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MICROSCOPIC STUDY OF OIL RECOVERY BY CARBON DIOXIDE

Final Report, October 1, 1978 - August 31, 1980

Work Performed for the Department of Energy
Under Contract DE-AS21-78ET12095

Date Published—December 1980

University of Alabama
Tuscaloosa, Alabama

U. S. DEPARTMENT OF ENERGY

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MICROSCOPIC STUDY OF OIL RECOVERY BY CARBON DIOXIDE

Final Report
October 1, 1978 – August 31, 1980

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I. ABSTRACT

Visual examinations of microscopic behavior of CO_2 flooding processes were conducted using a high pressure glass-bead packed transparent flow tube. The apparatus and techniques developed have made it possible to observe the physical phenomena of the displacement of oil by CO_2 under both miscible and immiscible conditions. Effects of CO_2 slug size and formation dip on oil recovery were also investigated. The results have been recorded in a series of magnified color photographs for after-run study and interpretation.

Four natural crude oils ranging from API gravities of 15 to 48 were chosen for displacement tests. The majority of the tests were performed at a temperature of 120°F and pressures from 1000 to 2500 psi.

Three types of displacement, namely, (a) immiscible, (b) semi-miscible, and (c) miscible, were observed within the testing pressure range. Types (a) and (c) have been well defined in the literature. Type (b) is a dispersion process in which oil is disintegrated into micro-size particles and transported in the CO_2 stream. More than one type of displacement could exist simultaneously in a flooding system.

A CO_2 slug that is too large is wasteful and can cause early CO_2 breakthrough. A slug that is too small would allow the trailing water to channel through the oil bank and thus degenerate to an ordinary waterflooding; nevertheless, the oil recovery would be significantly increased over that achieved by a straight waterflooding.

Recovery of residual oil in a waterflooded area could be accomplished by either semi-miscible (without oil bank) or miscible displacement. Oil swelling appears to play a major role in forming an oil bank. In a waterflooded heavy oil reservoir, the characteristics of oil swelling in

conjunction with viscosity reduction could create favorable conditions for CO_2 process including well stimulation.

Oil recovery can be increased considerably by down-dip CO_2 displacement. The increase is derived primarily by reducing CO_2 override and improvement in miscibility.

II. INTRODUCTION

The use of CO_2 for oil recovery has received much attention in enhanced oil recovery (EOR) methods. Various studies estimate that about 7 to 21 billion barrels of oil can be produced using CO_2 miscible method out of an estimated total EOR potential of 18-52 barrels.[1] Oil production from CO_2 projects currently is 22,000 barrels/day which accounts for about 6% of the total U.S. EOR production in 1979.[2]

Since the largest CO_2 project was instituted at the SACROC Unit of the Kelly Snyder field in 1972, the number of field CO_2 tests has grown to twenty in 1980. Some of these projects are commercial applications such as SACROC, Crossett and Twofreds, but the majority are field pilot tests. The purpose of these tests are to determine the applicability of CO_2 in heavy oil recovery, low temperature reservoirs, hot and steep dip reservoirs, etc.

Abundant data have been obtained from the field CO_2 tests. In many cases, the data are difficult to analyze and the interpretations are ambiguous because of the complicated nature of phase behaviors and reservoir conditions. Predicted performance using the available simulation models are not reliable because the interactions of CO_2-crude oil and the displacement mechanisms are not fully understood.

From field tests and laboratory studies, a number of problems have been identified. Some of these problems are discussed briefly as follows:

1. Phase Behavior of CO_2-Oil Systems

In the CO_2-oil recovery system, the compositions of the gas and liquid phases are changed constantly due to dissolving and extracting characteristics of CO_2. At certain conditions, as many as four phases,

including two liquids, a gas and a solid are in equilibrium.[3] In a PVT cell, the solid precipitates and the liquids tend to segregate because of density contrasts. Little information has been reported on how the multiple phase would affect CO_2 injection and oil recovery.

2. Miscible Pressure

Presently the pressure required for miscible displacement of oil by CO_2 is mostly determined by using the slim-tube method.[4] In this method, the miscible pressure is found indirectly based upon oil recovery efficiency. Under reservoir conditions, however, the development of a miscible zone is also affected by other parameters including gravity override, formation dip and injection rate. In addition, the presence of minor components such as methane or nitrogen can drastically increase the pressure needed for miscibility. Inaccurate miscible pressure data can result in great losses in oil production and create operational problems.

3. Displacement Mechanisms

Although the CO_2 process has been extensively applied in field tests, the nature of displacement mechanisms have not been fully understood. Homes[5] has reported the types of CO_2 floods based on recovery and hydrocarbon composition measurements from slim-tube tests. These experiments investigated the composition and extent of the transition zone from the effluence. However, the interactions between CO_2 and oil within the sand-pack are not known. Also, the mechanism of continuing recovery of residual oil behind the miscible zone and the effects of a gaseous phase on oil displacement are rarely known with much certainty.

4. CO_2 Channelling and Overriding

Although CO_2 reduces oil viscosity upon dissolving in oil, the viscosity ratios are still unfavorable causing premature CO_2 breakthrough. Water and gas injection (WAG) has been used to reduce the channelling effect[6], however, the injected water often would react with CO_2 and prevent CO_2 from contacting oil. This interference would be further aggravated in a waterflooded reservoir.

In-situ foam has been studied for CO_2 mobility control[7], but the problems of absorption and stability of foaming chemicals under reservoir conditions will require a large amount of research work to be solved.

CO_2 override appears to be another detrimental factor in reducing sweep efficiency. Although the density contact between CO_2 and reservoir oil is small compared to hydrocarbon gas flooding, the gravity override could still be a serious problem causing early CO_2 breakthrough especially in a waterflooded reservoir. Down-dip CO_2 displacement would reduce override should the formation be steeply inclined. Nevertheless, because of the complex phase change in the CO_2-oil-water system, the actual displacement mechanisms are still unproven.

From the above discussion, it is apparent that the CO_2 process is still very much in the research phase and some facets are speculative. Fundamental knowledge and understanding of the CO_2 displacement process are essential in order to achieve engineering and economical success in field application.

The research program at The University of Alabama is a microscopic study of CO_2 displacement mechanism. This program was planned to accomplish two tasks:

1. To construct a long, transparent high pressure laboratory flow model and to develop proper illumination techniques for taking detailed photographs of oil recovery processes via CO_2 flooding.

2. To conduct CO_2/water displacement tests under various conditions including miscible and immiscible CO_2 floodings performed as secondary and tertiary recovery modes. The results from the experiments will be presented in a series of photographs so that the flooding performance can be interpreted in terms of phenomena observed in the porous medium.

At the completion of the project, useful information and new concepts relative to CO_2-oil-water displacement will be acquired. As a result, realistic solutions to the existing problems and also new areas of research could be identified.

III. TASK 1

Construction of High Pressure Transparent Flow Tube

A. Transparent Flow Tube

The transparent flow tube is basically a high pressure liquid level gage manufactured by Jerguson Gage & Valve Company, Burlington, Massachusetts. The flow tube has an overall length of five feet to withstand a working pressure of 4500 psi at 120°F. A 316 stainless steel gage was originally ordered; however, due to unexpected difficulties, a carbon steel gage was used in order to avoid further delay in delivery.

The flow tube consists of five sections with an overall length of 152 cm. Each section has two 1.27 cm by 25.7 cm glass "windows", one on the front and one on the rear. The distance between the two glass windows in each

section is 1.67 cm. This distance proved too wide to retain its transparency when it was filled with glass beads and crude oil. The problem was finally resolved with the insertion of a specially made glass bar designed to reduce the clearance to 2.2 mm as shown in Fig. 1. This final clearance was adopted in order to accommodate two layers of glass beads and still permit light penetration for visual investigation and photography.

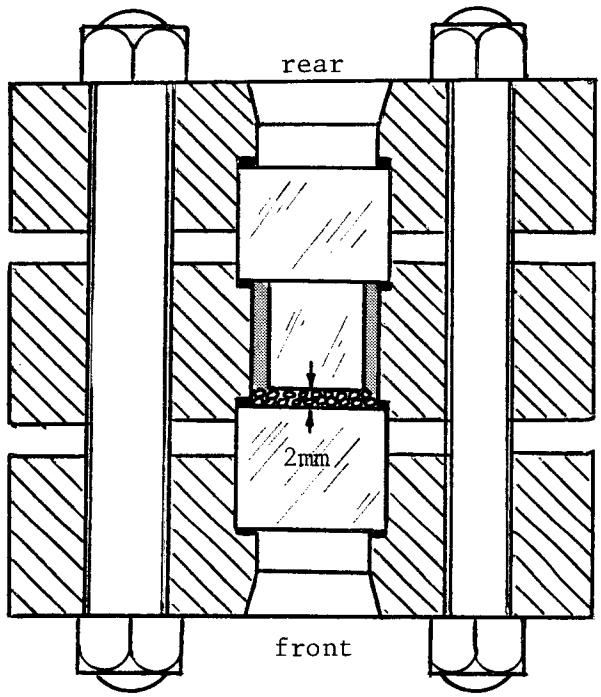
After glasses were installed, the bolts of the flow tube were tightened up to 40 in/lb using a torque wrench. The flow tube was then set in a vertical position and the clear glass beads (1.00-0.84 mm) were poured slowly into the flow tube. Slight tapping was needed to prevent clogs of glass beads at the tight spots. A double-layered glass-bead pack was formed as expected; however, single-layer packing was also found in the 4th and 5th sections of the flow tube.

The measured porosity of the glass-bead pack was 52% compared to the ideal 35% had the flow tube been perfectly packed. The excessively high porosity was primarily attributable to the single-layer pack and the large voids formed along the upper edge of the flow tube. The total pore volume was 47.8 cm^3.

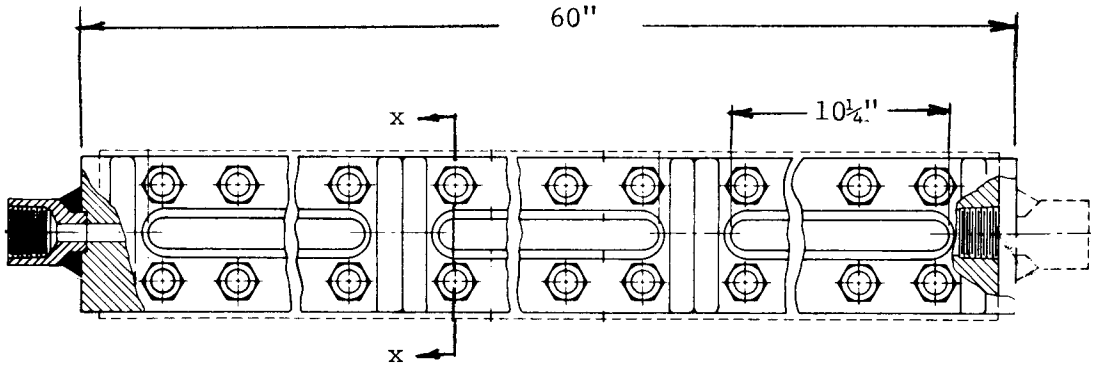
The packed flow tube was tested up to a maximum pressure of 3500 psi at room temperature and has performed satisfactorily without leaks or mechanical troubles. The only problem so far encountered has been a build-up of film on the glass beads. The brownish film not only reduced light intensity but also obscured the color effect.

Various solvents and cleaning agents were tested for removing the film. It was found that a 2N caustic solution was effective in loosening the film coatings from the glass beads after soaking for about 48 hours. The

Fig. 1. Sketch of Transparent Flow Tube



Enlarged x-x cross-section



Five-Section Transparent Flow Tube

disintegrated film was then flushed with high rate water injection. Squirts of CO_2 were required to provide surges for complete removal of the films from the porous medium.

This cleaning routine was required after six or seven runs. We found that the light Penn-Grade and the medium SACROC crude oil had more film problems than that of heavier Slaughter Estate crude. It is reasonable to assume that the same film would have precipitated in the reservoir rock thus causing plugging problems. The caustic flushing method which has been used successfully in the special laboratory unit may not be applicable in reservoir rock. A solvent in which the film is dissolvable would be most desirable for use in the natural rock matrix.

B. Microscope and Camera

An American Optical 52 ML-1 microscope equipped with a 35-mm camera was used for observations and photographic study. The microscope has a long working distance of 8 inches and five selective magnifications of 10x, 15x, 20x, 30x, and 40x. Most of the photographs were taken from sections 1, 3, and 5 of the flow tube. Normally magnifications of 15x and 20x were used for large area coverage and 40x for detailed demonstrations. The exposure time was one second. There is no aperture adjustment on the camera.

Five universal microscope illuminators, one for each window, were placed behind the transparent tube. The illuminator casts a light spot of approximately 2 cm diameter. An extra illuminator was placed at the entrance of the first window to give views of early arrival of CO_2 front.

Kodak Vericolor II color negative film was used initially. It was later changed to high-speed Kodacolor 400. The later gave better color rendition at low lighting conditions. Commercial service was used for developing and printing.

C. Pumps

A Ruska single-cylinder proportioning pump was used for water displacement. The discharge rate was set at 8 cm^3/hr corresponding to a pore space velocity of 101 feet/day. The discharge can be switched to either water or CO_2 injection.

A Milton-Roy Mini-Pump was employed for various auxiliary services including sand pack cleaning and resaturation.

D. Constant Temperature Chamber

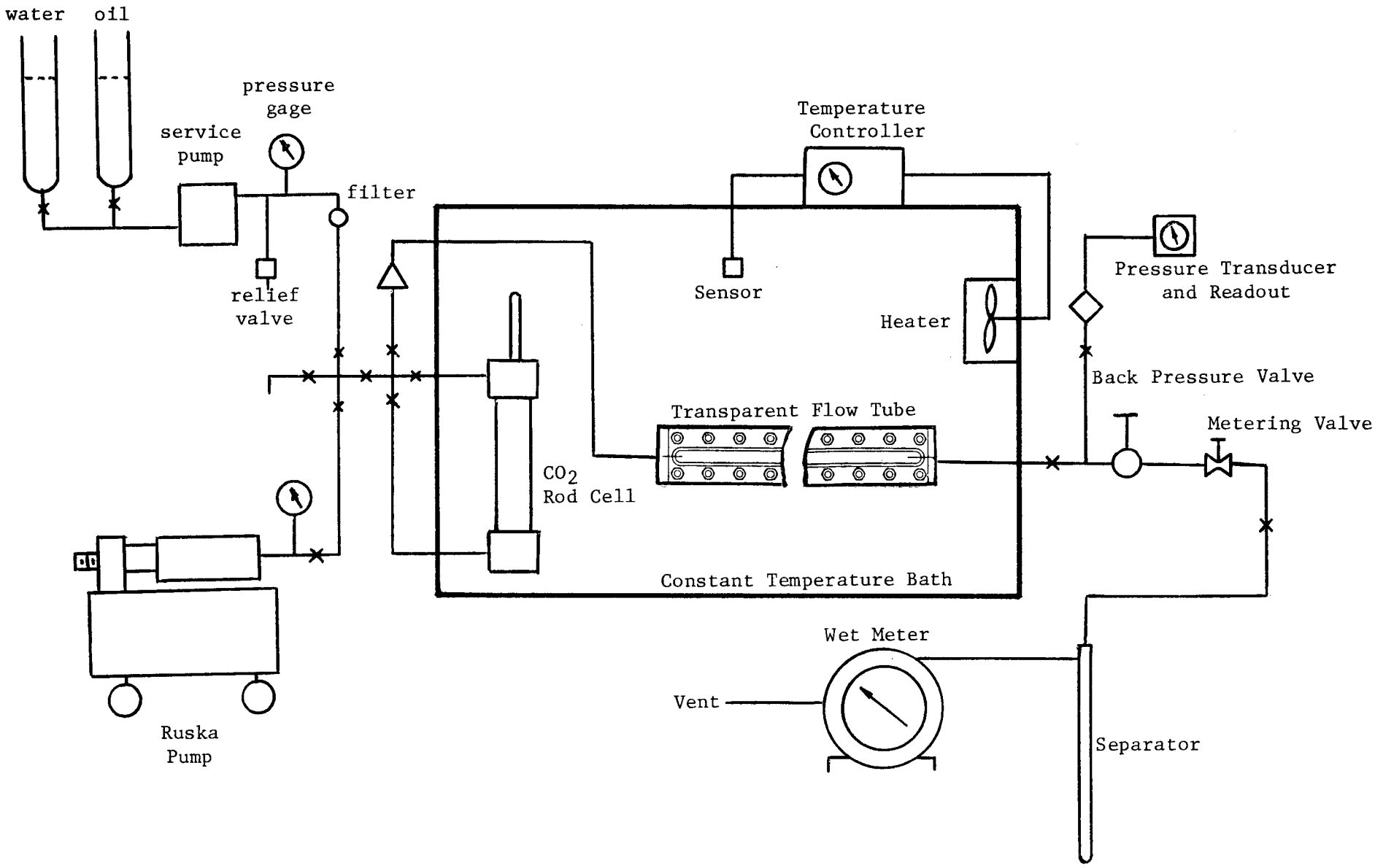
A 2'3" x 1'10" x 8' insulated chamber was fabricated to contain the transparent flow tube and CO_2 cell. The chamber was heated using a Nautilus heater and controlled by a RFL temperature controller. Insulated windows were used for temperature stability.

E. CO_2 Rod Cylinder

The rod cylinder serves as a transfer vessel within which CO_2 was stored at the desired pressure and temperature. In the cylinder, the CO_2 was separated by a piston to avoid direct contact with the driving water discharged from the Ruska pump. A rod to which the piston is connected was used as a flag to indicate the amount of CO_2 that remained in the cylinder. The cylinder was designed with a capacity of 210 cm^3 and a working pressure of 4000 psi.

The high pressure visual flooding system is diagrammatically shown in Fig. 2. Photos 1a and 1b exhibit the tranparent flow tube and other related installations.

Fig. 2. Diagram of High Pressure Visual CO_2 Flooding Systems



IV. TASK 2

Conduct CO_2/Water Displacement Tests

A. Selection of Crude Oil

Four different natural crude oils were used for CO_2 displacement tests. The physical properties of these crude oils are given below:

Crude Oil	API	Viscosity @ 120°F
Penn Grade	48°	1.4 cp
SACROC	41°	1.7 cp
Slaughter Estate	33°	3.9 cp
Wilmington Tar Zone	15°	27.3 cp

As can be seen, the specific gravity covers a wide range from light to heavy crude oil. All the samples were stabilized stock tank oil. Solution gas was not recombined for the CO_2 flooding tests.

The Penn Grade crude oil was from Bradford Field Pennsylvania supplied by the Pennzoil Company. This crude oil has a straw yellow color with good light transmissibility.

The SACROC and Slaughter Estate crude oils are from West Texas provided by Chevron and Amoco respectively. Both oils are black and impermeable to light. The Slaughter Estate crude has strong odor of H_2S and is viscous at room temperature. It shows colorful carmine red or orange-yellow when mixed with CO_2.

The Wilmington Field Tar Zone crude oil was provided by Champlin Petroleum Company. This crude oil was used only for CO_2-foam displacement tests. The field has been waterflooded and the company is now preparing for a large scale CO_2 test in the Tar Zone at Wilmington.

B. Preparation for CO_2 Flooding

The glass-bead pack was cleaned after each run with toluene then followed by isopropyl alcohol. The cleaning work was conducted at room temperature and atmospheric pressure.

Crude oil was then injected into the water saturated pack at the desired flood temperature and pressure. The amount of water displaced was measured and the irreducible water saturation was determined by material balance calculation. At this point, the transparent flow tube was ready for secondary CO_2 flooding process. Should a tertiary CO_2 flooding be desired, the oil saturated pack was waterflooded down to irreducible oil saturation before CO_2 injection was started. The CO_2 and water were injected at a rate of 8 cm^3/hour using the Ruska pump.

The CO_2 used for CO_2 floods has a purity of 99.8%. It was contained in a 1.54 ft^3 steel cylinder equipped with an eductor tube. The pressure of the liquid CO_2 in the cylinder was 830 psig at room temperature. The liquid CO_2 in the large cylinder was transferred to the CO_2 rod-cylinder in the temperature bath and compressed to the desired pressure by pumping water into the rod-cylinder. Tap water was used for saturation and injection. In order to identify CO_2 from water in the glass-bead pack, the water was dyed red with food color. It was later found, however, that the color was not stable and completely faded away when contacted with CO_2. Also, the dyed water had an undesirably weak light effect and masked the true color of the crude oil. For these reasons, the use of dyed water was discontinued after several Penn Grade oil runs.

C. CO_2 Flooding Tests

The CO_2 flooding tests were performed in the following sequences:

(a) Straight secondary CO_2 flooding tests: Flooding pressure started at 500 psi and was increased in steps until miscible displacement was observed. Three or four runs were usually required.

(b) Secondary CO_2 slug flooding tests: Four runs with CO_2 slugs of 0.1, 0.2 and 0.4 pore volume (pv) were normally performed. Displacements were conducted at miscible pressure as determined in (a).

(c) Tertiary CO_2 slug flooding tests: CO_2 slugs and pressure were similar to secondary flooding tests.

All the flooding tests were run at a temperature of 120°F.

1. Straight Secondary CO_2 Flooding

A total of nine runs were conducted using Penn Grade, SACROC, and Slaughter Estate crude oils. The purpose of the tests was to observe the interactions between CO_2 and crude oils at miscible and immiscible conditions. Test results are shown in Table 1 and plotted in Fig. 3.

