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3,180,142 METHOD FOR TESTING MULTIPLE COMPLETION WELLS

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The present invention relates to methods for the testing of oil wells and more particularly relates to an improved method for determining whether adjacent producing zones are segregated from one another in the vicinity of multiple completion wells. In still greater particularity, the invention relates to a testing method wherein a readily-identifiable organic compound is injected into one zone of a multiple completion well and fluids subsequently produced from a second zone are treated with a solvent to extract the organic compound and permit its identification.

The use of multiple completion wells substantially reduces the cost of developing oil fields overlying vertically-spaced producing zones. By completing the development wells in such a field so that fluids from two or more zones can be separately conducted to the earth's surface through parallel or concentric pipe strings, the several zones can be produced with fewer wells and less equipment than would otherwise be required. Certain practical difficulties are often encountered with such wells, however. Experience has shown that fluid losses from one producing zone to another are common. Faulty cementing of the casing in the wellbore, failure of the production packers, corrosion of the wellbore tubing, or the development of vertical fractures adjacent the wellbore may permit the flow of substantial quantities of oil between zones. When this occurs, the actual production from one zone may be considerably higher than the indicated production; while other zones may be producing considerably less oil than the production figures would indicate. The erroneous production data thus obtained may result in the adoption of recovery programs and practices which are unsuited for the conditions actually existing. Moreover, the escape of fluids from one zone to another may make compliance with conservation regulations difficult. In many areas such regulations restrict the amount of oil which can be produced from each zone of a multiple completion well and impose severe penalties in cases of over-production. The testing of multiple completion wells to determine whether adjacent zones are segregated from one another is required at frequent intervals in order to insure accuracy of the production records.

Multiple completion wells are usually tested to determine whether fluids are escaping from one zone to another in the vicinity of such wells by varying the production rate from one zone and recording the pressure in another. Any variation in pressure in the second zone generally indicates that the two zones are not segregated from one another. This method is satisfactory in some cases but is difficult to use where the pressure differences between the zones are small or where pressure measurements must be made with a down-hole recorder at the bottom of the wellbore. The pumps and crossovers must be removed from some wells before such a recorder can be lowered into place and hence testing by pressure measurements may be costly and time consuming. Moreover, the productivity index in some wells in such that large quantities

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of oil may flow from one zone to another without producing any significant change in pressure. A zone having a productivity index of 100 barrels of oil per day per pound per square inch for example, would show a pressure drop of only $\frac{1}{10}$ of a pound per square inch in response to the escape of 40 barrels of oil per day into an adjacent zone. Although the quantity of oil lost per day in this instance is relatively large, such a loss could not readily be detected by observing the resultant change in pressure. For these and similar reasons, the use of pressure measurements as a means for testing segregation in multiple completion wells has serious drawbacks.

Efforts to develop more effective methods for testing segregation in multiple completion wells have been generally unsuccessful. It has been proposed that the chemical and physical properties of the oil from each zone surrounding a multiple completion well be determined and that the produced oil from the well be analyzed at frequent intervals in order to detect any mixing of one oil with another. Experience has shown that the differences between oils are often small and that the analytical work necessary to detect these differences is difficult and time consuming. This method is therefore practical only in isolated instances. The use of spectral adsorption in the near infrared has also been suggested as a means for identifying oil from different producing zones. This method has been found inadequate because of the similarity of the adsorption characteristics of the oils. The injection of fluorescent, metallic or radioactive tracers into one zone of a multiple completion well and the testing of oil from a second zone for the tracer material has been proposed. This is unsatisfactory because of difficulties in identifying the tracers, adverse effects of the tracers upon catalysts used in subsequent refining operations, high costs, and hazards to personnel. Other methods investigated heretofore present similar problems. As a result, the use of pressure measurements for testing segregation in multiple completion wells continues.

