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Experimental Methods for Measuring Movable Shale Oil

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Although a large amount of hydrocarbon exists in shale, only a part of hydrocarbon is movable because of the shale oil's flow ability character and the adsorption between hydrocarbon and minerals, kerogen, etc. Nuclear magnetic resonance (NMR) cores analysis was commonly used to measure movable oil in conventional cores (Li Tai-wei, et al, 2012; Li Zhentao, 2011; Xiao Qiusheng, et al, 2009). The process is can be summarized as follow: Firstly, the initial conventional cores was analyzed with NMR to measured total oil content, secondly, the movable oil was separated from this core with centrifuge methods, and then the irreducible oil was measured with NMR, the difference of oil amount of two sample, before and after was the movable oil amount. However, both of porosity and permeability are so low in shale, thus, the movable oil rate was not credible measured by conventional NMR analysis. This paper intends to obtain movable oil rate in shale via simulation experiment associated with NMR cores analysis. The process is as follow:

(1) Fresh shale was crush in to suitable size particles, and then these particles were dip into 20% manganese chloride solution. Owing to paramagnetic manganese ion, the relaxation of water inside rocks has been enhanced, the signal magnitude of water has accelerated, with the relaxation of oil inside rocks keeping constant, through a change of relaxation, single oil signal was obtained ultimately to measure the total oil content.

(2) The shale samples were put into experimental apparatus that designed for movable oil simulation and sealed them up (Fig. 1). The water container has been filled with formation water for experiment. All of experimental devices were connected by line pipe and valves. In the first, the autoclave was vacuumed with vacuum pump and then was filled with formation water by boost pump. Meanwhile, outlet valve was switched off and the sample inside autoclave was exerted a pressure slightly lower than formation pressure. Then, the autoclave was heated to formation temperature, and holds it on. Simultaneously, the pressure inside autoclave was rose to formation pressure. 24hours later, Inlet valve was switched off and outlet valve was switched on. Thus, the fluid was gathered in a collecting container. The sample in autoclave was exert and released pressure repeatedly (7 times, 7 days usually) until no oil flow out of pipe can be detected. All of the oil fluid flows out of autoclave and collected was extracted with dichloromethane, and the oil was weighed after the dichloromethane being volatilized completely.

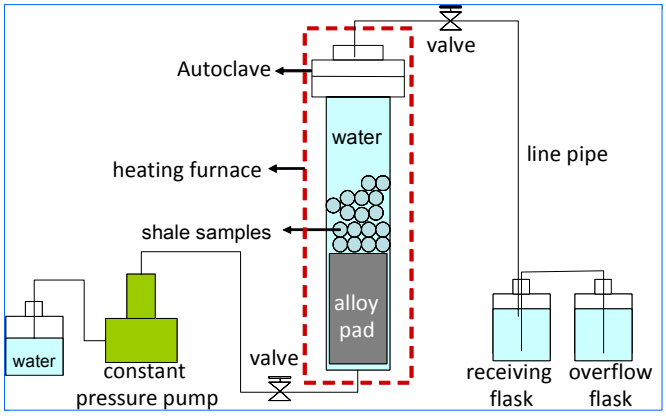

Fig.1 sketch map of apparatus for shale oil's move ability simulation

(3) The shale samples after simulated were get out from experimental apparatus and dip into manganese chloride, which concentration was 20%. the signal of oil were measured by NMR. The oil mass difference of samples before and after simulation is the movable oil mass of this sample.

In the diagram of oil signals vs T2 relations, which include signals of samples before and after simulation, the signal area envelop by curve of oil signal and T2 time axis was quantified to calculate oil content, and the signal area difference of samples before and after simulation represents the movable oil content. The movable oil signal having high T2 relation value than irreducible oil in T2

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distribution diagram shows that most of movable oil either occupies larger pore or was light oil. A series of experiment results show that the proportions of movable oil in shales range from 10% to 30% in Dongying Depression.

The Rock-Eval parameters of S1 and the chloroform bitumen "A" of samples before and after simulation were measured also to calculate movable oil ratio, The proportion of movable oil of shales obtained by three methods (Rock-Eval parameter S1, the chloroform bitumen "A" and NMR) showed similar variation trend with depth variation: the proportion of movable oil increased with depth increasing. By contrast, the regularity of NMR cores analysis are better than the others: for this method, the initial oil content and irreducible oil content can be measured on the same sample without sample's destroying. The difference of movable oil ratio for different shale may be caused by shale's strong heterogeneity.

More over, the cryopreserved shale samples were observed via fluorescence microscope, shale microfracture were mostly filled with light oil with blue-green fluorescence, the existence of movable oil was proved by fluorescence effect.

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