(a) Immiscible Displacement

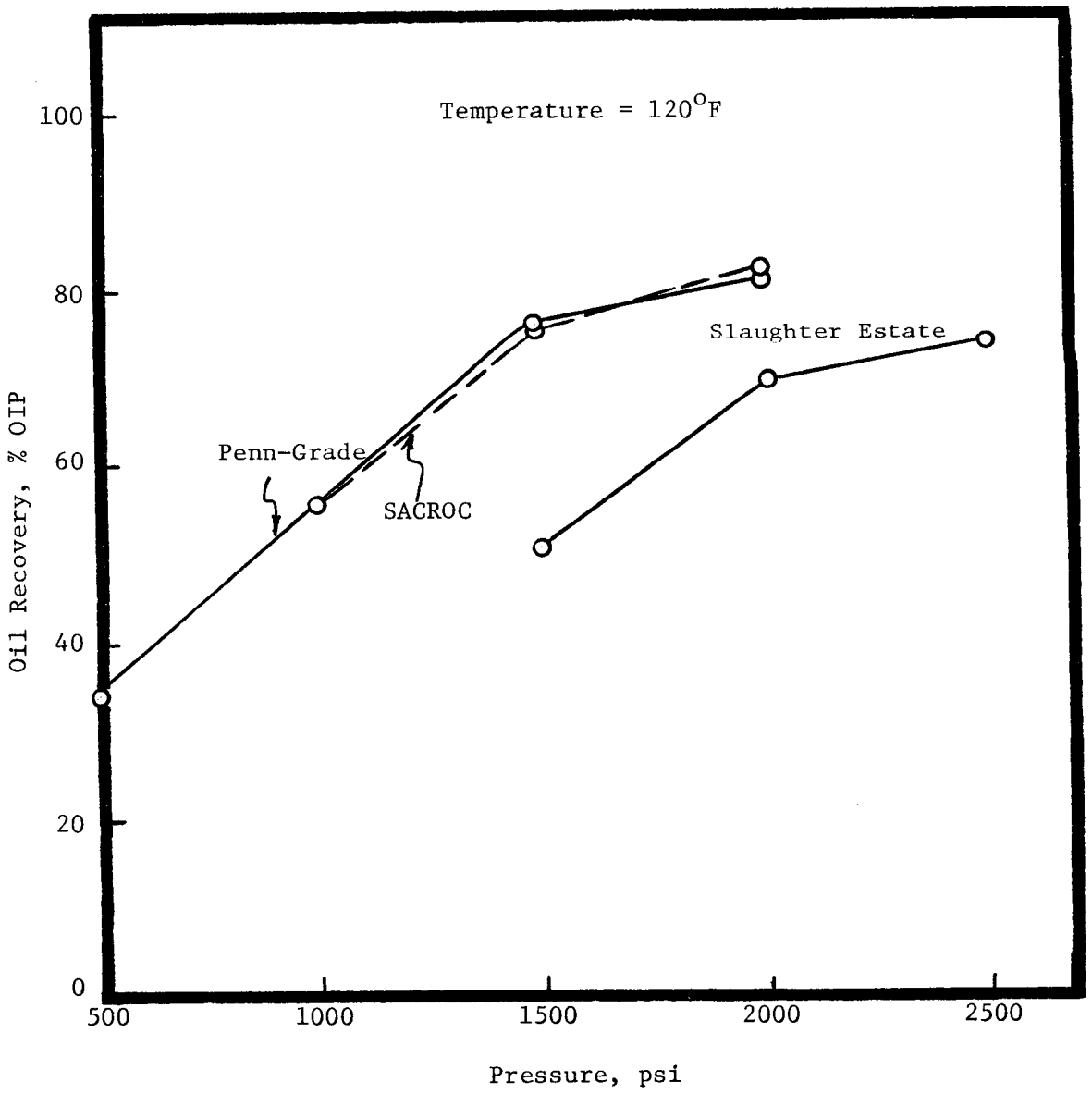
At the pressures of 500 psi and 1000 psi, (run nos. PG-1 and SA-1), both the Penn Grade and SACROC oils were not miscible with CO_2. Gaseous CO_2 was overriding and flowing along the upper edge of the flow tube. After an initial surge of CO_2 at the inlet, some of the overriding CO_2 went into solution in oil and connate water. This caused the advancement of CO_2 to slow down or sometimes retreat due to oil expansion.

TABLE 1

Straight Secondary CO_2 Flooding
120^oF

Crude Oil	Run Number	Pressure psi	Oil Recovery % OIP
Penn-Grade	PG-1	500	33.50
	PG-2	1500	76.34
	PG-3	2000	81.32
SACROC	SA-1	1000	55.55
	SA-2	1500	75.88
	SA-3	2000	82.27
Slaughter Estate	SE-1	1500	50.48
	SE-2	2000	69.18
	SE-3	2500	72.62

Figure 3. Pressure Effect on Oil Recovery



Besides moving horizontally, the overriding CO_2 also encroached downward to form a teeth-shaped phase boundary with the underlying crude oil. Fig. 4 is a diagrammatic sketch illustrating immiscible-type CO_2 displacement. Photos 2a to 2f show CO_2 override and CO_2-oil boundaries. As can be seen, the SACROC oil changed from reddish brown to yellow when solution CO_2 was increased. The residual oil in the CO_2 invaded zone existed in two forms: (a) clusters or globules occupying the large pores, and (b) oil rings surrounding the pendular connate water.

Oil recovery was 33.5% of OIP for Penn Grade oil at 500 psi and 55.5% of OIP for SACROC oil at 1000 psi. This is the lowest recovery percentage compared to those at higher displacement pressures. The low recovery was caused by CO_2 override and large residual oil saturation.

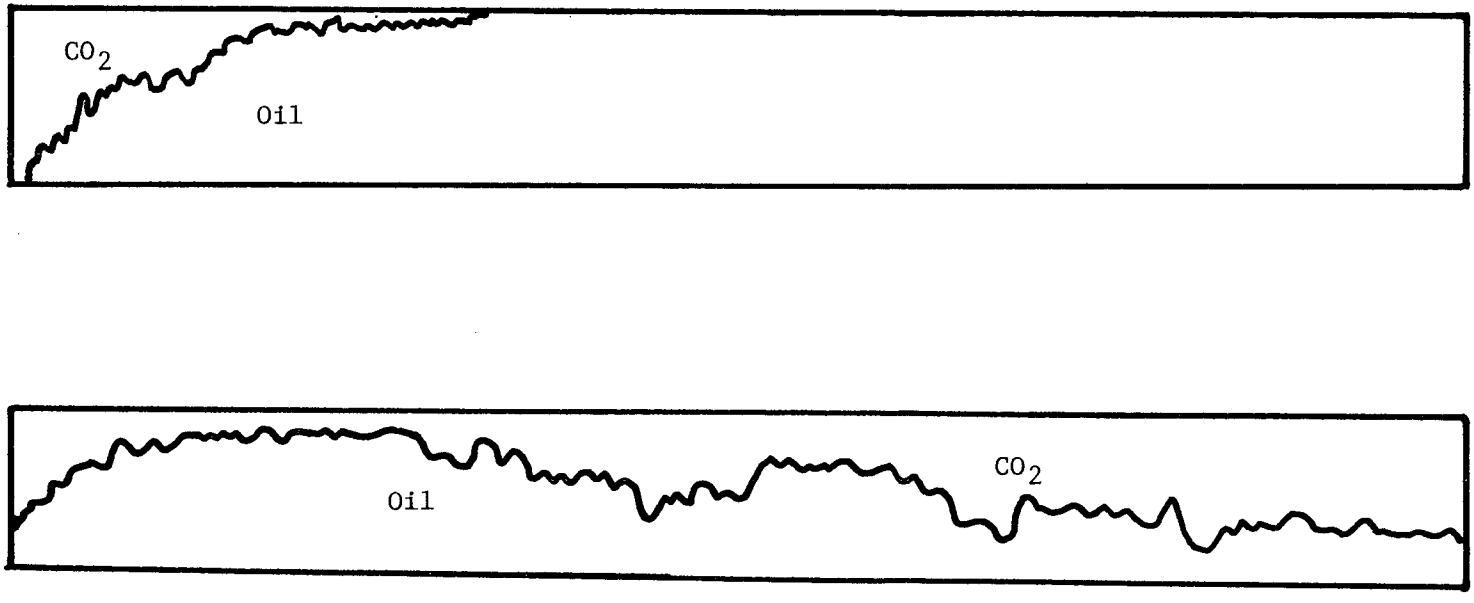
Though the recoveries from CO_2 flooding were low, the blowdown recovery accounted for 15.6% of OIP for Penn Grade oil and 22.2% for the SACROC oil. It was observed that when the system pressure was bleeding down, gas bubbles evolved vigorously from the residual oil. As a result, significant amounts of the trapped oil was mobilized by gas expansion and and recovered during the blowdown stage.

The lowest flooding pressure for the Slaughter Estate oil was 1500 psi. At the pressure, immiscible displacement as described above was observed in most of the flow tube. A small miscible mixture, however, was observed in the last section of the flow tube. It is not known whether a miscible displacement could be developed should the flow tube be longer.

(b) Miscible Displacement

Miscible condition was achieved for both Penn Grade and SACROC crude oils when injection pressure was increased to 1500 psi. The miscible

Figure 4. Sketch of Secondary Immiscible Displacement



mixture was formed instantly at the inlet. However, the Slaughter Estate crude oil required a longer distance for the development of miscible zone.

Penn-Grade crude oil: Prior to CO_2 injection, the oil saturated pack showed that the red connate water (dyed) was existing in pendular rings. Small clusters of water lodged in large pores were also observed.

As soon as CO_2 emerged at the entrance of the flow tube, the oil and water colors quickly faded and no CO_2-oil phase boundary could be observed near the CO_2 front. Interestingly, the pendular connate water also became invisible in the miscible zone.

The disappearance of connate water may be explained in terms of Reflectory Index. The Reflectory Index for Penn-Grade crude oil was 1.457, that of water, 1.332, and that of CO_2, 1.195. The higher solubility of CO_2 in oil than in water could bring the oil and water indexes so close as to obscure the phase boundary.

The CO_2-oil miscible zone was short initially, but continued to expand while CO_2 injection was in process. Meanwhile, the crude oil ahead of the miscible zone was increasingly affected by CO_2 as evidenced by its fading color. The miscible front advanced rather uniformly. No severe override was observed at the CO_2 front.

After the miscible front had passed, the pendular connate water re-emerged and the residual oil was found in both rings and clusters. As CO_2 injection was continued, it was observed that the amount of residual oil was decreasing and the color was gradually changed from light yellow to dark brown. This was probably caused by the continuous loss of light hydrocarbons

into the subsequent CO_2 injection. The reduction of residual oil should contribute in part to the oil recovery after CO_2 breakthrough. Photos 3a - 3c illustrate the initial condition before CO_2 flooding, the miscible zone, and the residual oil distribution after CO_2 flooding.

The distribution of residual oil in the flow tube was characterized by high saturation at the injection side and decreasing saturation toward the outlet. This result was in reverse order to water-flooding. From visual observation, it was noticed that the effect of CO_2 was extended beyond the miscible front and grew wider with CO_2 injection. As this occurred, the oil closer to the outlet would have longer time to develop high quality miscibility and consequently, better displacement efficiency.

Miscible displacement was observed for both 1500 psi and 2000 psi runs; however, the oil recovery at 2000 psi was 5% higher than that of 1500 psi. An examination of the photographic record (Photos 4a-b) has revealed that the high pressure runs contain more oil rings and less ganglia type residual oil than those at lower pressures. These results indicate that the higher recovery efficiency at 2000 psi was achieved by reducing large clusters of residual oil in large pores.

SACROC crude oil: The transition zone for the SACROC oil was more clearly visible than Penn-Grade oil because of better color effects. The color in the transition zone changed smoothly from nearly clear in the CO_2 region, to yellow red-brown, and ended with dark brown in the unaffected zone. Photos 5a-f show the transition zone and distribution of residual oil.

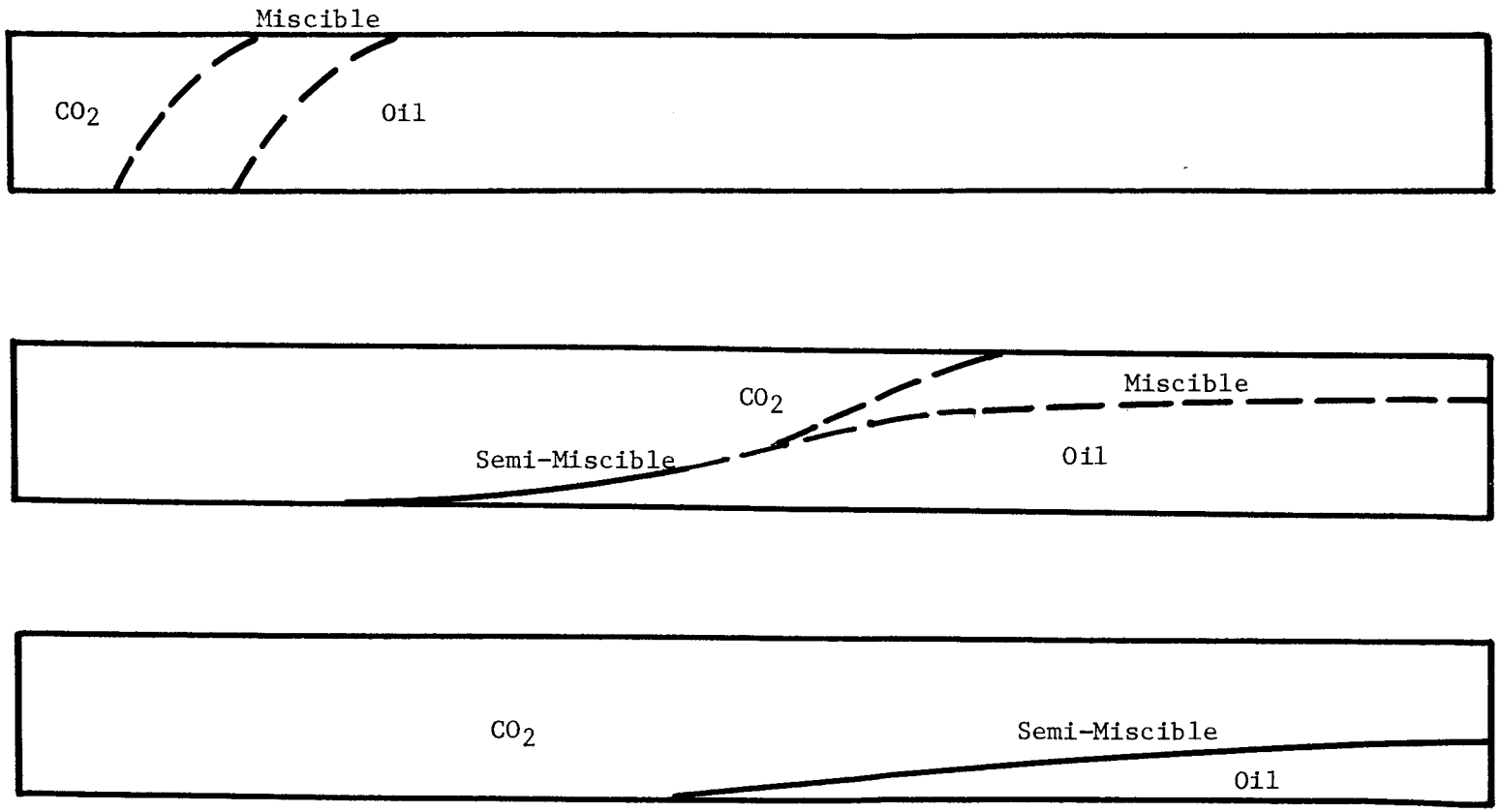
Attention was paid to the possible existance of multiple phase mixtures in the transition zone as reported by other investigators.[8] Our observations showed that there was no clear indication of two-lqiuid or CO_2 gas in the transition zone. Layered liquid was occasionally observed near the CO_2 front. However, this layered liquid was locally developed and would vanish in a short period of time. Photos 6a-b show the layered liquid phenomenon.

The unrecoverable residual oil, as described previously, was in the form of pendular rings and clusters as shown in Photo 4c. In the later stage of CO_2 injection, precipitation of clear to light brownish liquid was observed on the glass beads and windows. The precipitation was not readily soluble in toluene or water. It was probably a wax material extracted from the residual oil which fell out from the CO_2 stream when it struck the solid surface. The physical views of the precipitation are shown in Photos 7a-b.

Due to high permeability of the glass-bead pack, the fluid flow was largely controlled by gravity forces, and therefore severe segregation of miscible mixture and crude oil occurred. The miscible mixture, because of its light weight, tended to segregate and flow at a relatively higher velocity in the transverse direction than in the longitudinal direction and, as a result, an inclined front was formed. The angle of the inclination became more acute as the traveling distance increased. These characteristics are diagrammatically sketched in Fig. 5.

From visual examination, the fore front of the tilted transition zone has the best quality of miscibility and efficient oil displacement. The miscibility was deteriorated toward the rear side of the front line, and in

Figure 5. Sketch of Secondary Miscible CO_2 Displacement



the meantime, the oil displacement was degenerated from miscible type to so-called semi-miscible displacement. In the process, CO_2 was first in contact with oil and went into solution in oil. When solution CO_2 was increased to a certain point, numerous micro-size spherical particles erupted from the oil surface and were carried away in the CO_2 stream. The eruption started with strong turbulence and then gradually settled down until most of the oil was displaced. This type of displacement was usually observed in the area behind the miscible zone. Typical views of the displacement are shown in Fig. 6 and Photos 8a-f.

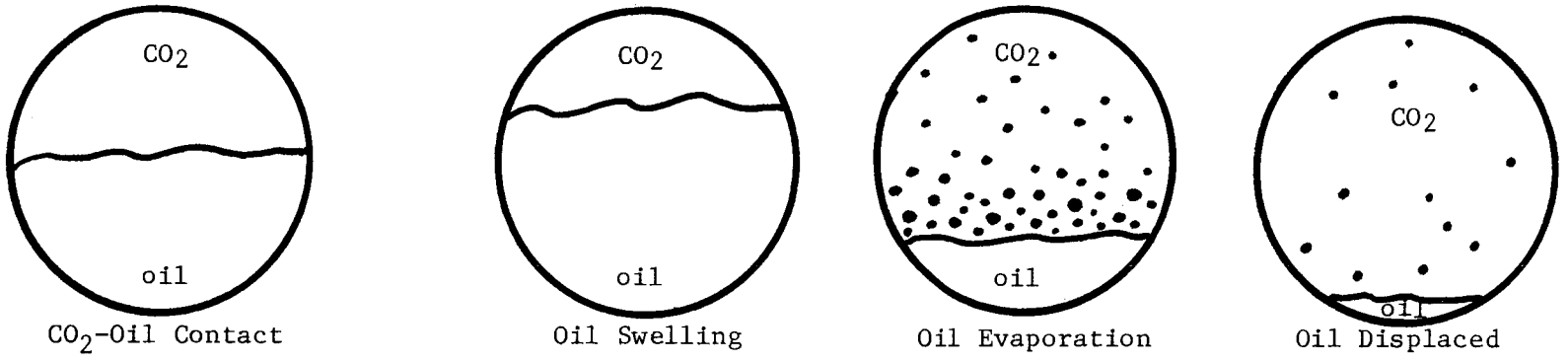
The total oil recovery was improved by 6.4% when injection pressure was increased from 1500 to 2000 psi. The incremental oil recovery, based on observations, was largely due to decrease in CO_2 override.

Slaughter Estate crude oil: The displacement phenomena were similar to SACROC crude oil. The only difference was that a longer distance was required for the development of the miscible zone. Also, as anticipated, CO_2 segregation and override were more severe than with the other two crude oils.

At the injection pressure of 1500 psi (SE-1), miscible zone was not developed until the CO_2 front had traversed 4/5 of the flow tube, thus, only 1/5 of the glass-bead pack was miscibly displaced. The miscible zone formed, however, was overriding the unaffected oil zone and would be quickly displaced by the pursuing CO_2. After that, the displacement would be only of the semi-miscible type. Photos 9a-c illustrate this transformation.

Displacements at higher pressures of 2000 and 2500 psi (SE-2 and SE-3) resulted in an earlier development of miscible zone. Miscible zone

Figure 6. Sketch of Semi-Miscible Displacement



was found in the third window at 2000 psi and in the first window at 2500 psi. Oil recovery, as shown in Table 1, was increased from 50.48% at 1500 psi to 69.18% at 2000 psi and 72.62% at 2500 psi.

2. Secondary Slug CO_2 Flooding

All slug CO_2 displacement tests were conducted at 2000 psi and 120° F. Based on previous straight CO_2 flooding tests in (1), displacement should have occurred at this pressure and temperature condition. CO_2 slugs of 0.1, 0.2, and 0.4 pv were chosen for the tests. The flooding results are summarized in Table 2 and plotted in Figs. 7-9.

(a) Penn-Grade crude oil

From Fig. 7, it is apparent that oil recovery increases with a larger slug size. Slugs larger than 0.4 pv would have no further improvement in total oil recovery.

In the small slug oil pv test, all the CO_2 injected was dissolved in oil or connate water. We found no CO_2 gas phase in the transition zone after CO_2 slug injection was completed. After switching to water injection, the solution CO_2 in the first window dispersed steadily through 1/2 of the flow tube. The displacement was essentially an ordinary waterflooding; however, the oil recovery was improved considerably over that obtained in waterflooding.