It is therefore an object of the present invention to provide an improved method for testing multiple completion wells to determine whether adjacent producing zones are segregated from one another in the vicinity of such wells. It is a further object of the invention to provide a reliable method for testing segregation which does not require the removal of equipment from the wellbore, can readily be carried out in the field without extensive laboratory facilities, and does not adversely affect subsequent processing of the produced oil. Other objects will become apparent as the invention is described in greater detail hereafter.

In accordance with the present invention, it has now been found that multiple completion wells can be tested to determine whether adjacent producing zones are segregated from one another in the vicinity of the wells by injecting a readily-identifiable organic compound into one zone of the well in question, shutting in the portion of the well communicating with that zone, producing oil from a second zone of the well, contacting samples of the produced oil with a solvent in which the organic compound is preferentially soluble, and thereafter testing the solvent for the presence of the organic compounds. Detection of the organic compound in the solvent clearly indicates that communication exists between the two zones; whereas absence of the organic compound after oil has been produced for a reasonable period of time can be taken as a reliable indication that the zones are effectively segregated from one another in the vicinity of the well. Lab-

oratory and field tests have shown that the method of the invention permits the detection of extremely small quantities of oil escaping from one producing zone to another, that it is readily applicable to wells in which pressure measurements and other testing methods cannot be used without the removal of equipment from the wellbore, that it has no adverse effect upon subsequent processing of the produced oil, and that reliable results can quickly be obtained in the field at relatively low cost. These features make the method of the invention considerably more attractive from an engineering and economic standpoint than methods available heretofore.

The organic compounds utilized in carrying out the method of the invention are compounds which can readily be extracted from crude oil with a suitable solvent and identified by color or a characteristic reaction following their extraction from the oil. The compounds employed should be soluble or readily dispersible in crude oil, should have low solubility in water and brine, and should be appreciably soluble in dilute acid or basic solution. They should also be stable at reservoir conditions of temperature and pressure, should be inert to crude oil and other materials found in underground reservoirs, and should not be readily adsorbed upon subsurface formations. Finally, the compounds should not occur naturally in subsurface reservoirs. A variety of organic dyes, acid-base color indicators and organic compounds capable of reacting to produce a characteristic reaction product possesses these properties and may be employed for purposes of the invention.

Specific examples of dyes suitable for purposes of the invention include gambine, picric acid, Congo red, alizarin, indigo, crystal violet, and quinoline blue. Acid-base indicators which may be employed in carrying out the invention include phenolphthalein, thymolphthalein, dichlorophenol, p-xylenesulfonphthalein, benzeneazodiphenylamine, n-cresolsulfonphthalein, thymolsulfonphthalein, o-tolueneazo-o-toluidine, o-cresolsulfonphthalein, 2,6-dinitrophenol, 2,4-dinitrophenol, p-dimethylaminoazobenzene, tetrabromophenolsulfonphthalein, tetraiodophenolsulfonphthalein, tetrabromo-m-cresolsulfonphthalein, dichlorophenolsulfonphthalein, p-nitrophenol, dibromo-o-cresolsulfonphthalein, dibromothymolsulfonphthalein, phenolsulfonphthalein, m-nitrophenol, o-cresolphthalein, sodium nitrobenzeneazosalicylate, sodium 2,4-dihydroxyazobenzene-4-sulfonate, sodium indigodisulfonate, and the like. Other compounds which may be extracted from crude oil and readily identified in similar manner include aniline, o-phenylenediamine, and related aromatic amines. The extracted organic dyes and acid-base indicators can usually be identified by their color without further treatment of the solutions containing them and hence the use of these materials is preferred for purposes of the invention.

It will be recognized that certain of the compounds described above are not of themselves readily soluble or dispersible in crude oil. Such compounds may nevertheless be employed for purposes of the invention by first dissolving them in a suitable oil-miscible solvent and then adding the resultant solution to crude oil. The solvent employed will depend primarily upon the solubility characteristics of the organic compound utilized. Suitable solvents include ethanol, isopropanol, 2-chloroethanol, trichloroethylene, acetone, ethyl ether, benzene, ethylene glycol, triethylenetetramine, isoamyl acetate, pentane, petroleum fractions boiling in the range between about 100° F. and about 500° F., and mixtures of these and similar materials. The organic compounds employed are not soluble in all of these solvents to the same extent and hence certain of the solvents are more effective with particular compounds than are others.

