adjusting solution, distilled water, special solid phase extraction column.

The process is as follows: activating equilibrium column with 3mol / L HNO3, cleaning column with pure water, adjusting pH value with pH adjustment solution, sample column, leaching with 3mol / L HNO3 solution. Then the solution is loaded into solid phase extraction instrument, adsorption rate is 5mL / min, elution rate is 2.5mL / min, after setting the program, the instrument automatically separate and enrich to complete the sample treatment.

Separation and enrichment of the most conditional experiment

The binding capacity of rare-earth to solid phase extraction columns will be different at different concentrations. The enrichment behavior of rare-earth on the column at the concentration of 0.1-5 mol / L is tested. The results are shown in Table 3. When the concentration is 3 mol / L, the rare-earth element has the highest recovery rate, and the elements are in the range of 95% -100%, The recovery rate at other concentrations is relatively low. Therefore, 3 mol / L was chosen as the best column adsorption concentration.

Table 3 Adsorption Concentration Experiment

Element	Recovery Rate，%			
	0.1mol/L	1mol/L	3mol/L	5mol/L
Sm	75.9	79.6	96.8	89.2
Nd	78.2	82.9	99.5	88.4
Pr	80.1	85.5	100.1	81.3
Tm	72.9	87.1	97.4	84.0

The column adsorption rate is also a key issue that affects the accuracy of the experiment.The effect of column adsorption rate on the enrichment effect was tested under the concentration of 3mol / L, the flow rate was 5.0mL / min in the experiment, and the recovery rate of rare earth elements was 95-100%, indicating that the enrichment effect is very good.

In this experiment, the effect of elution rate on the enrichment effect was tested under 3 mol / L concentration. The results showed that the optimum elution rate was 2.5 mL / min, which could elute the element completely and did not cost a large volume of the eluent. After the test, using the best elution rate, only 50mL 3mol / L of HNO3 completely elute the elemets.

Table 4 Recovery Test Experiment

Element	Concentration（ng/L）		Recovery rate%，
	Initial	Tested	
Sm	100	97	97
Nd	100	99	99
Pr	100	98	98
Tm	100	97	97

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Table 5 is the statistical results of the four measurements of the water samples. From the test results, RSD is between 1% -5% , the precision is very good.

Table 5 Tested Results for four water samples

Element	1	2	3	4	RSD，%
Sm	54ppb	52ppb	55ppb	52ppb	1.5
Nd	88ppb	85ppb	91ppb	84ppb	3.2
Pr	16ppb	12ppb	18ppb	22ppb	4.2
Tm	111ppb	108ppb	105ppb	107ppb	2.5

2. Study on Micro - element Automatic Dilution Injection Method

Due to ICP-MS requirements, high concentrations of samples should not be measured directly in actual sample analysis, this usually leads to cone effect, system contamination, reducing detector life and sensitivity accuracy. So we have to dilute the sample. For a large number of samples to analyzed, artificial dilution is obviously time-consuming and low efficiency. We use the AXS-520 auto-sampler with ID-100 automatic dilution machine to automatically dilute the sample. The enriched sample is automatically diluted after putting into the machine. ID-100 is suitable for dilution of unknown samples, it can automatically dilute those samples beyond the dynamic linear range, also can reduce the signal suppression problem in the high matrix sample solution. It can carry out sample pre-scanning, and determine the most suitable dilution program intelligently, and then achieve the instrument requirement— sample salinity of less than 1g / L, so the measurement results are accurate and reliable and also can extend the life of the instrument.

The ICP-MS peristaltic pump suction tube is connected to automatic diluent machine, so the sample can be automatically added. A three-way hose is used to add the sample; the entire system can complete the sample injection and dilution automatically, improving the sample processing and injection efficiency.

IV. CONCLUSION

1. Tracer monitoring technology is pumping the tracer with the fracturing fluid into the reservoir, continuous sampling to detect the concentration of tracer in the flowback fluid, and then confirming the flowback situation through the amount of tracer and flowback volume , and then analyzing the multistage hydraulic fracturing effect . This method can achieve qualitative and quantitative analysis of flowback situation, the flowback situation of each stage is also an important information on the future production, to determine the main production stage.
2. The indoor evaluation experiment shows that the 12 kinds of tracer have the advantages of stability, solubility, adsorption and good compatibility with fracturing fluid.
3. Through the study of trace element separation-enrichment method and dilution -injection method, this tracer technology can apply on multistage fracturing monitoring..

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