Transition zone was observed in the 0.2 and 0.4 pv runs. The color changes and distribution of residual oil were similar to those discussed in the straight CO_2 injection. As soon as water injection was resumed, the pendular connate water was merged with the injection water and vanished from

TABLE 2

Secondary Slug CO_2 Flooding
2000 psi, 120°F

Crude Oil	Run Number	Slug PV	Oil Recovery % OIP
Penn-Grade	PG-4	0.1	50.79
	PG-5	0.2	60.59
	PG-6	0.4	81.45
	PG-3	1.0	81.32
SACROC	SA-4	0.1	58.25
	SA-5	0.2	61.98
	SA-6	0.4	72.36
	SA-3	1.0	82.27
Slaughter Estate	SE-4	0.1	58.00
	SE-5	0.2	68.80
	SE-3	1.0	72.62

Figure 7. Secondary and Tertiary CO_2 Flooding
Penn-Grade Crude Oil
2000 psi, 120°F

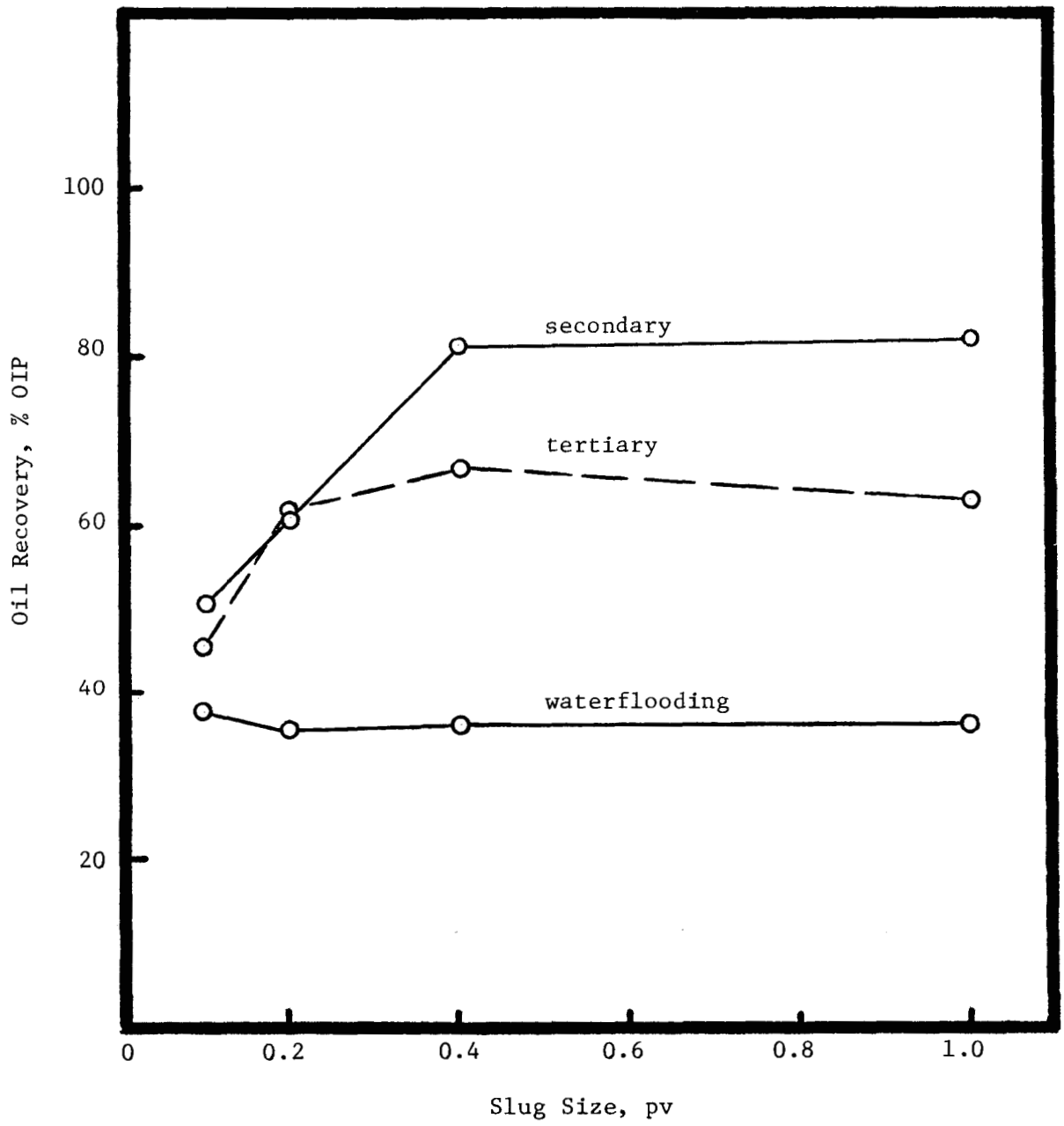


Figure 8. Secondary and Tertiary CO_2 Flooding
SACROC Crude Oil
2000 psi, 120° F

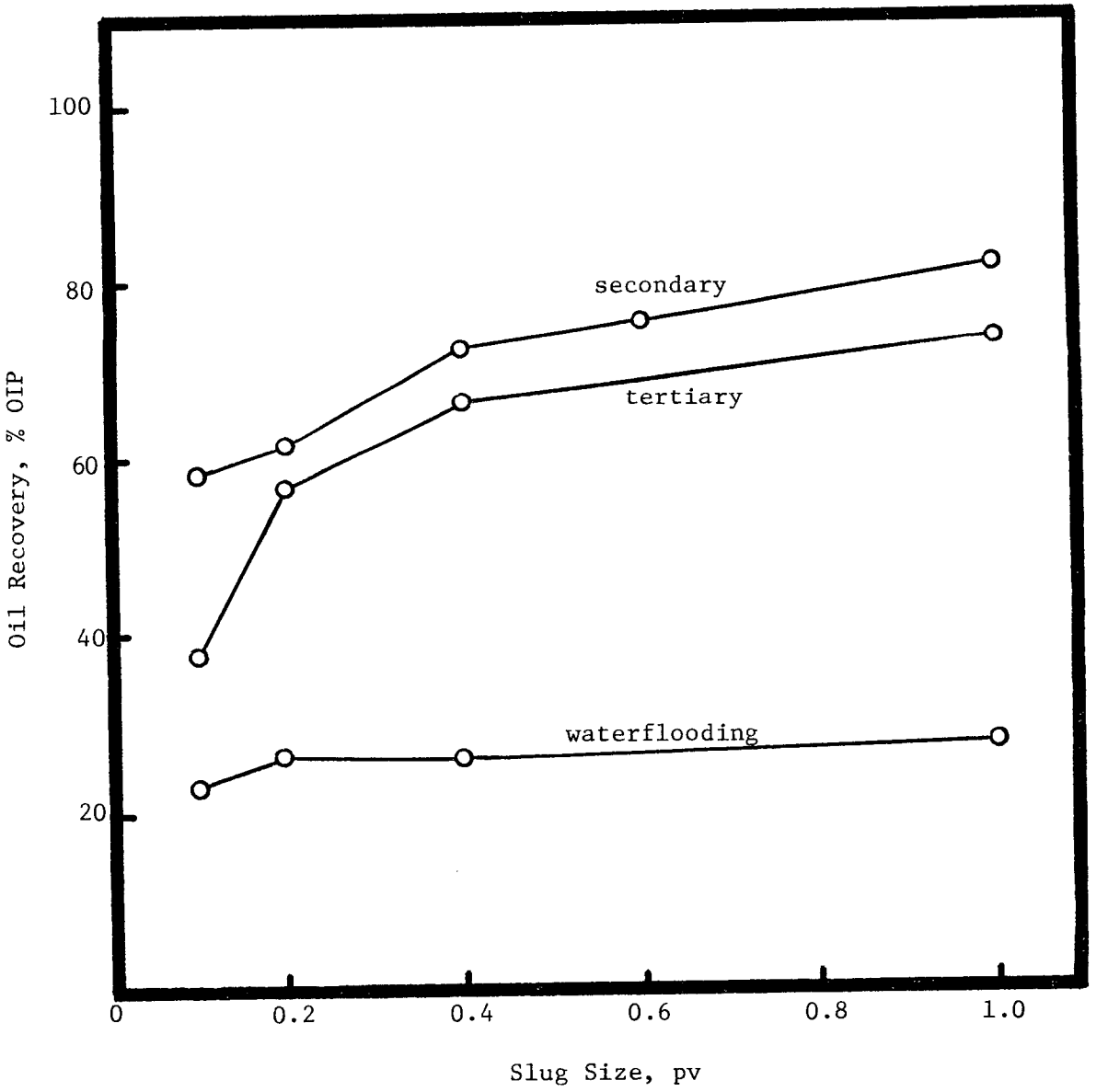
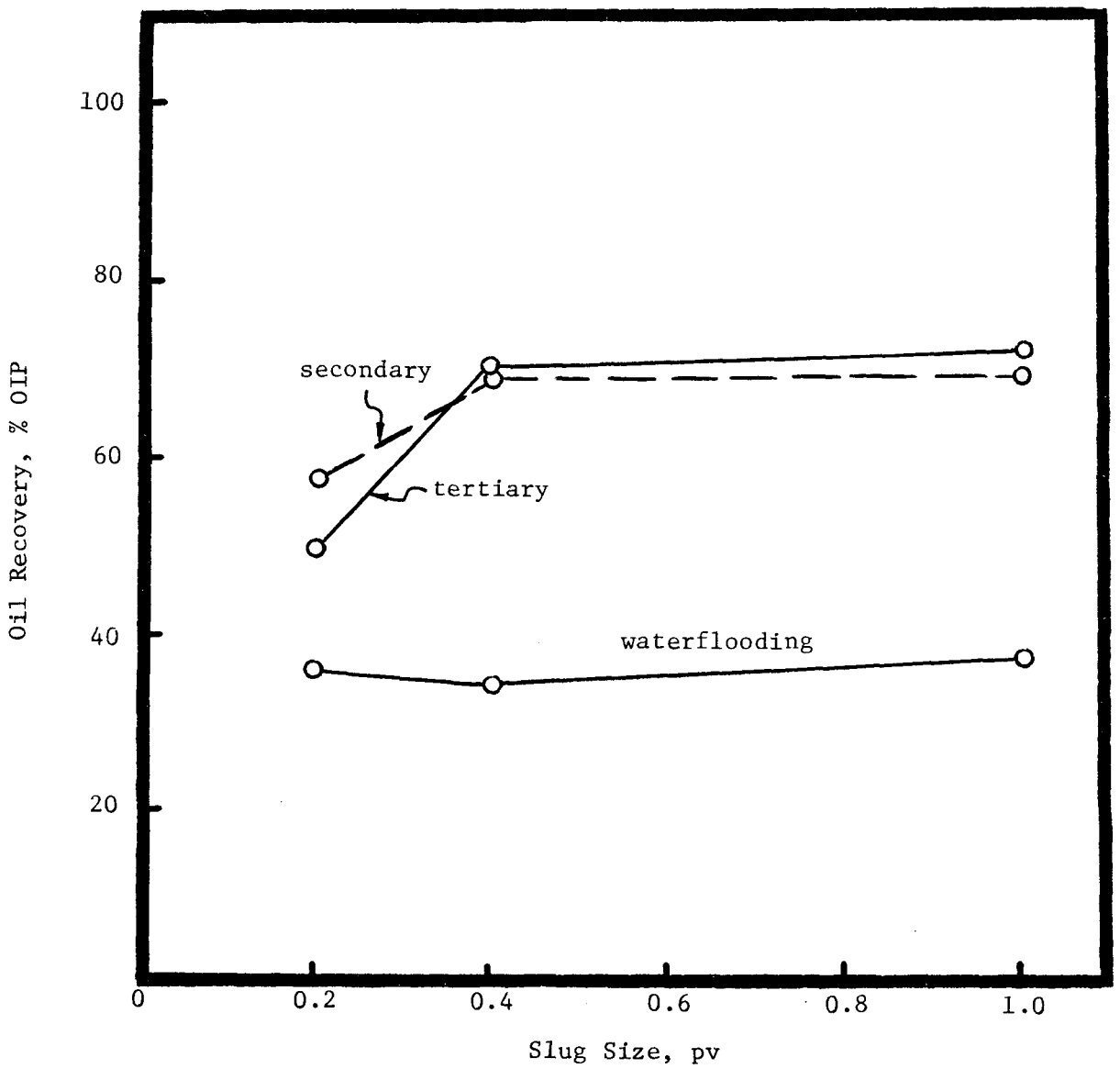


Figure 9. Secondary and Tertiary CO_2 Flooding
Slaughter Estate Crude Oil
2000 psi, 120° F



the scene. Also, the surrounding oil rings were collapsed and regrouped into small clusters or globules lodged in the large pores. In spite of the disturbance, the residual oil units still remained immobile. Photos 10a-b demonstrate the transformation of oil rings into clusters in the water-flooded area.

It is interesting to note that the water production following CO_2 breakthrough changed from clear to turbid. Microscopic examination showed that the water contained brownish emulsion particles. The origins of this substance are now known. It is possible that certain types of miscible mixture of CO_2, oil and water might have been developed under the injection pressure and temperature. When pressure was reduced to atmospheric, the oil phase was separated out in the form of oil-water emulsion.

(b) SACROC crude oil

The displacement results indicate that oil recovery can be improved with increased CO_2 slug size up to 0.4 pv. Slugs larger than 0.4 contribute little in additional oil recovery. Smaller slugs of 0.1 and 0.2 pv would recover less oil than that of 0.4 pv but still significantly more than in straight waterflooding.

In the small slug run (0.1 pv), the injected CO_2 was completely spent in solution. Because there was no CO_2 gas buffle zone between oil and injected water, the displacement was practically a water-oil immiscible type and, as a result, an appreciable amount of CO_2-saturated-oil was bypassed and trapped in the waterflooded zone. Photos 11a-c show this result.

(c) Slaughter Estate crude oil

In the Penn-Grade and SACROC slug CO_2 displacement, the subsequent water injection did not mobilize residual oil left behind the miscible zone. In the Slaughter Estate oil tests, as mentioned earlier, the residual oil in the CO_2 flooded zone was considerably high because of severe gravity segregation. For this reason, the water injection after CO_2 slug showed some effect in displacing residual oil in the CO_2 swept area. A small oil bank was observed at the water front and an increase of oil production was noticed at the breakthrough of the oil bank.

3. Tertiary CO_2 Displacement

In the tertiary displacement tests, the oil saturated glass-bead pack was first waterflooded before CO_2 slug was injected. Four slug sizes, 0.1, 0.2, 0.4, and 1.0 pv were used for the tests. All runs were conducted at 2000 psi and 120°F and the results are summarized in Table 3 and plotted in Figs. 7-9.

(a) Penn-Grade crude oil

Most of the residual oil after waterflooding was scattered in the middle part of the flow tube. The oil was confined in large pores in the form of ganglia or globules. As soon as the CO_2 slug entered the glass-bead pack, the CO_2 gas quickly segregated and began overriding on the top of the flow tube. Later, the horizontal advancement of CO_2 slowed down and moved downward.

During the encroachment of CO_2, some of the residual oil droplets were in contact or surrounded by the invading CO_2. The oil was first decolored and then evaporated in numerous particles into CO_2. This process was continued until the residual oil droplet was completely removed. Photos 12a-c illustrate this process.

TABLE 3

Tertiary Slug CO_2 Flooding
2000 psi, 120°F

Crude Oil	Run Number	Slug, pv	Oil Recovery % OIP
Penn-Grade	PG-7	0.1	7.71
	PG-8	0.2	25.85
	PG-9	0.4	30.71
	PG-10	1.0	30.60
SACROC	SA-7	0.1	14.93
	SA-8	0.2	30.77
	SA-9	0.4	40.15
	SA-10	1.0	45.74
Slaughter Estate	SE-6	0.2	13.50
	SE-7	0.4	35.27
	SE-8	1.0	34.90

Not all the residual oil had the chance to be contacted by CO_2 and eventually displaced. Some of the residual oil was affected by CO_2 by diffusion, but was still encircled by water and remained immobile.

Unlike the SACROC crude oil, there was no oil bank build-up during CO_2 injection, but there were small increases of oil saturation ahead of CO_2 slug. The displacement was therefore primarily a semi-miscible type. The possible reasons for not forming an oil bank are: (1) low residual saturation after waterflooding, and (2) severe override of oil by CO_2 due to density contact relative to water. The fast transport of oil on the top of glass-bead pack lessened the chance to form an oil bank.

The 0.1 pv slug case (SA-7) recovered 7.71% of OIP and 29.36% in blowdown. The relationship of low CO_2 flooding recovery versus high blowdown production was found in nearly all secondary and tertiary displacement tests. The blowdown process was initiated after CO_2 injection had been concluded. The system was bled down to atmosphere in about five pressure steps. Normally, solution CO_2 would come out from the liquids when pressure was brought down to about 800 psi. The strong surge of CO_2 gas would distort the geometrical pattern of residual oil. Most of the ring type oil saturation would be broken up and regrouped in irregular shape. Meanwhile, a part of the residual oil was unlocked and recovered with accompanying CO_2.

(b) SACROC crude oil

Displacement of residual oil in the waterflooded area was clearly demonstrated in the SACROC oil tests. In the 1.0 pv run (SA-10), it was observed that when CO_2 entered into the first section, water was displaced by CO_2 and thus effectuating contact and displacement of residual oil. After

the first section was completely swept by CO_2, the originally high water saturation was reduced to pendular units and the residual oil was transformed into circular rings. The displacement at the early stage was largely semi-miscible.

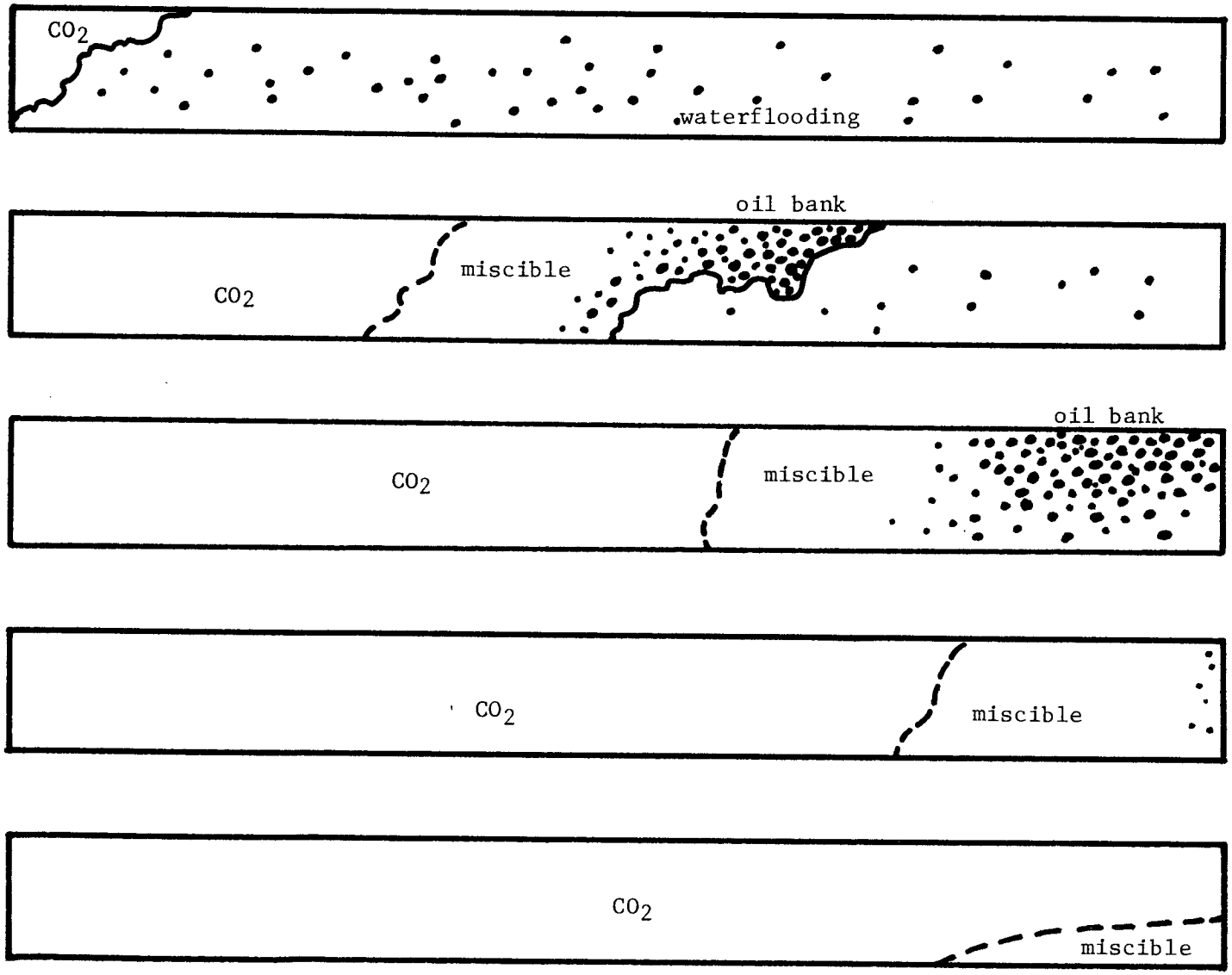
Before the first section was completely swept, an increase of oil saturation was observed in the second and third sections. Later, a solid oil bank and a miscible zone were developed in the third section. The size of both transition zones and oil bank was expanding as travel distance increased. Gravity segregation of the lighter miscible mixture and oil bank was observed but was less severe than that observed in the Penn-Grade oil displacement. Fig. 10 and Photos 13a-c illustrate the procedure in developing miscible zone and oil bank in a waterflooded zone.

In the 0.4 pv slug test, the CO_2 slug was large enough to displace most recoverable oil before water injection was initiated. The water injection, therefore, had little effect on total oil recovery. In the CO_2 flooded area, the water injection would displace CO_2 and break up pendular oil rings.

A small oil bank was formed in the 0.1 slug run (SA-7). Because the slug was so small, there was not enough free CO_2 behind the miscible zone to protect the oil bank. As a result, the oil bank was channelled through by the trailing water, leaving large amounts of unrecoverable oil in the water invaded zone. Photos 14a-c show the water invasion and disintegrated transition zone.