In utilizing the method of the invention to determine whether adjacent producing zones are segregated from one another in the vicinity of a multiple completion well, a batch of crude oil containing the dye or other readily-

identifiable organic compound to be employed is normally prepared at the surface and injected into one section of the well. Sufficient treated oil to fill the tubing string and open wellbore below it and to permit the injection of from 5 to 10 barrels or more of oil into the producing zone will ordinarily be required. The total amount necessary for a particular well can readily be calculated. The organic compound or a solution containing it is generally dissolved or dispersed in the crude oil in an amount sufficient to give a concentration between about 50 and about 1,000 parts per million. Laboratory tests have demonstrated that phenolphthalein and similar compounds can readily be extracted from crude oil and identified when present in the oil in concentrations as low as 10 parts per million. Somewhat higher concentrations are usually advisable, however, because of the possibility that some of the organic compound may be adsorbed on the tubing or subsurface strata with which the oil comes into contact. The use of higher concentrations also assures identification of the compound even though the injected oil may be diluted with a large quantity of oil from an adjacent producing zone. Use of the organic compound in concentrations between about 250 and about 750 parts per million is preferred.

The crude oil containing a dye or similar organic compound is pumped through the tubing into the high pressure zone of the well to be tested. An indication of the pressures in the various zones can ordinarily be obtained from the production records of the well. Where all of the zones are at essentially the same pressure, a series of tests as described hereafter may be advisable. After from about 5 to 10 barrels of the treated crude oil has been injected into the producing zone, the tubing or other conduit used for injection purposes is closed off at the surface. Production of crude oil from the adjacent producing zones is then resumed. Initial samples of the produced crude are collected. After each of the adjacent zones has produced a quantity of oil corresponding to that initially present in the wellbore, sampling of the produced fluids at periodic intervals is commenced. The sampling rate will depend primarily upon the rate at which fluids are produced from the well. In general it is preferred to collect one or more samples for each four to five barrels of crude oil produced over a period of at least 24 hours. If the daily production of the producing zone is less than the amount of treated crude oil injected into the well, the sampling period may be continued for a somewhat longer period. At the end of the sampling period, a final sample of the injected oil is obtained from the high pressure zone of the well. Each sample should be carefully identified at the time that it is collected.

The samples of crude oil collected as described above are treated with an oil-immiscible solvent in order to extract any dye or other injected organic compound present in the oil. The solvent employed will depend primarily upon the particular compound to be extracted. In the case of organic dyes for example, a dilute aqueous solution of sulfuric or hydrochloric acid will ordinarily be satisfactory. Aniline and similar compounds can generally be extracted from the crude oil with a dilute aqueous solution of an inorganic acid or with an aqueous solution of alcohol to which a small amount of acid has been added. The acid-base indicators may be extracted with water or with a dilute acid or alkaline solution. If water is employed for the extraction of an indicator, the pH of the extract may later be adjusted to determine whether the indicator is present. An alternate procedure is to employ an aqueous buffer solution to extract the indicator from the crude oil. Satisfactory buffers include solutions of hydrochloric acid and potassium chloride, solutions of hydrochloric acid and potassium acid phthalate, solutions of sodium hydroxide and potassium acid phthalate, solutions of sodium hydroxide and potassium diacid phosphate, and solutions of sodium hydroxide and boric acid. The buffer solution utilized will depend, of course, upon

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the pH level at which the indicator to be extracted undergoes a color change and should be selected so that the solution will have a pronounced color if the indicator is present.

In order to extract the dye or other injected organic compound from the oil, the crude oil sample is mixed with an equal volume of aqueous solution and agitated. The resultant mixture is then allowed to stand until an aqueous phase and a separate oil phase form. Formation of the separate phases can usually be accelerated by warming the mixture, centrifuging it, or filtering it through a water-wet filter. The addition of a demulsifier to the mixture to promote breaking of the emulsion should generally be avoided because of the possibility that the demulsifier may prevent identification of the dye or other compound extracted from the oil.