It should be mentioned that oil banks were developed in all runs with different slug sizes. Based on visual observations, it is believed

Figure 10. Sketch of Oil Bank Development in Tertiary CO_2 Flooding



that the oil bank was formed not only by the oil swept out from the flooded zone, but also due to swelling of the residual oil. The swelling factor, defined as the ratio of swelled oil volume at saturation temperature and pressure to oil volume without CO_2, was 1.43 for SACROC crude oil. The large swelling factor would increase the volume of residual oil by 40% thus promoting the formation of oil bank. The effect of swelling on oil recovery would be more critical when residual oil saturation is high, such as in waterflooded heavy oil reservoirs. The swelling characteristics will be further discussed in the Wilmington crude oil tests.

(c) Slaughter Estate crude oil

Residual oil after waterflooding was in large clusters or ganglia. The injection of CO_2 would displace water and reduce the water saturation to pendular form outline by wide-rim oil rings.

Increase of oil saturation was observed ahead of CO_2, but the size of oil accumulation was small compared to SACROC crude oil. Oil displacement was essentially immiscible or semi-miscible type. An overriding miscible mixture was occasionally seen at the front of CO_2. Photos 15a-c demonstrate displacement of residual oil after waterflooding.

4. Temperature Effect on Oil Recovery

The miscible pressure is affected by oil composition and reservoir temperature. Yelling and Metcalf[4] have found that the CO_2 minimum miscible pressure would be increased by approximately 15 psi/^{o}F over a temperature range from 95 to 192°F.

In this report, the temperature effect on recovery was studied at 75° and 120°F for three crude oil samples. The results are shown in Table 4.

TABLE 4

Temperature Effect on CO_2 Displacement

Crude Oil	Run No.	Temperature °F	Pressure psi	Oil Recovery Breakthrough % OIP	Oil Recovery Total % OIP	Displacement Type
Penn-Grade	PG-11	75	1500	39.01	(71.04)*	M
	PG-2	120	1500	41.33	76.34	M
SACROC	SA-11	75	1000	37.78	70.91	M
	SA-1	120	1000	32.84	55.55	I
Slaughter Estate	SE-9	72	1500	33.58	64.07	I-M
	SE-1	120	1500	30.65	66.83	I-M

* Oil Recovery did not approach maximum

M = Miscible

I = Immiscible

For the Penn-Grade crude, CO_2 injection was conducted at 1500 psi. Visual observations indicate that the displacement at 75° and 120°F were both miscible. The oil recoveries at CO_2 breakthrough were almost equal. The oil recovery for the 75°F run did not approach its maximum and thus it should not be used for comparison with 120°F run.

The displacement tests for the SACROC oil were performed at 1000 psi. The 120°F run which was discussed earlier in this report, was immiscible. At the same pressure, but at the lower temperature of 75°F, the displacement became miscible in all sections. The total oil recovery was 70.91% at 75°F compared to 55.55% at 120°F.

The recoveries for Slaughter Estate crude oil at 72° and 120°F are fairly close and the displacement mechanisms are similar. The displacement started with immiscible displacement in the first and second window and upgraded to semi-miscible in the third window and finally to miscible on the upper part of the fourth and fifth windows. Severe CO_2 override was observed in both runs.

It is apparent from above discussions that a lower temperature would reduce CO_2 miscible pressure. On the other hand, temperature would have little effect on total oil recovery should the displacement have been in miscible state.

D. Down-Dip Displacement Test

1. Procedure

To facilitate down-dip CO_2 displacement (up-dip injection), a steel skid was constructed to support the flooding assembly consisting of

transparent flow tube, constant temperature bath, and control manifold. One end of the skid was lifted by means of a chain block anchored on the ceiling. Inclination of the skid can be adjusted by operating the chain block. A portable lift jack was used to support the microscopic camera so that photographs can be taken at different positions alongside the inclined flow tube. The set-up of the rearranged system is shown in Photos 16a-b.

Slaughter Estate crude oil was selected for the test because the high viscosity of the crude would show more clearly the effect of inclination on CO_2 displacement efficiency. Angles of inclination were set at 30°, 15°, and 0° (horizontal). Higher angles more than 30° were not performed because we found that the gravitation override of CO_2 was completely eliminated at an angle of 30° and the oil recovery was maximized up to 92% OIP at 2000 psi and 120^oF.

All the displacements conducted were secondary recovery. Tertiary recovery was found to be difficult to perform because when water injection was stopped, the residual oil was apt to migrate up-dip and accumulate at one end. This situation could not allow for an accurate evaluation and, therefore, the originally planned tertiary program was not carried out.

2. Results of Displacement and Microscopic Investigation

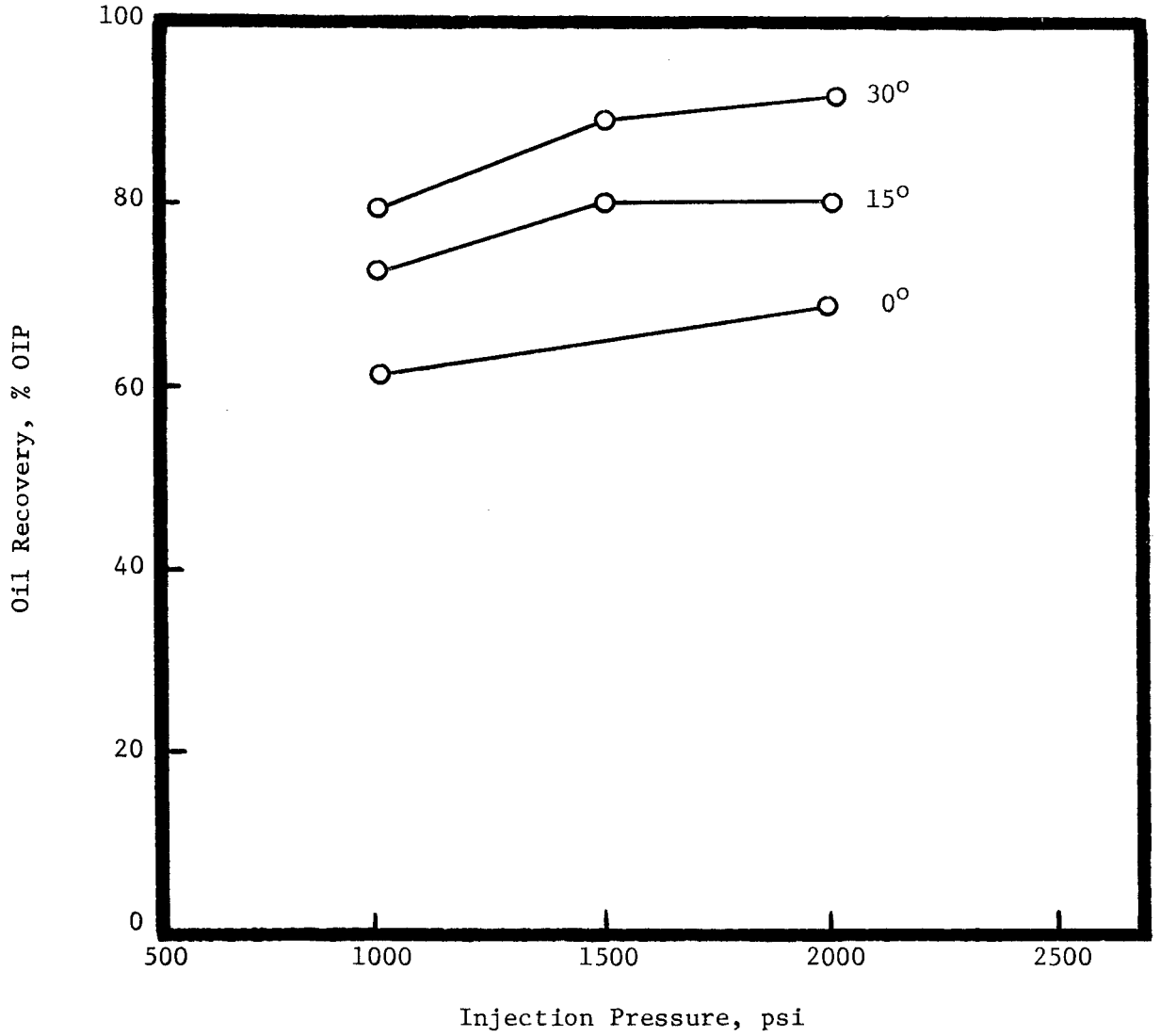
The results of the CO_2 displacement as summarized in Table 5 and plotted in Figure 11 clearly indicate that oil recovery is significantly affected by dip angles. Approximately 30% increase of oil recovery was obtained when changing horizontal flooding to 30° down-dip displacement. This effect was noticed at all pressures ranging from 1000 to 2000 psi.

TABLE 5

Results of Down-Dip Displacement
Wilmington Crude Oil, 120°F

Run No.	Pressure psi	Dipping Angle	Oil Recovery, % OIP
SE-9	1000	0°	60.58
SE-10	1000	15°	72.72
SE-11	1000	30°	79.99
SE-1	1500	0°	66.83
SE-12	1500	15°	80.78
SE-13	1500	30°	89.78
SE-2	2000	0°	69.18
SE-14	2000	15°	80.31
SE-15	2000	30°	91.99

Figure 11. Oil Recovery by Down-Dip Displacement
Temperature 120°F
Slaughter Estate Crude Oil



From microscopic observation, it was found that the increased oil recovery by down-dip displacement was achieved from improvement of CO_2 override. In the 2000 psi and 30° flooding, miscible front was formed quickly in the first section of the flow tube. No CO_2 overriding or gravity channelling was observed throughout the entire flooding sections. The miscible interval was narrow when it was initially formed and widened as traveling distance increased. This performance was in sharp contrast with that of horizontal displacement (SE-2) in which severe override and late miscible zone development resulted.

The CO_2 swept area was relatively clean. The residual oil was in the form of clearly defined rings. Ganglia and cluster-type residual oil as observed in the horizontal displacement was not found in this case. Photos 17a-c illustrate miscible zone and oil rings in the swept area.

Microscopic observation (40x) also revealed that the miscible transition zone contained CO_2 and numerous dust-like oil particles. The population of oil particles were gradually decreased toward the displacing CO_2 phase but increased rapidly toward the oil bank side. Immediately next to the transition zone, the oil has a deep red color and changed to black in the unaffected zone.

Photos 18a-c and Photos 19a-c illustrate displacement sequence similar to Photos 17a-c, but at lower pressures of 1500 psi and 1000 psi, respectively. It can be seen in Photo 18b that the mixing zone is not perfectly homogeneous and the residual oil appears to be high. In the 1000 psi run, the CO_2 and crude oil are immiscible. Photos 19b and 19c show CO_2-oil phase boundaries and large clusters of residual oil. Comparing Photos

17, 18, and 19 one can see the differences of pressure effect on miscibility and oil recovery at conditions without gravity segregation.

At 15° down-dip displacements, CO_2 override was observed at all pressures. Generally, the development of miscible zone was delayed compared to 30° floodings. The displacement was miscible at the forefront and semi-miscible at the trailing front. Photos 20a-c illustrate CO_2 override at 15° down-dip, 2000 psi displacement. Photo 20a shows the receding CO_2 due to oil swelling. Photo 20c indicates high residual oil saturation compared to the 30° case in Photo 17c.

E. CO_2 Foam Displacement

Microscopic study of CO_2 foam flooding was conducted in cooperation with Dr. John T. Patton of New Mexico State University. The purpose of the study was to examine visually the mechanism of CO_2-foam displacement and the stability of the CO_2 foam in porous media.

The crude oil used for the test was from the Tar Zone in the Wilmington Field in California provided by the Champlin Petroleum Company. The crude oil is an asphaltic type containing a small amount of gasoline and large amounts of high molecular-weight cyclic compounds. The oil has a gravity of 15.6^{o} API and a viscosity of 27 cp at reservoir temperatures of $120^{o}F$. The field is presently being waterflooded.

1. Procedure

Two tests were conducted in order to determine the effectiveness of CO_2-foam flooding. The first test was a straight CO_2 tertiary displacement without using foaming agent. In the test, the glass-bead pack was first saturated with Wilmington oil, then thoroughly waterflooded before CO_2 injection was initiated.

The flooding pack for the second test was prepared in similar ways except that the salt water used in waterflooding was treated with 10% by weight surfactant foaming agent provided by New Mexico State University. The basic concept of the CO_2-foam process is to generate foam spontaneously when injected CO_2 is mixed with formation water containing foaming agent.

2. Results and Microscopic Observation

(a) Run Foam 1 (without foaming agent)

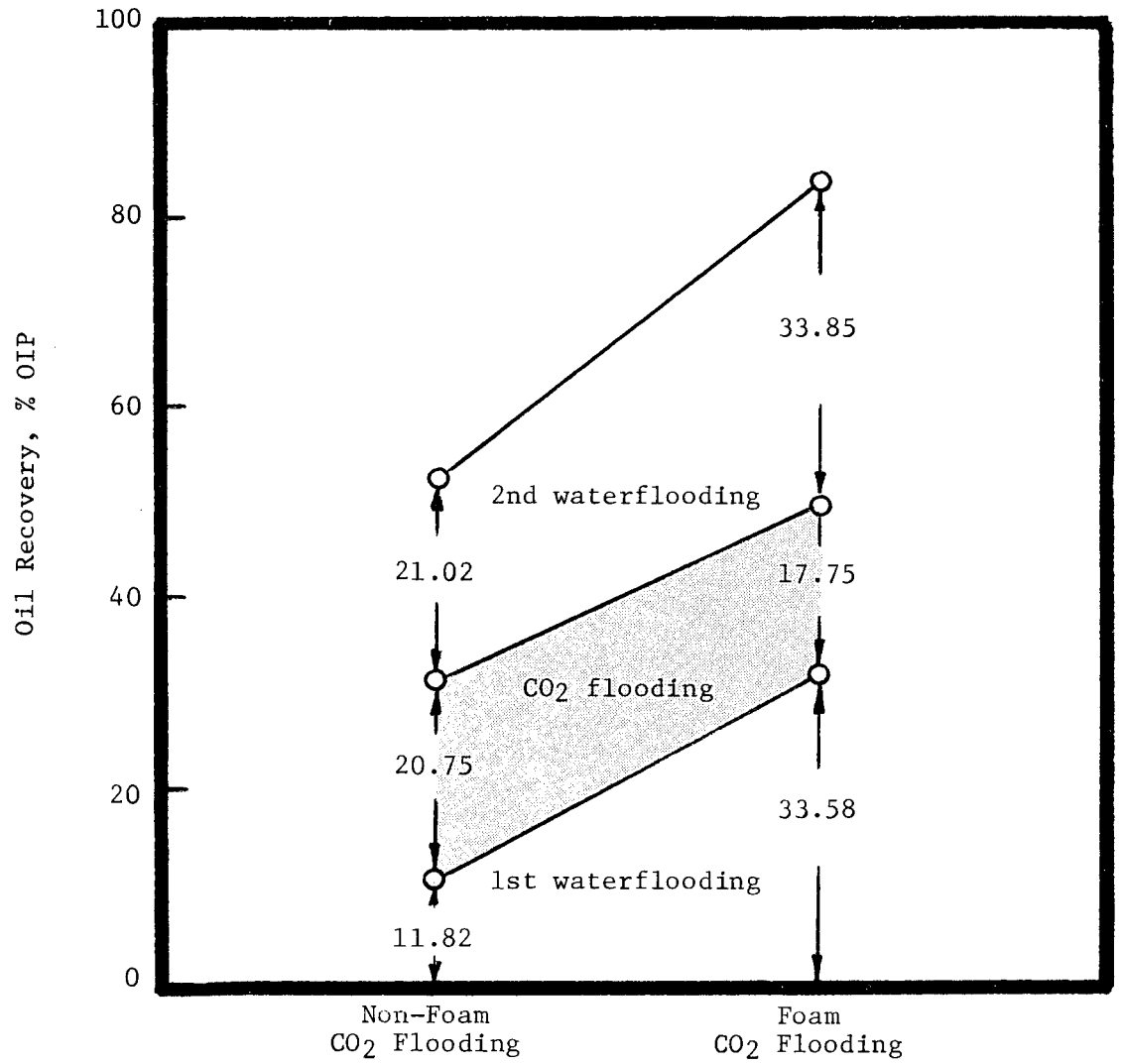
Shortly after CO_2 injection was started, the oil saturation increased drastically and, in the meantime, water was expelled from the waterflooded area. The increase of residual oil saturation was expected because of swelling characteristics of oil-CO_2 mixture; however, the magnitude of swelling was spectacular.

Despite the increase of residual oil saturation, displacement of oil was ineffective due to CO_2 override. The oil recovery was 20.75% of OIP as shown in Fig. 12.

In order to maximize the oil recovery, a second waterflooding was conducted following CO_2 injection. As a result, an additional 21.02% of OIP was recovered. It is interesting to note that the oil recovery from the second waterflooding was higher than the first. The reduced oil viscosity after CO_2 injection was probably creditable to the improved waterflooding result.

Photo 21a shows the oil and water saturations after the first waterflooding. Photo 21b illustrates CO_2 overriding and swelling residual oil. Photo 21c shows the reduced oil saturation after the second waterflooding.

Figure 12. Oil Recovery by Foam-CO_2 Displacement
Temperature 120° F
Pressure 1200 PSI
Wilmington Tar Zone Crude Oil, 15.6° API



(b) Run Foam 2 (with foaming agent)

High oil recovery up to 33.58% of OIP was obtained from the initial waterflooding. The surfactant additives were apparently responsible for the high recovery. Oil recovery from CO_2 injection was 17.75% of OIP compared to 20.75% in Foam 1. The poor CO_2 flooding performance can be explained by two facts: (1) no foam was generated because the water containing foam agent was mostly expelled as a result of oil expansion, and (2) the CO_2 was overriding and was therefore prevented from reacting with the underlaying water (see Photo 22a).

Although it failed from a standpoint of CO_2 foam flooding, the second waterflooding (water containing surfactant) recovered as much as 33.85% of OIP. The large recovery apparently resulted from oil swelling and reduction of surface tension. The residual oil, as shown in Photo 22b, was dispersed probably due to surfactant effect.

V. CONCLUSIONS

Based on visual observation and results of microphotographic study, the following conclusions may be drawn:

1. Three types of CO_2 displacement of oil have been observed, namely, (a) immiscible, (b) semi-miscible, and (c) miscible. Types (a) and (c) have been well defined in the literature. Displacement of Type (b) relies on dispersion of micro-size oil particles into the CO_2 phase. More than one type of displacement could exist simultaneously in a flooding system. Increase of pressure could enhance miscibility and reduce semi-miscible displacement.
2. A part of residual oil in the CO_2 swept zone can be recovered by extraction of the subsequent CO_2 injection.
3. For the flow model used in this study, severe gravity override of CO_2 or CO_2-oil mixture occurred in both immiscible and miscible displacements. Improvements on override could be achieved with increased injection pressure.
4. From microscopic observations, the transition zone consists of numerous moving oil particles in the CO_2 gas stream. The number of oil particles increases rapidly from the displacing CO_2 side to the miscible mixture on the oil bank side. The size of transition zone expands proportionally to the traveling distance.
5. There exists an optimum CO_2 slug size. A slug too large would be wasteful or possibly cause early CO_2 breakthrough. On the

other hand, a slug too small would cause water channelling through the miscible zone and downgrade the displacement to an ordinary waterflooding.

6. A small CO_2 slug (0.1 pv for the experiment conducted) would improve oil recovery substantially compared to a straight waterflooding. Also, a larger blowdown oil recovery would normally be obtained for a small CO_2 slug.

7. There are two types of tertiary recovery processes being observed:

   (a) Semi-miscible displacement: The injected CO_2 would first displace water and then contact with the immobile residual oil. After exchanging components of each other, the residual oil would be evaporated in dust-like particles and carried away in the CO_2 stream. No oil bank is formed in these processes.

   (b) Miscible displacement: In this process, a solid oil bank would be formed and displaced miscibly by CO_2. Reservoirs with high residual oil saturation and/or high swelling factor are likely to have this type of displacement.

8. Generally lower temperatures would promote CO_2 miscibility; however, if pressures are well above miscible condition, temperature would have little effect on oil recovery.

9. Oil recovery can be increased considerably by down-dip CO_2 displacement. The increase is derived by reducing CO_2 override

and improvement in displacing efficiency.

10. In a waterflooded heavy oil reservoir, the characteristics of oil swelling and viscosity reduction would create favorable conditions for waterflooding or CO_2 stimulation.

11. In-situ or spontaneous CO_2-foam process may not be effective should excessive oil swelling take place.

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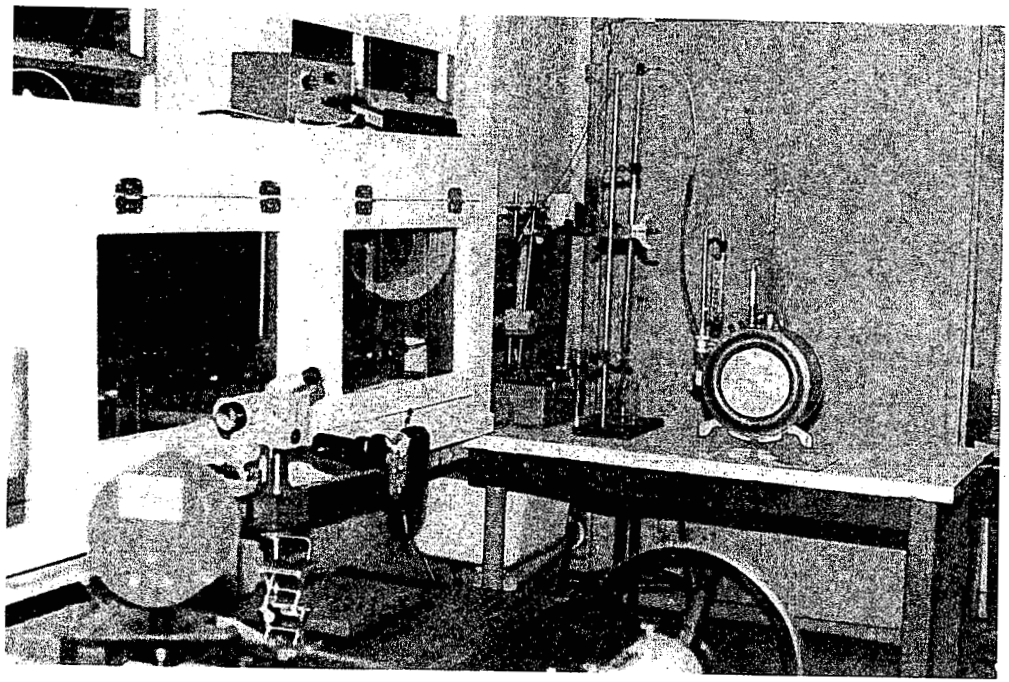
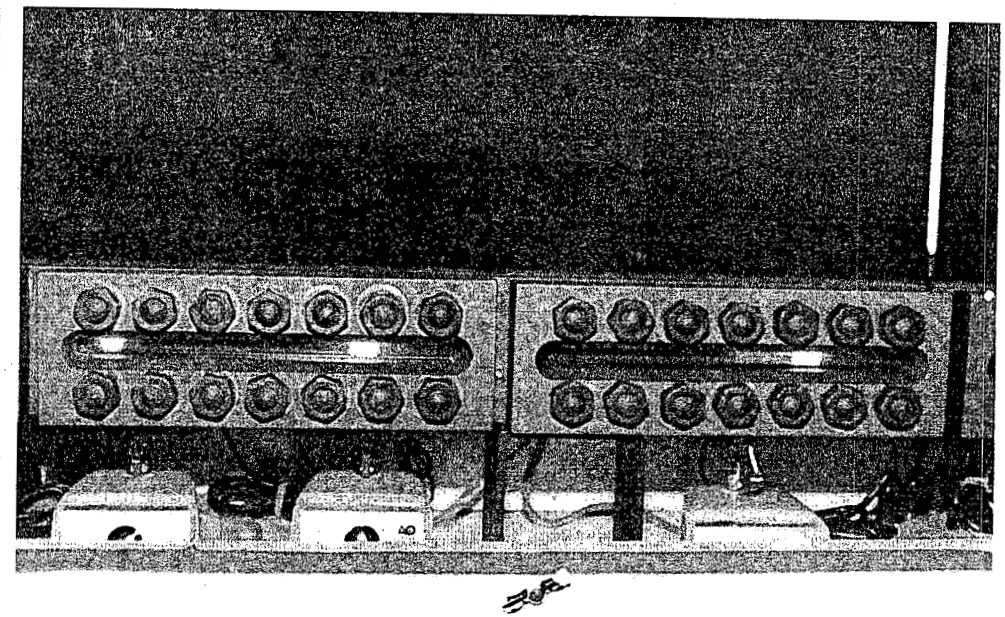
Photo 1. High Pressure Transparent Flow Model

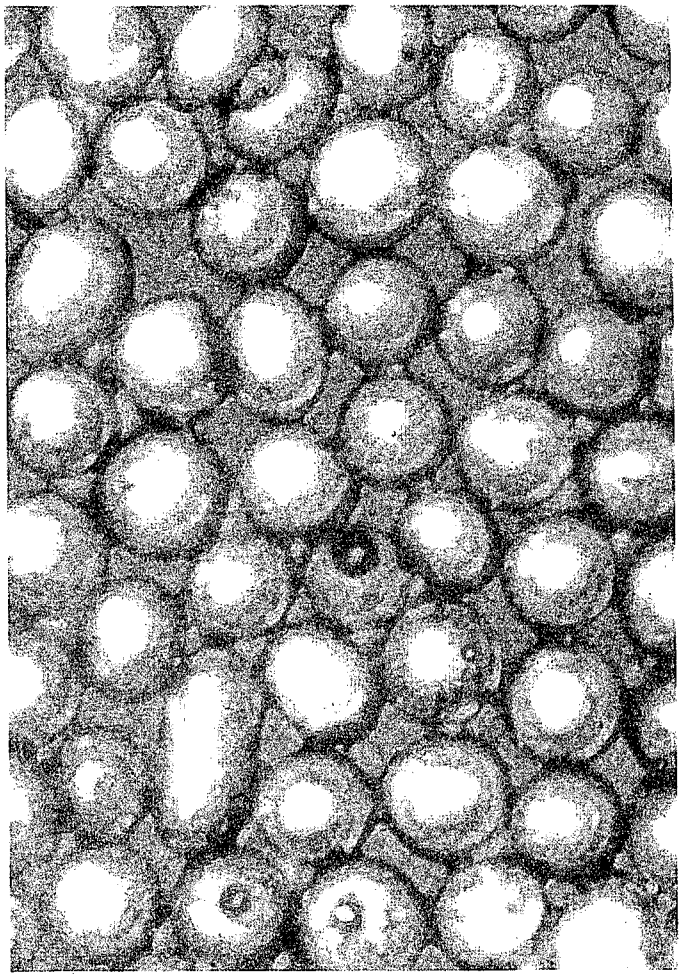


1-a
CO_2 Flooding System

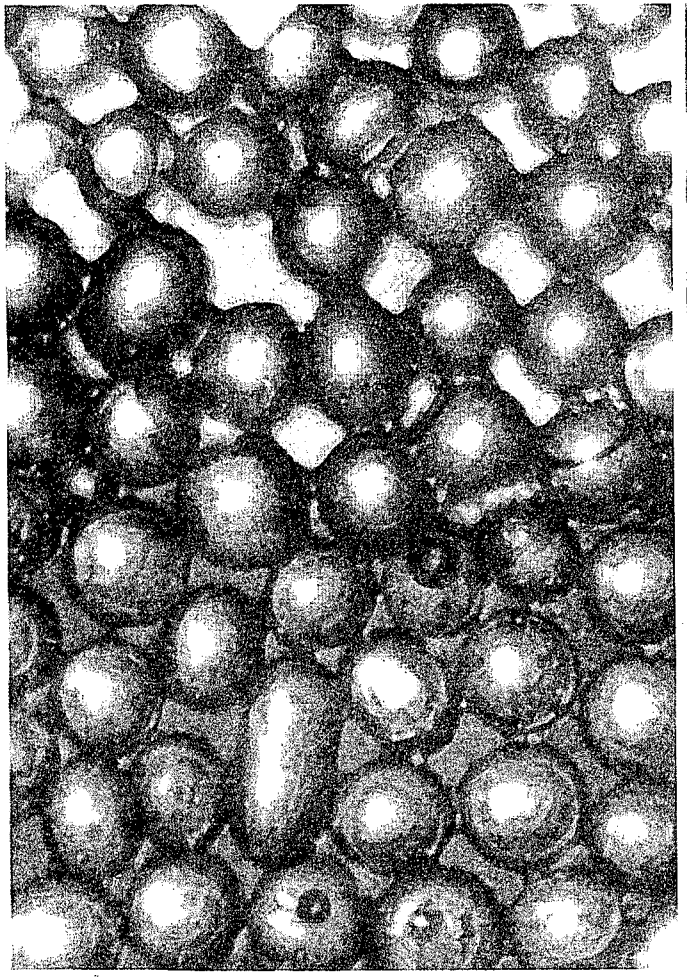


1-b
Transparent Flow Tube

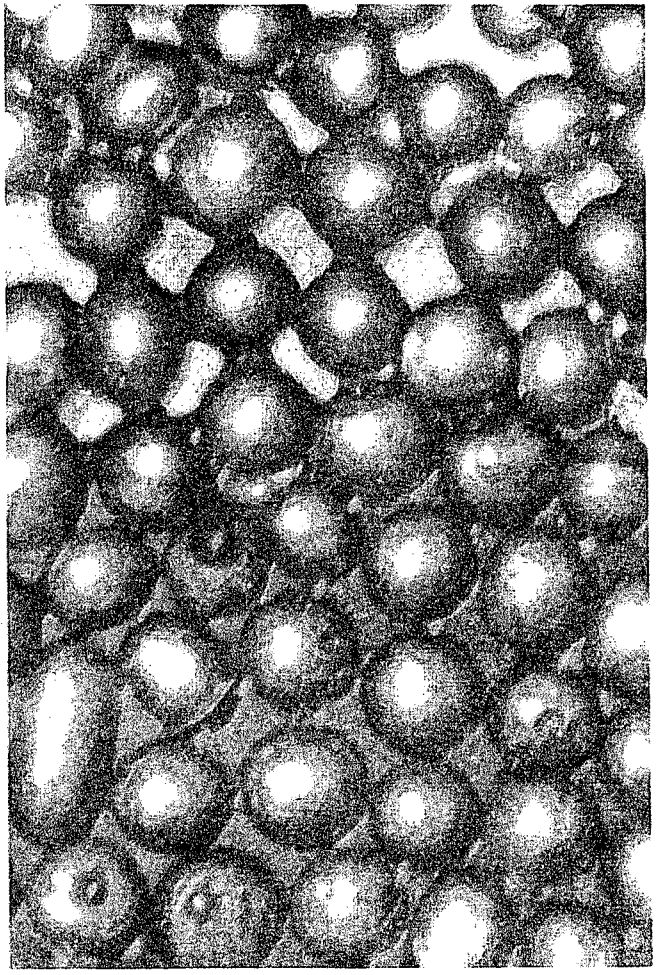
Photo 2. Immiscible CO_2 Displacement



2-a
20x

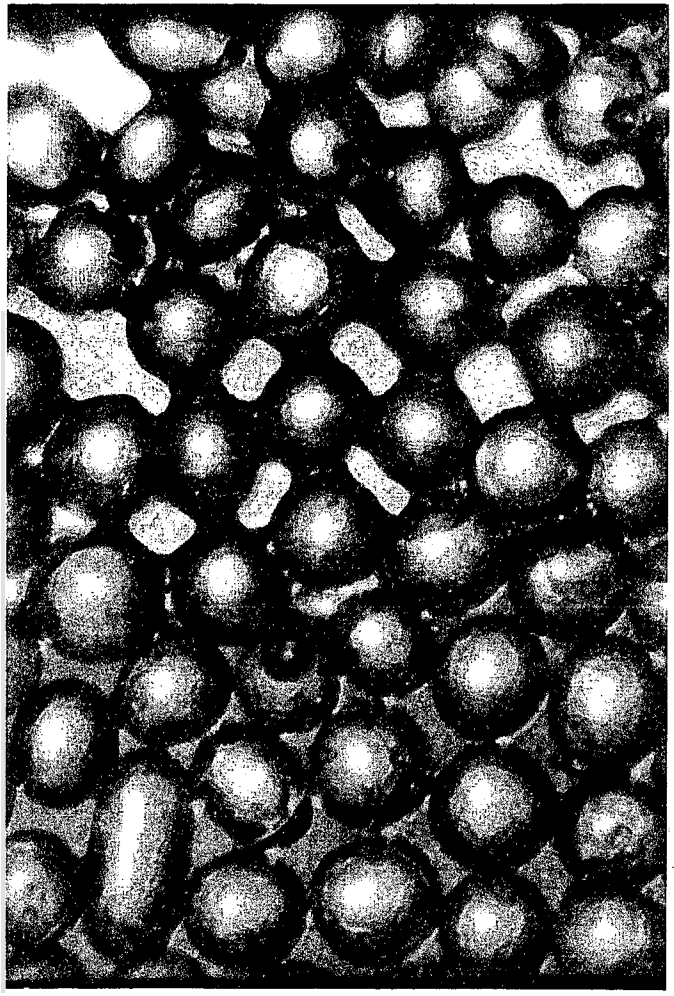


2-b
20x

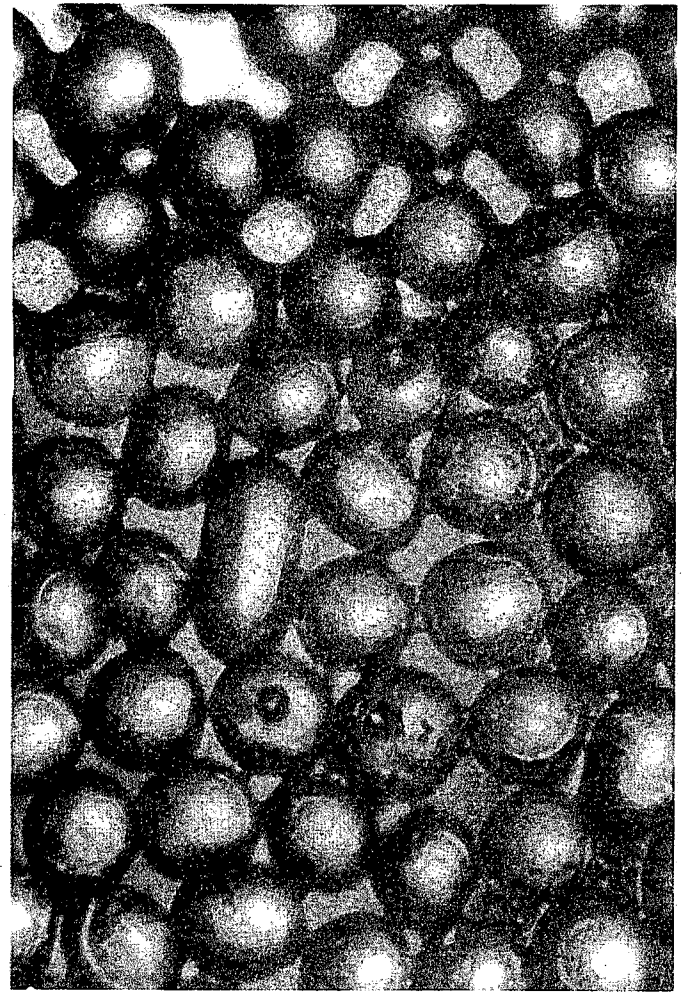


2-c
20x

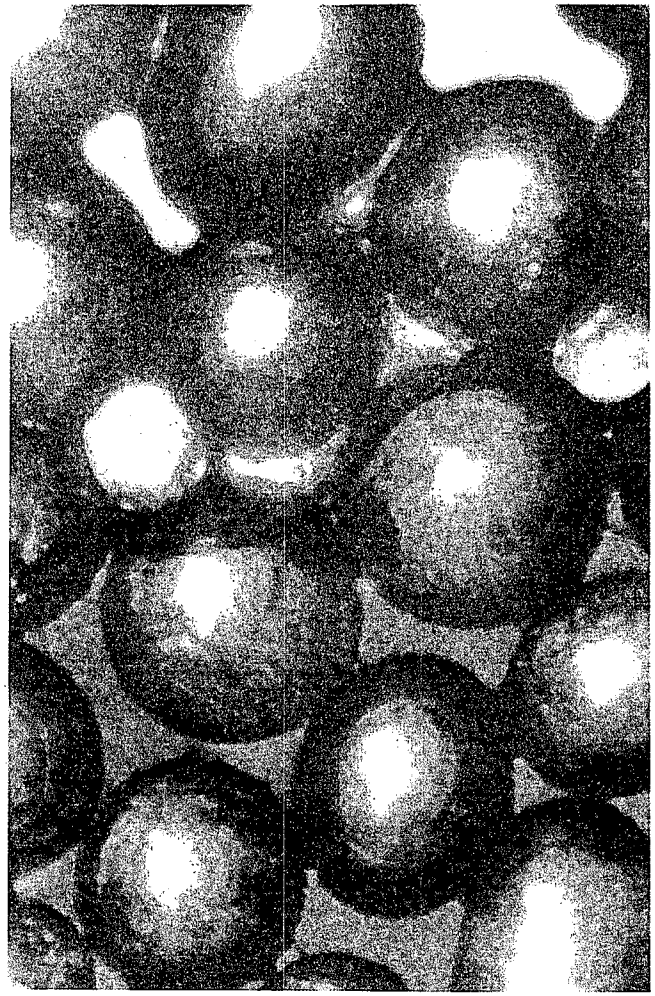
Photo 2. (continued)