The method utilized to identify material extracted from the crude oil sample will depend upon the particular compound injected into the well. If a dye has been injected and communication exists between adjacent producing zones, an aqueous dye solution having a pronounced color will form beneath the oil as the mixed oil and aqueous solvent separate. If the producing zones are segregated from one another, on the other hand, the aqueous solvent phase will remain essentially colorless due to the absence of dye in the crude oil sample. Where the injected organic compound is an acid-base indicator and the producing zones are not segregated from one another, the color of the aqueous layer beneath the oil will again indicate the presence of the indicator in the oil sample. Unless a buffer solution is used to extract the indicator from the oil, it may be necessary to adjust the pH of the aqueous phase in order to obtain the indicator color. Even though the pH is adjusted, the aqueous phase will remain colorless if no communication exists between producing zones in the vicinity of the well. Aniline injected into one producing zone and extracted from an oil sample recovered from an adjacent zone can be detected by adding a small amount of solid lead peroxide to the aqueous phase following the extraction step and then warming the solution slightly. The appearance of a green color attests to the presence of aniline and indicates that communication exists between the zones. A failure to obtain the green color can be taken as an indication that the zones are effectively segregated. Similar procedures can be utilized with other organic compounds which readily react to form a characteristic color or undergo a characteristic reaction upon treatment with a particular reagent.

By testing the crude oil samples in the manner described above, it can readily be determined whether any of the samples included oil from the producing zone into which the dye or other organic compound was injected. The testing of samples of the injected oil and samples of oil recovered from adjacent zones at the onset of the operation provides a means for checking the results obtained.

As pointed out earlier, it may be necessary to carry out a series of tests where pressures in adjacent producing zones are essentially the same or are unknown. In such instances, each producing zone may be tested in sequence in the manner described above. A different organic compound should be employed in each test and the compounds should be selected so that one will not prevent the detection of another. One method for doing this is to employ acid-base indicators which are colorless below pH levels. The use of a series of such indicators with buffer solutions of increasing pH values permits the ready detection of each indicator even though those employed earlier were not completely removed from the system.

The technique described in the preceding paragraphs provides a mean for determining whether communication exists between adjacent producing zones within a multiple completion well or in the vicinity of such a well. In some cases it may be desirable to test for leaks in the tubing or crossover within a well without injecting oil into the producing formation. In that even, a suitable organic

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compound may be incorporated in a stick of wax or other oil-soluble material and dropped to the bottom of one conduit of the well. The organic compound may also be introduced in a soluble or frangible container dropped into the well. Oil recovered from an adjacent conduit can then be treated with a solvent to extract the organic compound. Presence of the compound in the oil thus treated indicates that a leak exists between the conduits.

The nature and object of the invention are further illustrated by the results of tests carried out to determine the effectiveness of the method.

Example 1

In a first series of tests, phenolphthalein was added to a mixed methanol-isopropanol solution in a concentration of one pound per gallon. This solution was then added to samples of crude oil in amounts sufficient to give phenolphthalein concentrations ranging from about 10 parts per million to about 500 parts per million. These samples and samples of the same crude oil containing no phenolphthalein were mixed with equal volumes of an aqueous buffer solution containing boric acid and sodium hydroxide in 0.1 molar concentrations. The buffer solutions had a pH of about 10.5. The mixed oil and buffer solution samples were vigorously agitated and then allowed to stand for a period of about 8 hours. The emulsion formed by shaking the samples gradually settled and oil and aqueous phases were formed in each sample bottle. At the end of the 8-hour period it was found that the aqueous phase beneath each oil sample containing phenolphthalein had a pronounced red color, even in those cases where the phenolphthalein had been added to the crude oil in a concentration of only 10 parts per million. The buffer solution contacted with the oil samples containing no phenolphthalein remained essentially colorless. It is thus apparent that the aqueous buffer solution extracted the phenolphthalein from the crude oil and that this technique can be employed to detect the dye in extremely low concentrations.