2-d
20x

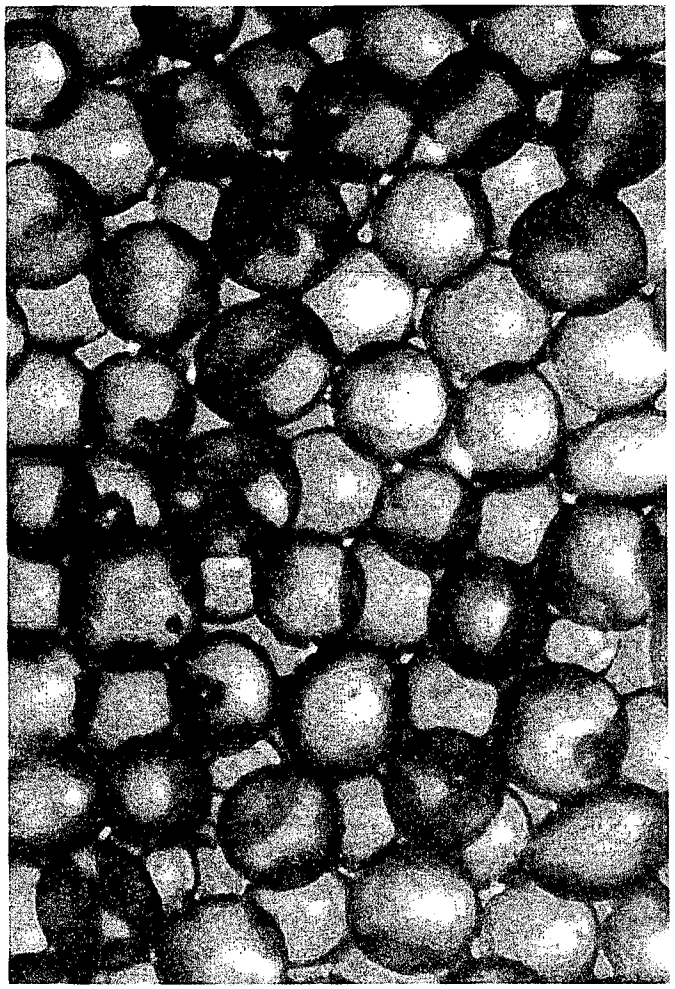


2-e
20x

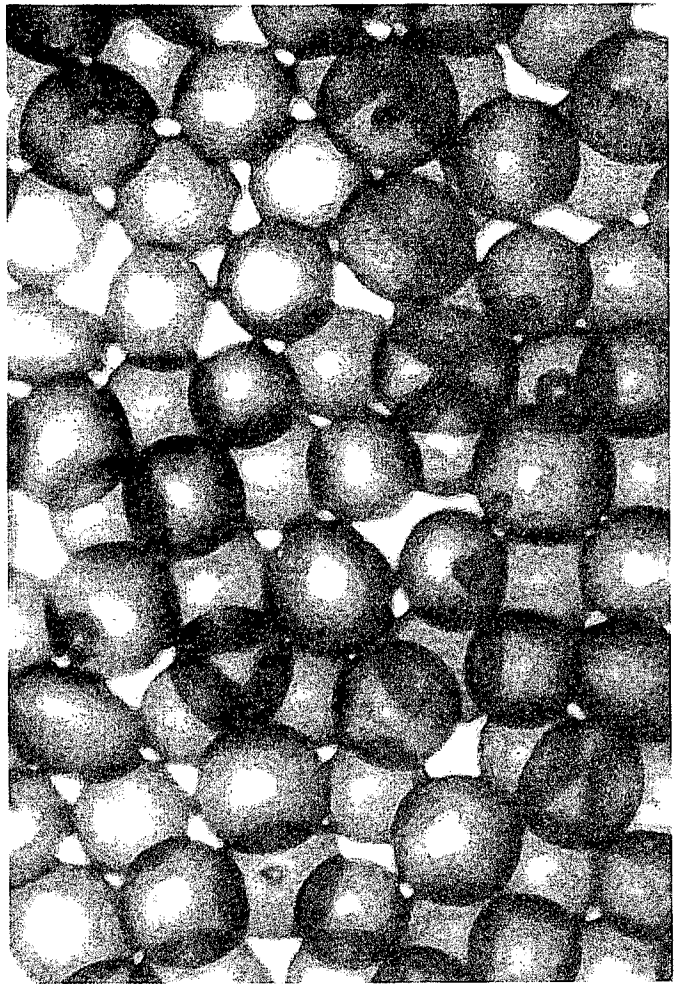


2-f
40x

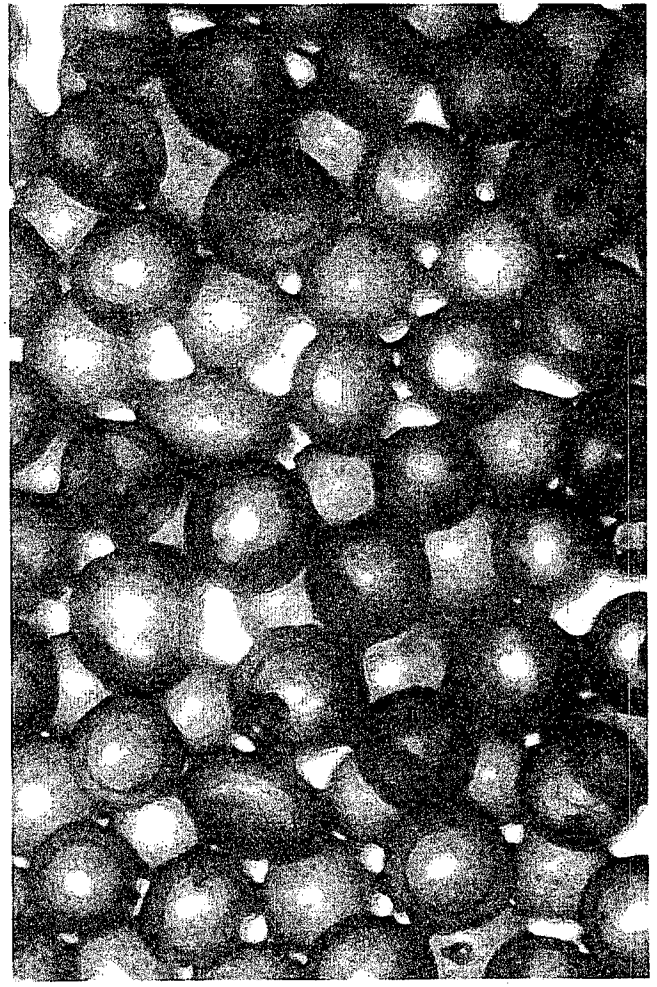
Photo 3. Miscible CO_2 Displacement



3-a
20x
Original

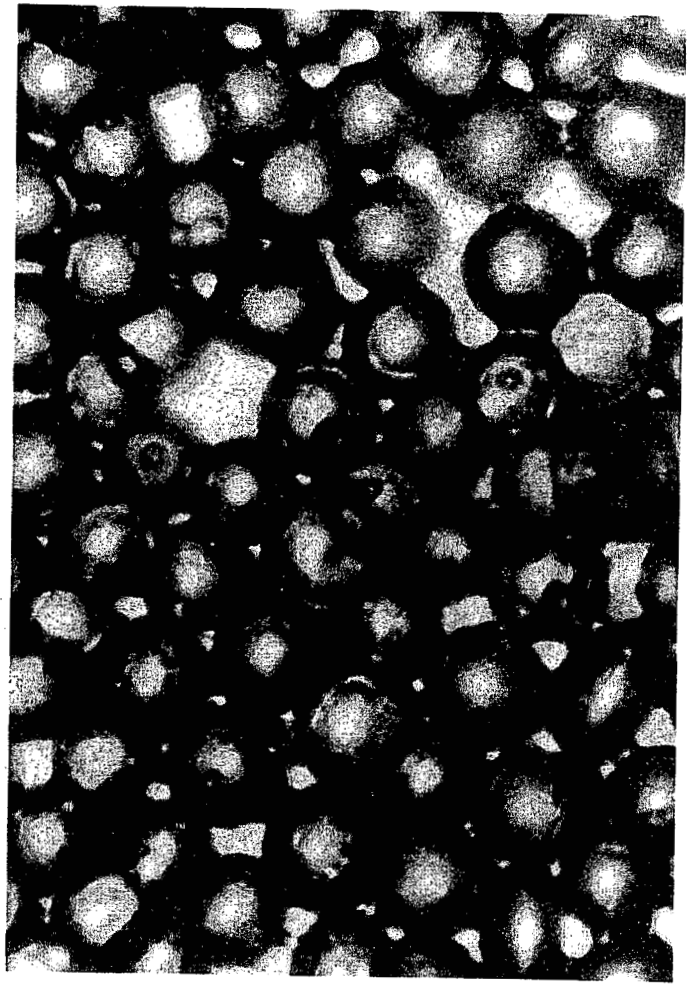


3-b
20x
Miscible Zone

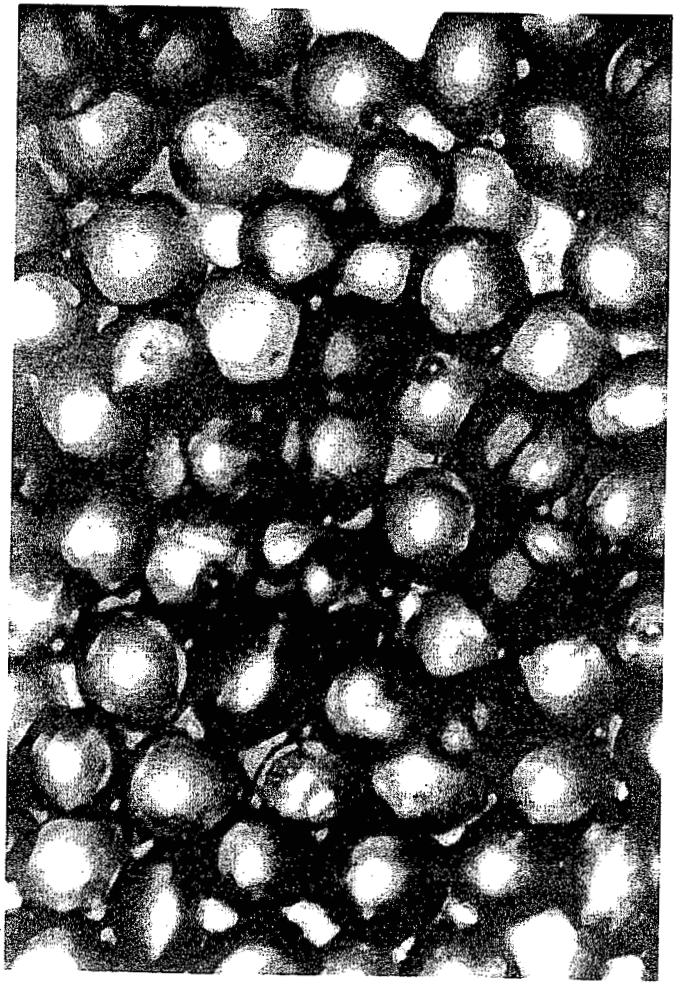


3-c
20x
Residual Oil

Photo 4. Residual Oil After CO_2 Flooding
SACROC Crude Oil

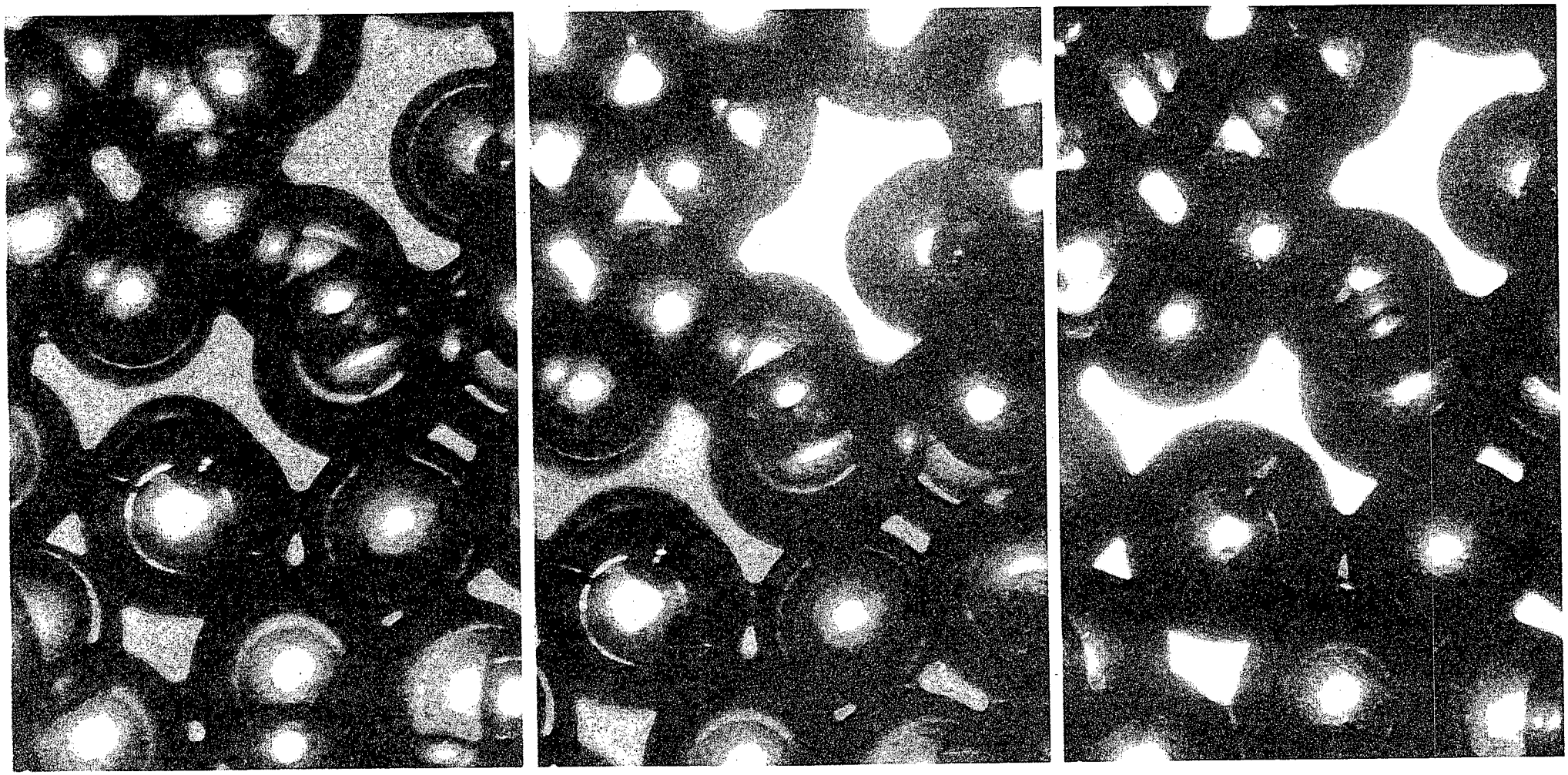


4-a
20x
2000 psi
Most Oil Rings



4-b
20x
1500 psi
Oil Clusters Increased

Photo 5. Miscible Zone and Residual Oil After CO_2 Flooding
SACROC Crude Oil

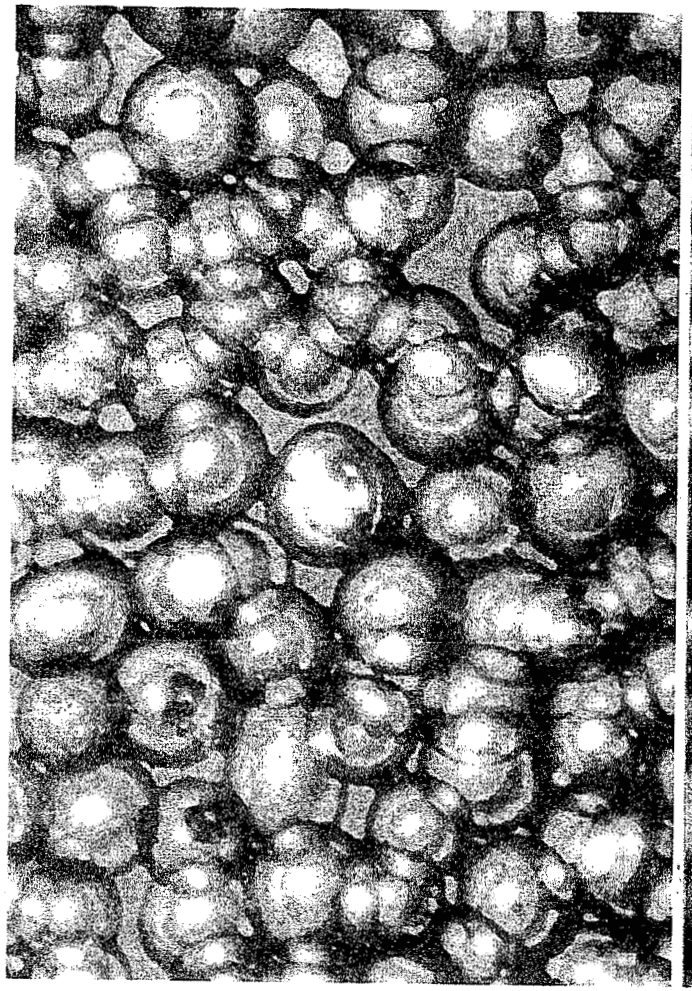


5-a
40x
Original

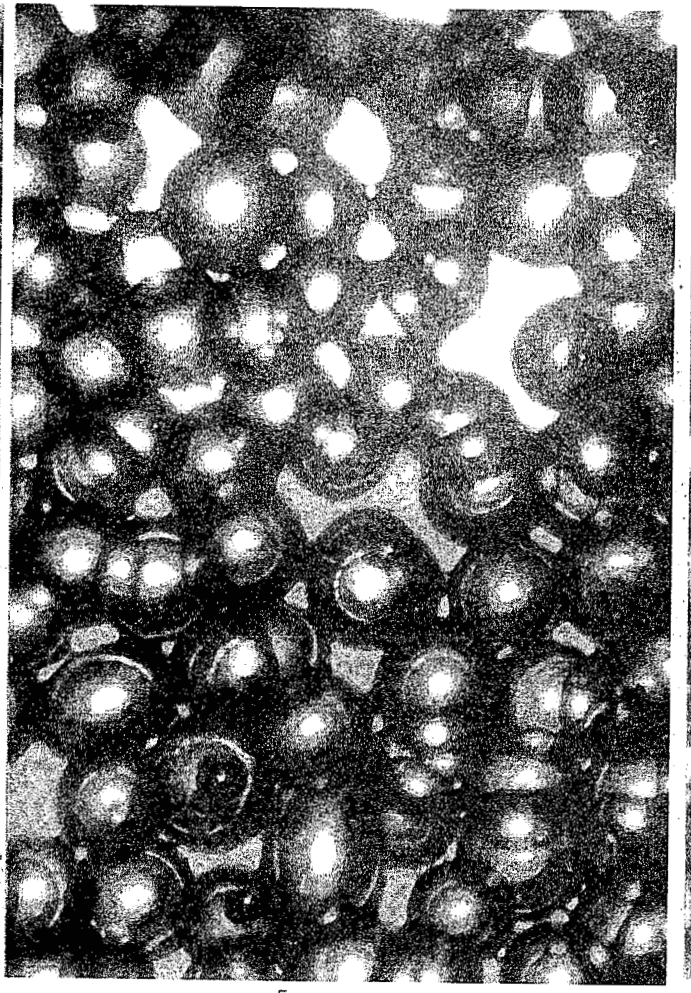
5-b
40x
Miscible Zone

5-c
40x
Residual Oil

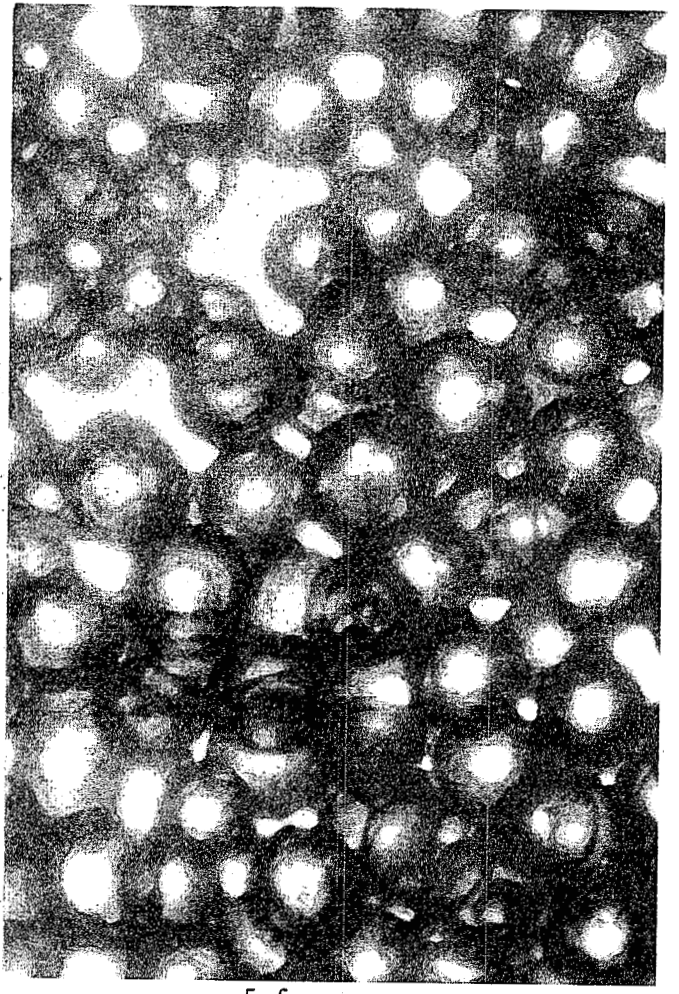
Photo 5. (continued)



5-d
20x
Original



5-e
20x
Miscible Zone



5-f
20x
Residual Oil

Photo 6. Layered Oil-CO_2 Interface
SACROC Crude Oil

6-a
40x
Layered Oil Formed

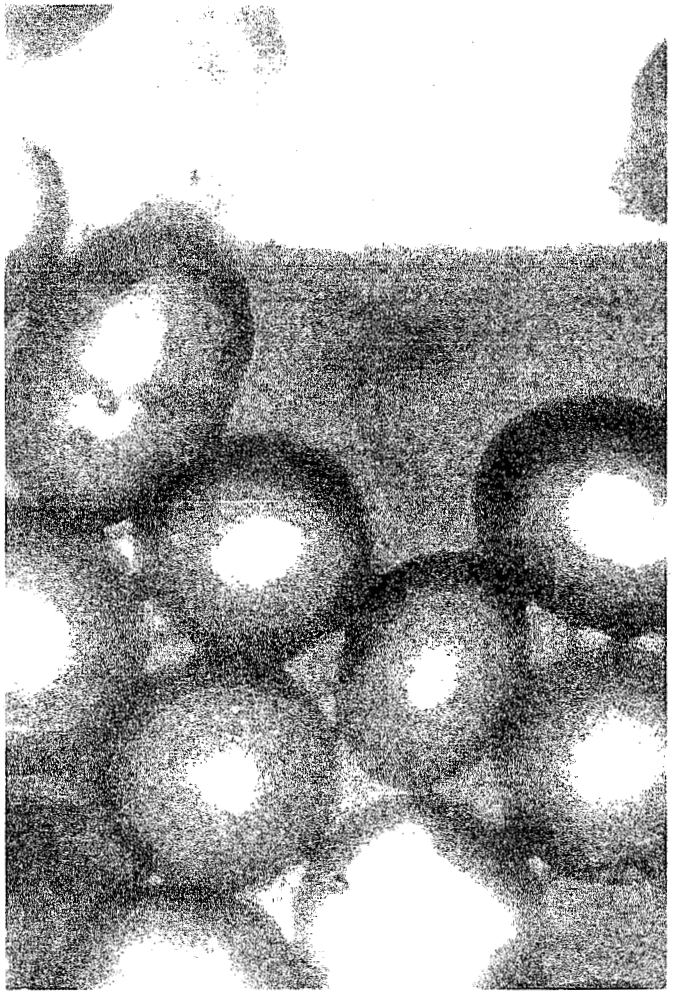
6-b
40x
Layered Oil Dispensed

Photo 7. Waxy Depositions
Slaughter Estate Crude Oil

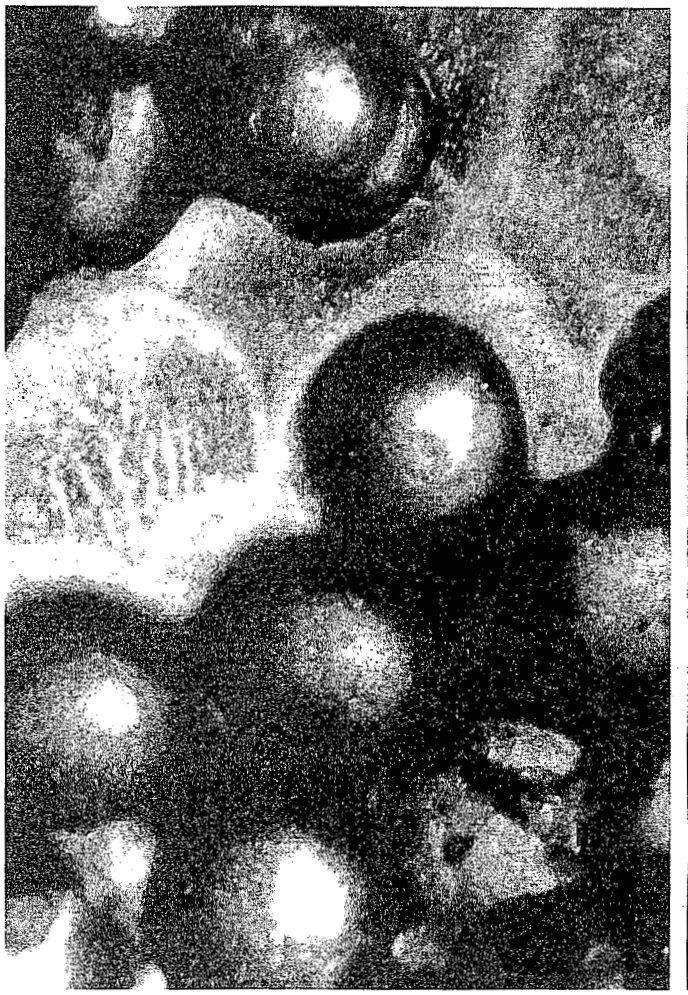
7-a
40x
Miscible Phase

7-b
40x
Waxy Depositions

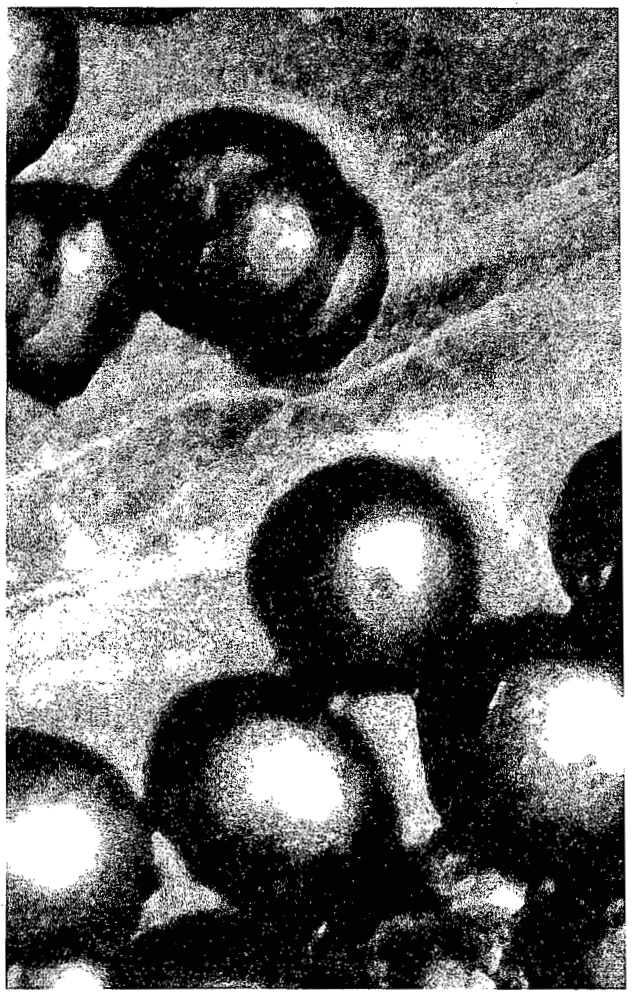
Photo 8. Semi-Miscible CO_2 Displacement



8-a
40x

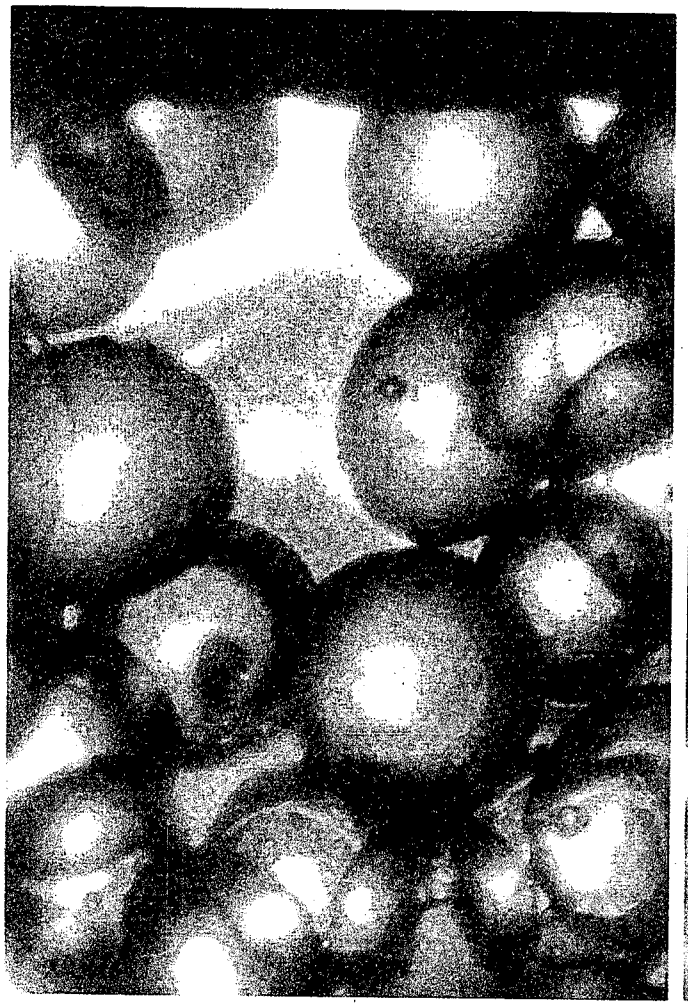


8-b
40x

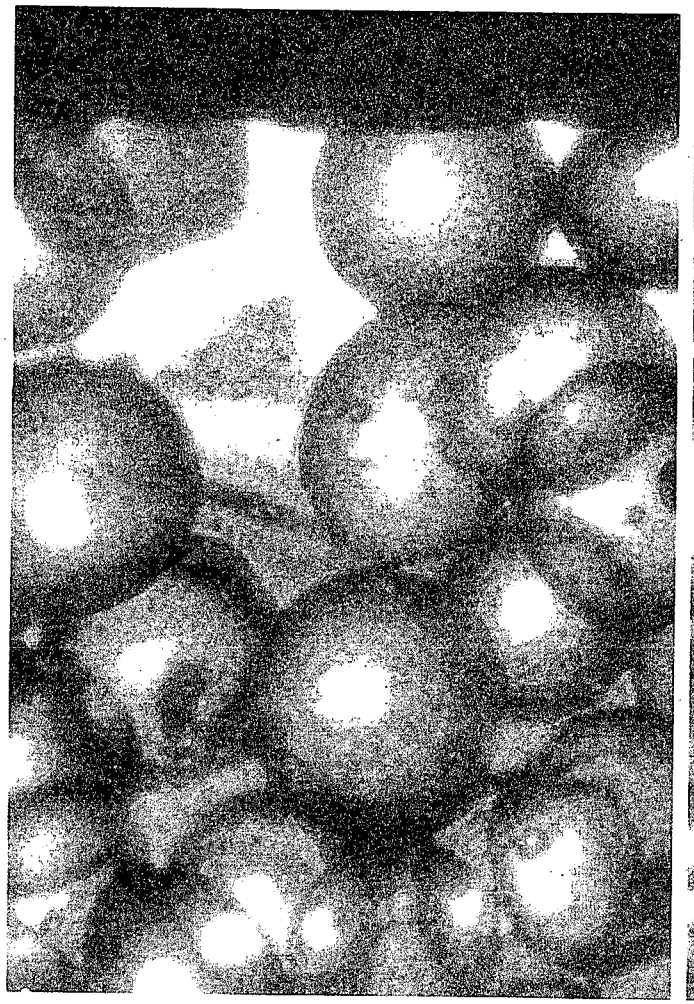


8-c
40x

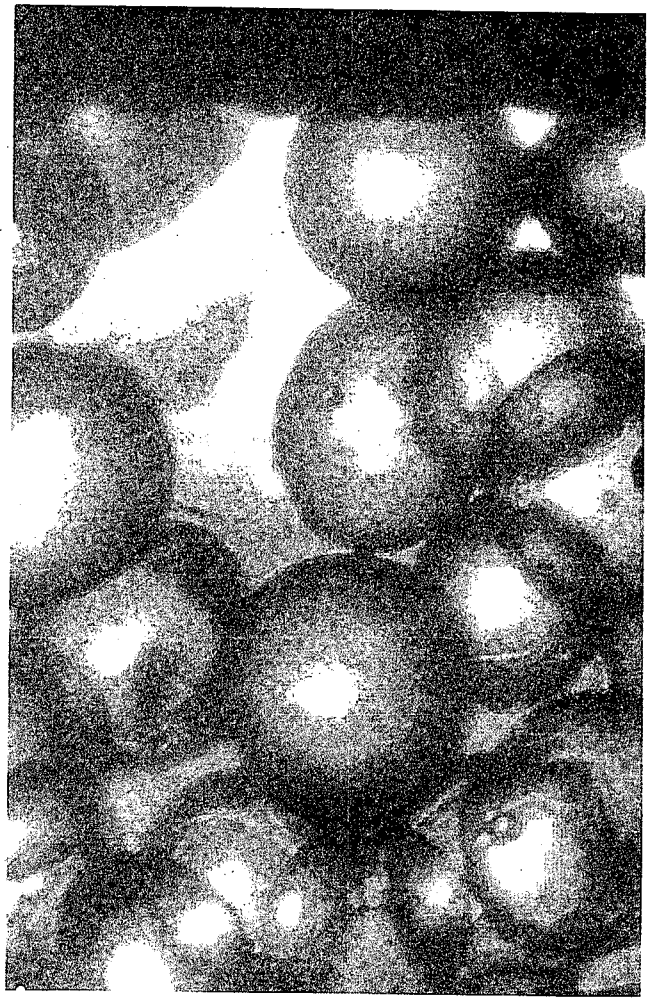
Photo 8. (continued)



8-d
40x
Oil Expansion

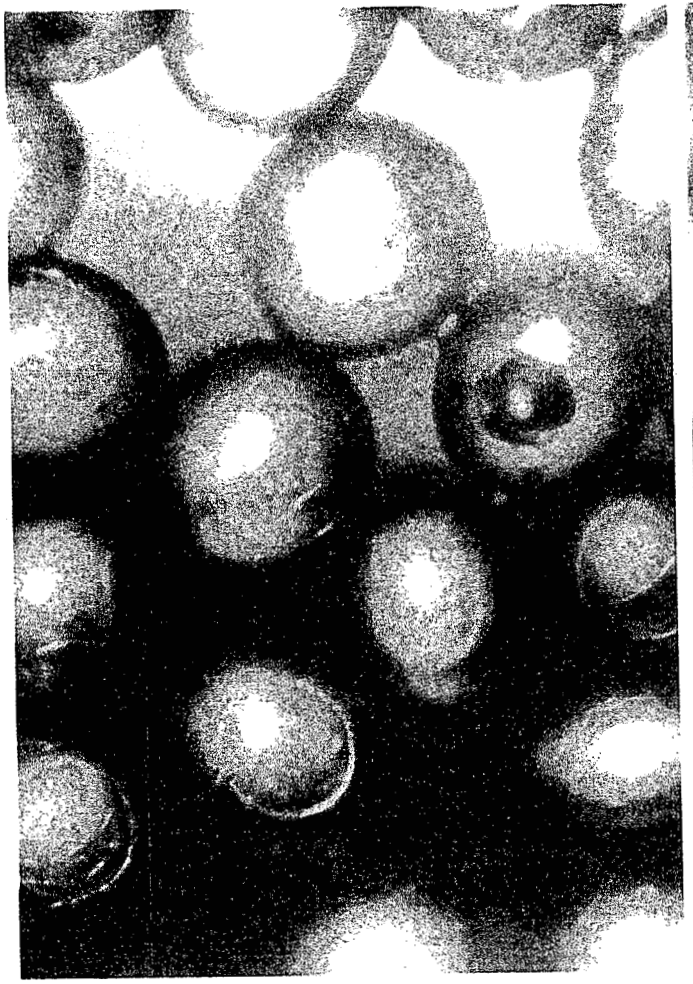


8-e
40x
Oil Evaporation

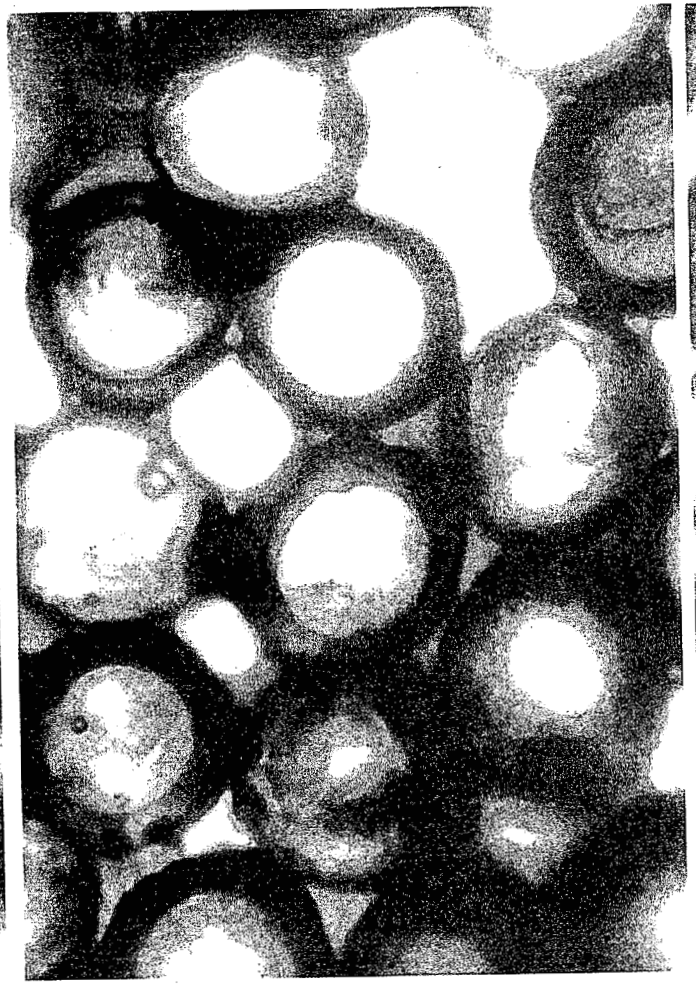


8-f
40x
Oil Displaced

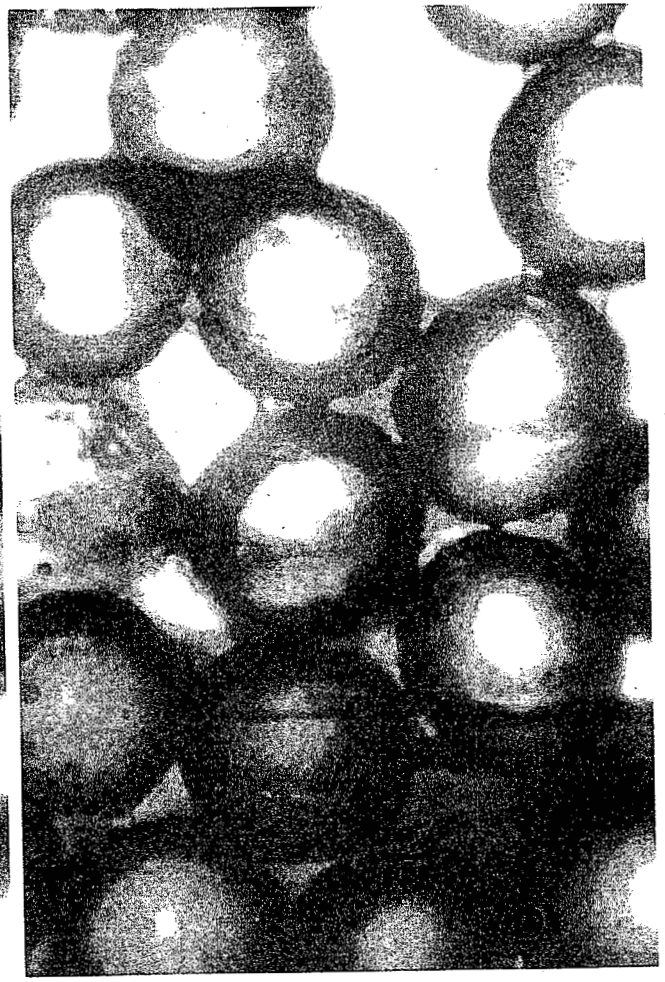
Photo 9. Transformation from Miscible to Semi-Miscible Displacement
Slaughter Estate Crude Oil