Example 2

One milliliter of aniline was added to a hundred milliliters of crude oil in the first of a second series of experiments. The oil containing aniline was then mixed with an equal volume of 2 percent sulfuric acid solution. The mixture was shaken vigorously for several minutes and allowed to stand until the emulsion produced had settled and separate oil and water phases had been formed. A small amount of solid lead peroxide was then added to the water phase. The solution was heated slightly. The heating resulted in the formation of a bright green color characteristic of the reaction of aniline with lead peroxide. Similar tests at other concentrations demonstrated that the aniline could be extracted from crude oil and identified in concentrations as low as 10 parts per million. The tests also showed that a 5 percent hydrochloric acid solution could be employed in lieu of the sulfuric acid.

Example 3

Further tests were carried out by adding p-dimethylaminoazobenzene to samples of crude oil. These samples were mixed with equal quantities of a 2 percent solution of sulfuric acid. The oil-acid mixture was shaken for several minutes. The emulsion formed was then broken by filtering the mixture through a double water-wet filter paper. Upon separation of the crude oil and acid, the characteristic red-yellow color of the aqueous phase confirmed the presence of p-dimethylaminoazobenzene. Further evidence of the presence of the dye was obtained by adding dilute sodium hydroxide to the aqueous phase. Addition of the base produced a change in pH such that the color quickly changed to yellow. It was found that these color changes permitted detection of the p-dimethylaminoazobenzene in concentrations as low as 10 parts per million.

Example 4

The effectiveness of the method of the invention was further tested in a multiple completion well containing a leak in the down-hole assembly. The leak had been deliberately produced by drilling a $\frac{3}{32}$ inch hole through the crossover in the tubing string. Such a leak is much too small to be detected by pressure measurements. A test solution was prepared by first dissolving phenolphthalein in isopropyl alcohol in a concentration of one pound per gallon and then adding this solution to crude oil from the well at the rate of one gallon for each 252 gallons of oil. The concentration of the phenolphthalein in the prepared crude oil was thus about 500 parts per million. A sample of the treated oil and untreated oil samples from each of the producing zones in the well were collected. The treated oil was then injected into the high pressure tubing string in the well until about 5 barrels of oil had been forced into the formation. This tubing string was then shut in at the surface and oil was produced from an adjacent producing zone through a second tubing string. After a quantity of oil equivalent to the calculated volume of the low pressure section of the well had been recovered, samples of the produced crude were taken at 4-hour intervals over a 24-hour period. Each sample consisted of about 300 milliliters of oil and was carefully labeled to indicate its source and the time at which it was taken. At the end of the 24-hour period, final samples from both the high pressure zone and the low pressure zone of the well were collected.

Each of the samples obtained in the above manner was mixed with an equal volume of an aqueous buffer solution containing boric acid and sodium hydroxide. The solution employed had a pH slightly in excess of 10. The samples of oil and buffer solution were shaken and allowed to stand for a period of 8 hours in order to permit separation of the oil and solution. It was found that the aqueous solution in contact with the samples of treated oil and the solution in contact with samples produced from the low pressure zone of the well had an intense red color. The solution contacted with oil samples taken prior to the introduction of the phenolphthalein into the well were essentially colorless. The results thus showed that the treated oil containing phenolphthalein flowed from the high pressure tubing string into the low pressure zone in the well and confirmed the existence of the leak in the down-hole assembly.

Example 5

Additional field tests were carried out using phenolphthalein in the manner described above to test segregation in multiple completion wells which had been previously tested by conventional pressure measurements. It was found that the method of the invention corroborated the existence of communication between adjacent producing zones in those instances where the prior pressure tests had indicated that the zones were not segregated from one another. Where the earlier pressure tests had indicated segregation, the method of the invention gave similar results.

It will be understood that the method of the invention is not restricted to the use of the particular compounds recited in the foregoing examples and that other organic compounds which can be readily extracted from crude oil with an aqueous solution and identified by their color or by the formation of a characteristic reaction product may be employed. A variety of suitable compounds in addition to those set forth above will suggest themselves to those skilled in the art.

What is claimed is:

1. A method for determining whether communication exists between adjacent zones in a multiple completion well which comprises introducing an organic compound foreign to subsurface oil-bearing reservoirs into a first zone of said well, said compound being selected from the

group consisting of oil-soluble and oil-dispersible dyes, acid-base color indicators and aromatic amines which react to form products of characteristic color; producing crude oil from a second zone of said well; collecting samples of the oil produced from said second zone at periodic intervals; treating said samples with sufficient quantities of an oil-immiscible solvent in which said organic compound is preferentially soluble to extract said compound from said samples and thereafter determining the presence or absence of said organic compound in said solvent.

2. A method as defined by claim 1 wherein said organic compound is an oil-soluble dye.

3. A method as defined by claim 1 wherein said organic compound is an acid-base color indicator.

4. A method as defined by claim 1 wherein said organic compound is aniline.

5. A method as defined by claim 1 wherein said organic compound is introduced into said first zone by incorporating said compound in a stick of oil-soluble material and dropping said stick into said first zone.

6. A method as defined by claim 1 wherein said organic compound is introduced into said crude oil in said first zone by dissolving said compound in an oil-miscible solvent and introducing said solvent into said first zone.

7. A method for testing segregation in a multiple completion well which comprises introducing an acid-base color indicator into a first producing zone surrounding said well, shutting in said first producing zone, producing crude oil from a second producing zone surrounding said well, collecting samples of crude oil at periodic intervals as said oil is produced from said second zone, extracting samples recovered from said second producing zone with an aqueous solution in which said acid-base indicator is preferentially soluble, and thereafter determining the presence or absence of said indicator in said solution.

8. A method as defined by claim 7 wherein said acid-base indicator is phenolphthalein.

9. A method as defined by claim 7 wherein said aqueous solution is a dilute solution of an inorganic acid.

10. A method as defined by claim 7 wherein said acid-base indicator is p-dimethylaminoazobenzene.

11. A method for testing segregation in a multiple completion well which comprises injecting crude oil containing aniline into a first producing zone surrounding said well, shutting in said first producing zone, producing crude oil from a second producing zone surrounding said well, contacting crude oil from said second producing zone with a dilute aqueous solution of an inorganic acid, and thereafter treating said dilute aqueous solution with lead peroxide.

12. A method for determining whether communication exists between adjacent producing zones in the vicinity of a multiple completion well which comprises injecting crude oil containing from about 10 to about 1000 parts per million of phenolphthalein into a first of said producing zones through one conduit of said well, shutting in said first conduit, producing crude oil from a second of said producing zones through a second conduit of said well, collecting samples of crude oil produced from said second conduit, contacting said samples with an aqueous medium in which phenolphthalein is soluble, and thereafter determining the presence or absence of phenolphthalein in said medium.

13. A method as defined by claim 12 wherein said aqueous medium has a pH in excess of about 10.

14. A method as defined by claim 12 wherein the pH of said aqueous medium is adjusted to a value in excess of about 10 after said samples are contacted therewith.

15. A method for testing segregation in a multiple completion well penetrating two subterranean oil-bearing zones which comprises dropping wax containing an acid-base color indicator into a first conduit in said well penetrating a first of said oil-bearing zones, producing crude oil from

a second conduit in said well penetrating a second of said oil-bearing zones, collecting samples of the crude oil produced from said second conduit at periodic intervals, treating said samples with an oil-immiscible solvent in which said indicator is preferentially soluble to extract said indicator from said oil, and determining the presence or absence of said indicator in said solvent.

16. A method as defined by claim 15 wherein said indicator is phenolphthalein.

17. A method as defined by claim 15 wherein said indicator is p-dimethylaminoazobenzene.

18. A method as defined by claim 15 wherein said solvent is a dilute acid solution.

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