9-a
40x
Miscible Mixture

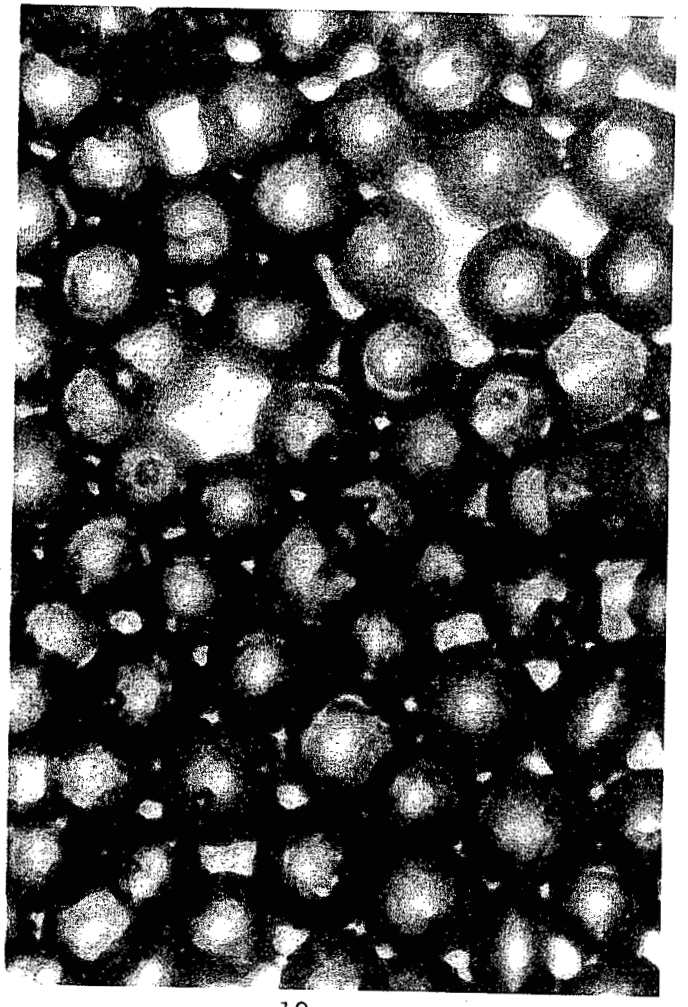


9-b
40x
Miscible Mixture Displaced

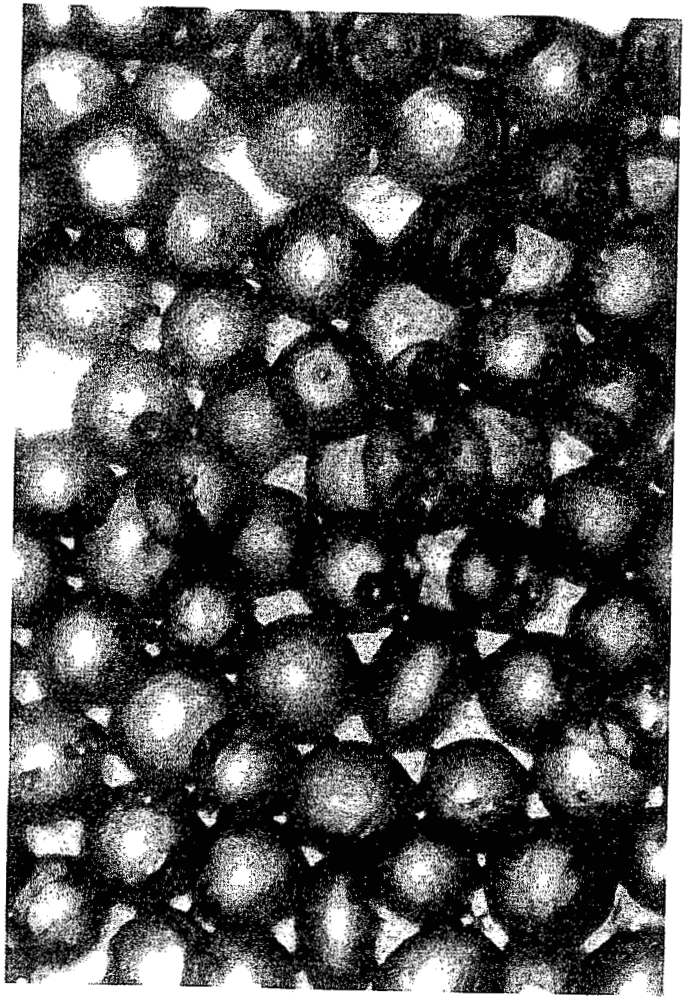


9-c
40x
Semi-Miscible Displacement

Photo 10. Residual Oil in CO_2 and in Water
SACROC Crude Oil

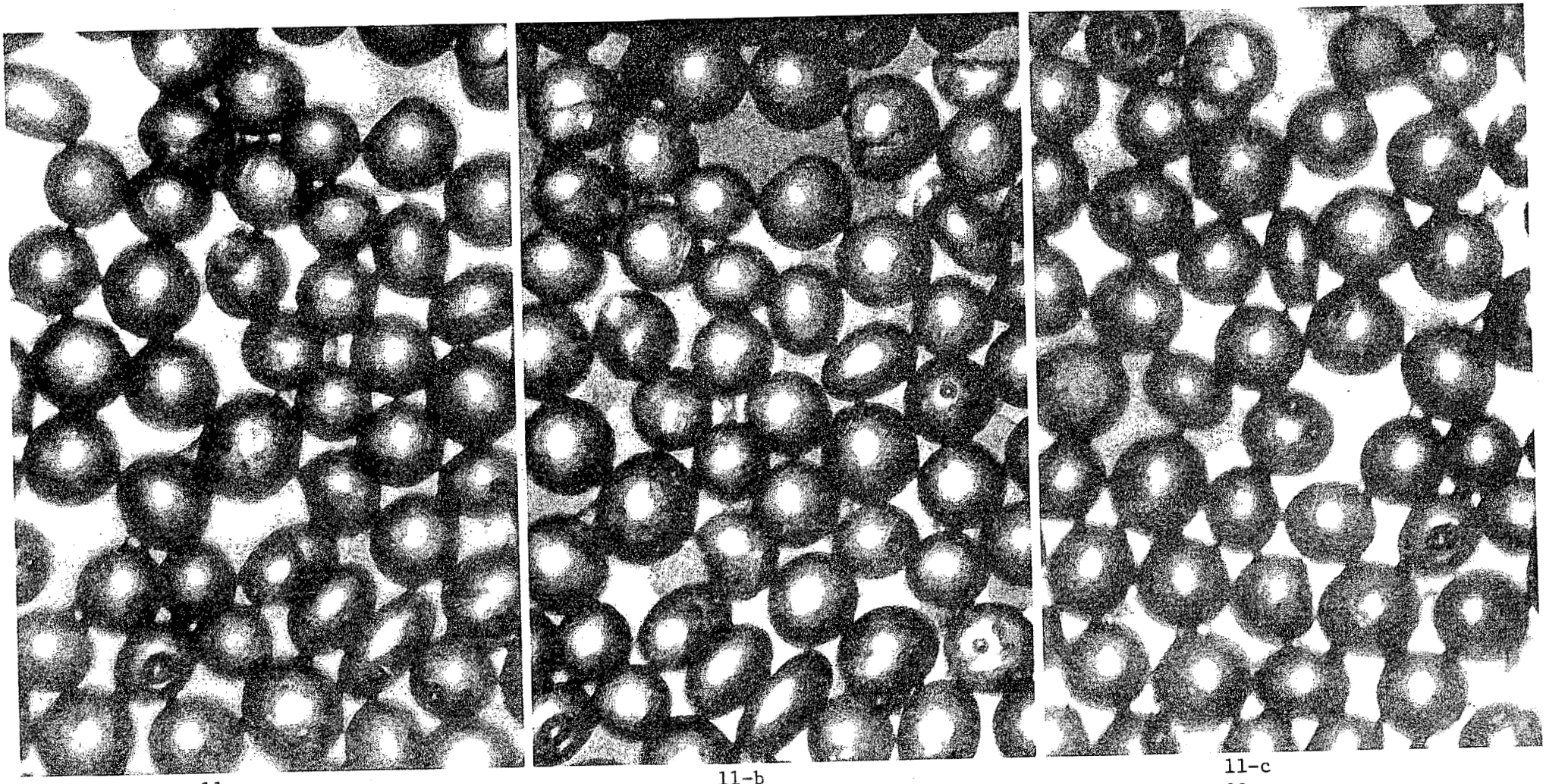


10-a
20x
Residual Oil After CO_2 Flooding



10-b
20x
Residual Oil Regrouped in Water

Photo 11. Water Channelling Through Miscible Zone
SACROC Crude Oil

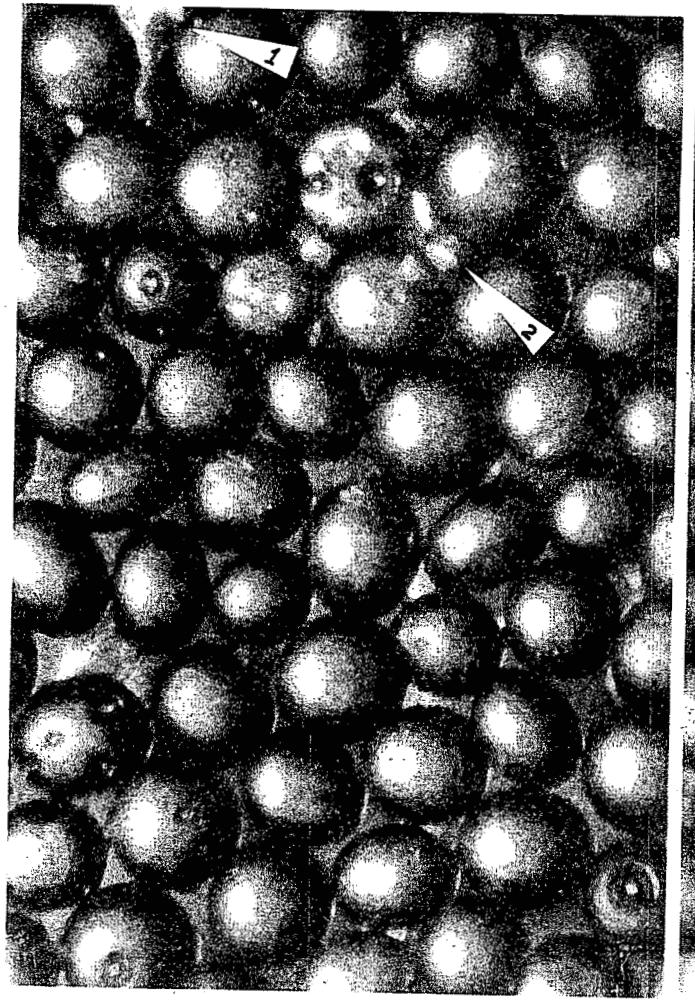


11-a
20x
Miscible Mixture

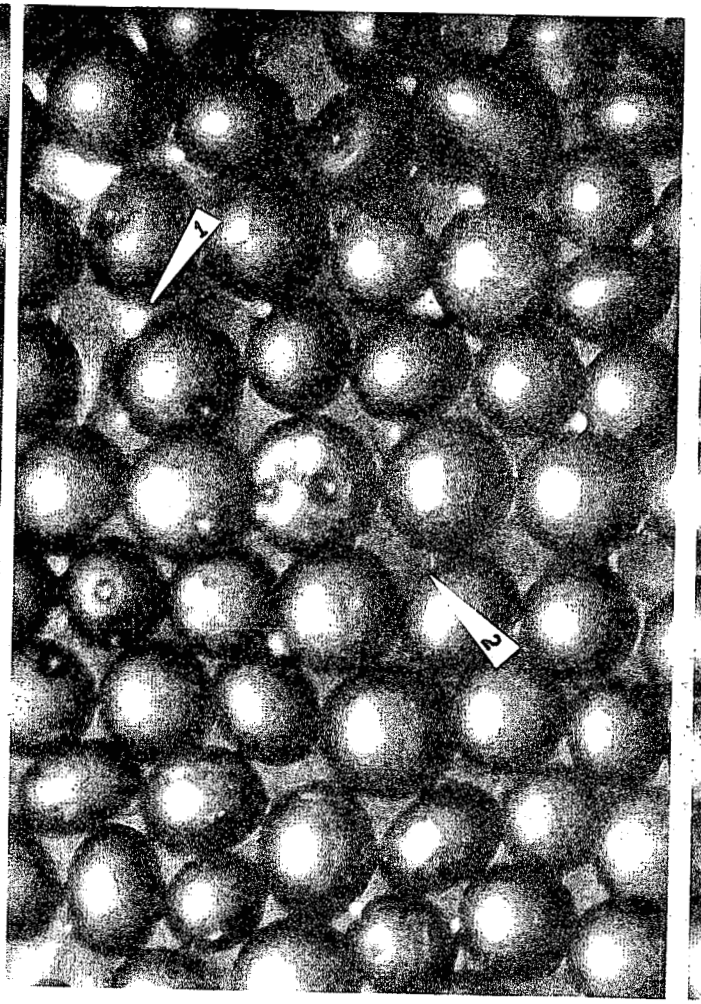
11-b
20x
Water Channelling

11-c
20x
Oil Trapped

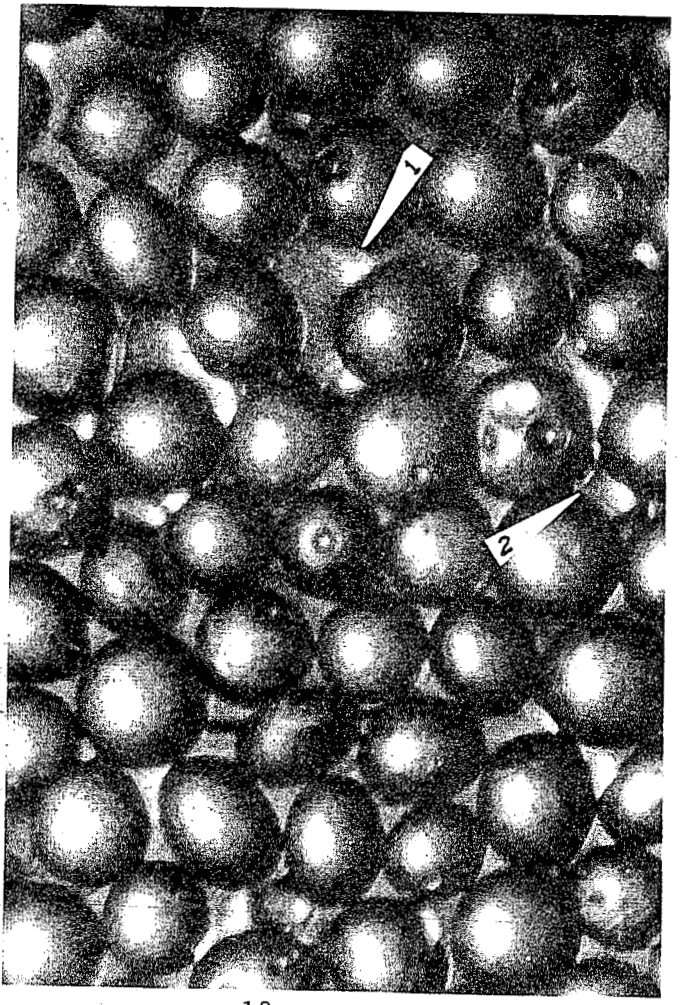
Photo 12. Semi-Miscible Displacement of Oil in Waterflooded Area
Penn-Grade Crude Oil



12-a
20x
Residual Oil

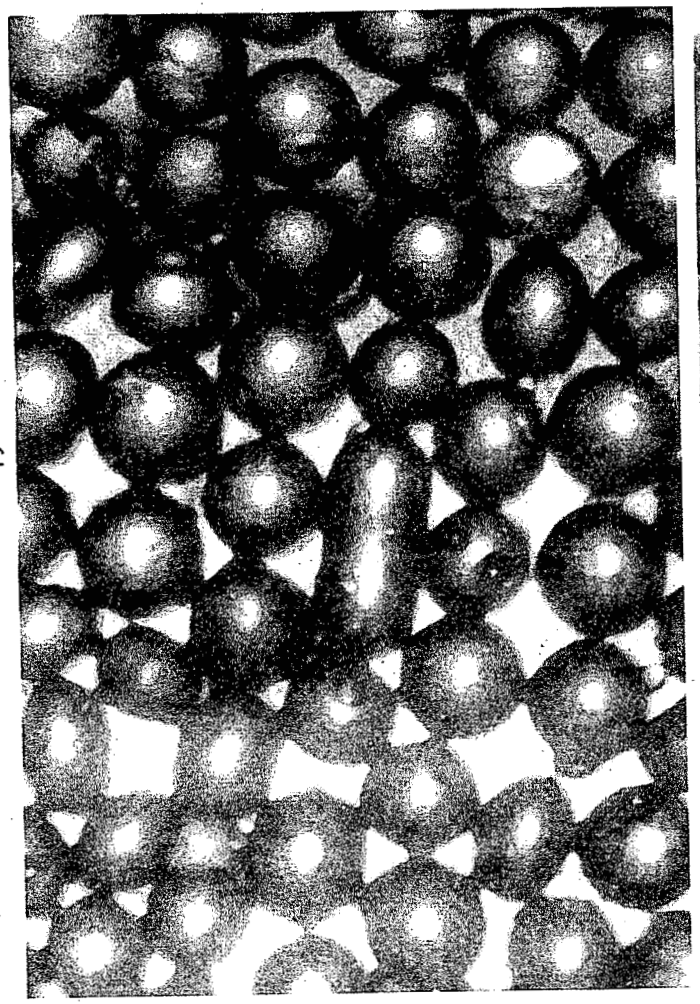


12-b
20x
CO_2 Encroaching and Oil Expansion

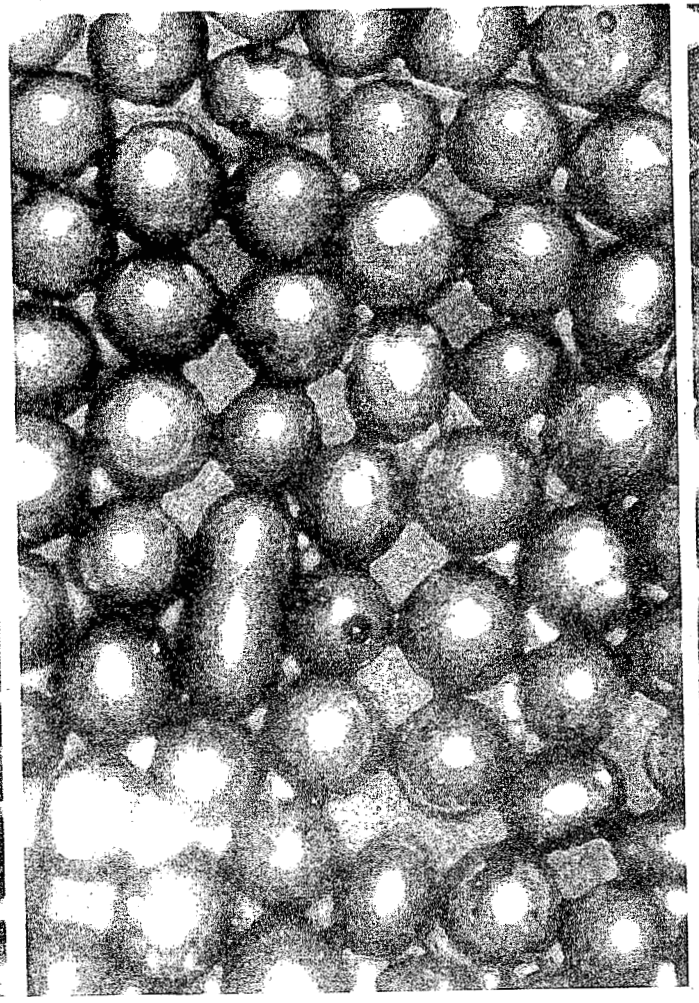


12-c
20x
Oil Displaced

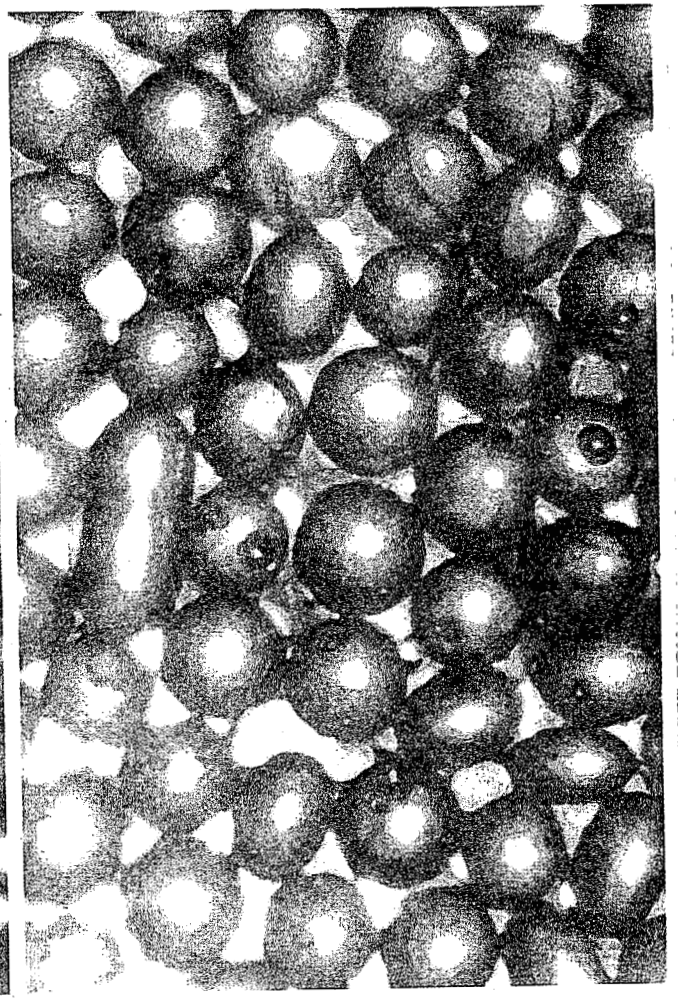
Photo 13. Development of Oil Bank in Waterflooded Area
SACROC Crude Oil



13-a
20x
Original Condition
After Waterflooding

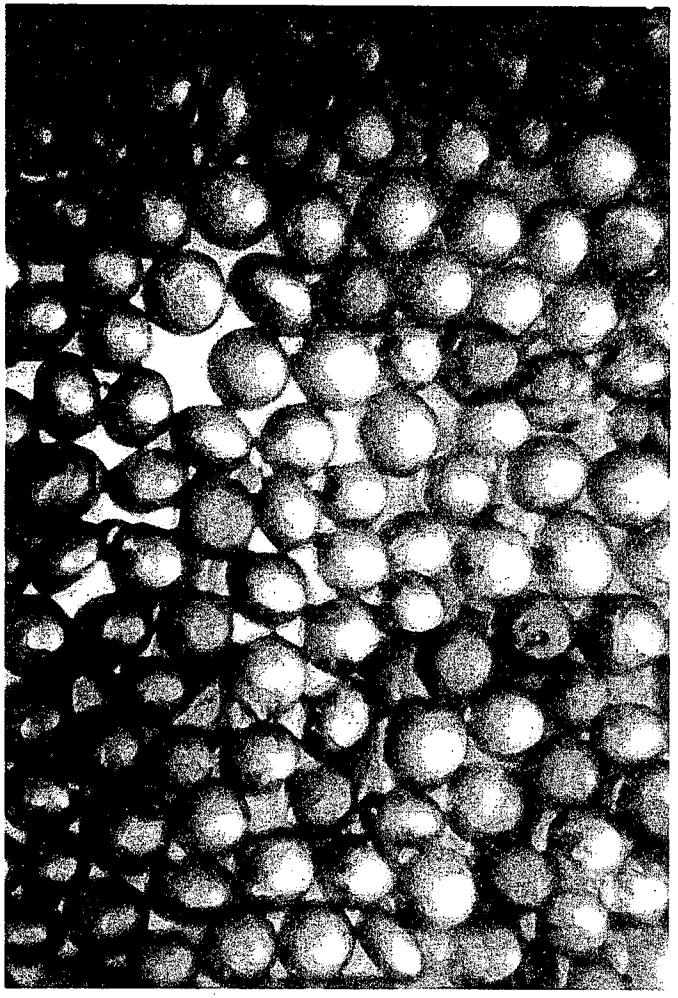


13-b
20x
Oil Bank

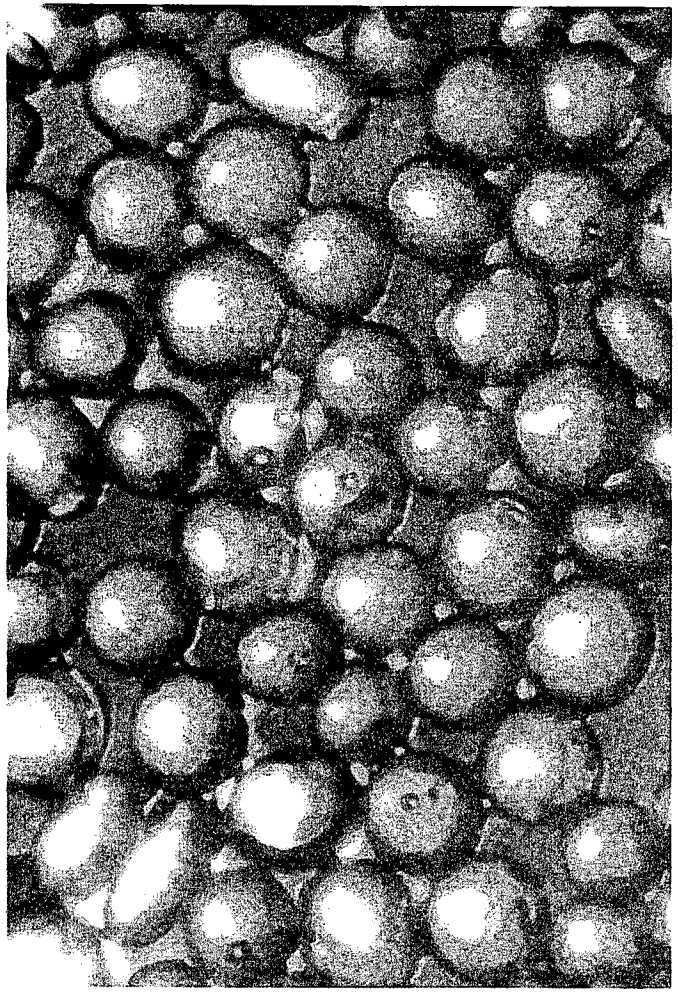


13-c
20x
Residual Oil After
CO_2 Flooding

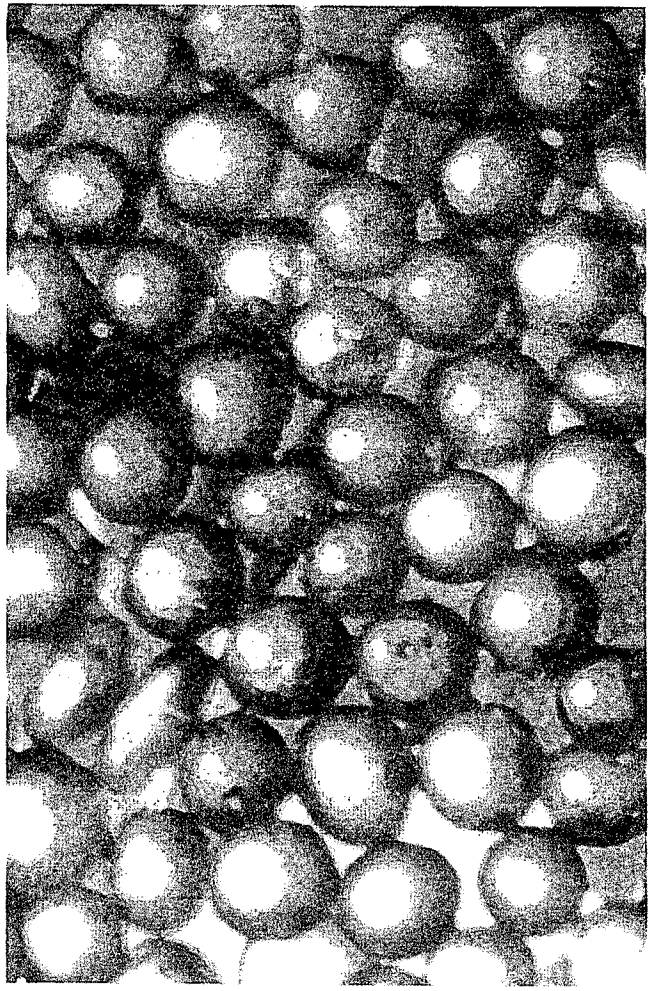
Photo 14. Disintegration of Oil Bank by Water
SACROC Crude Oil



14-a
15x
Residual Oil After
Waterflooding

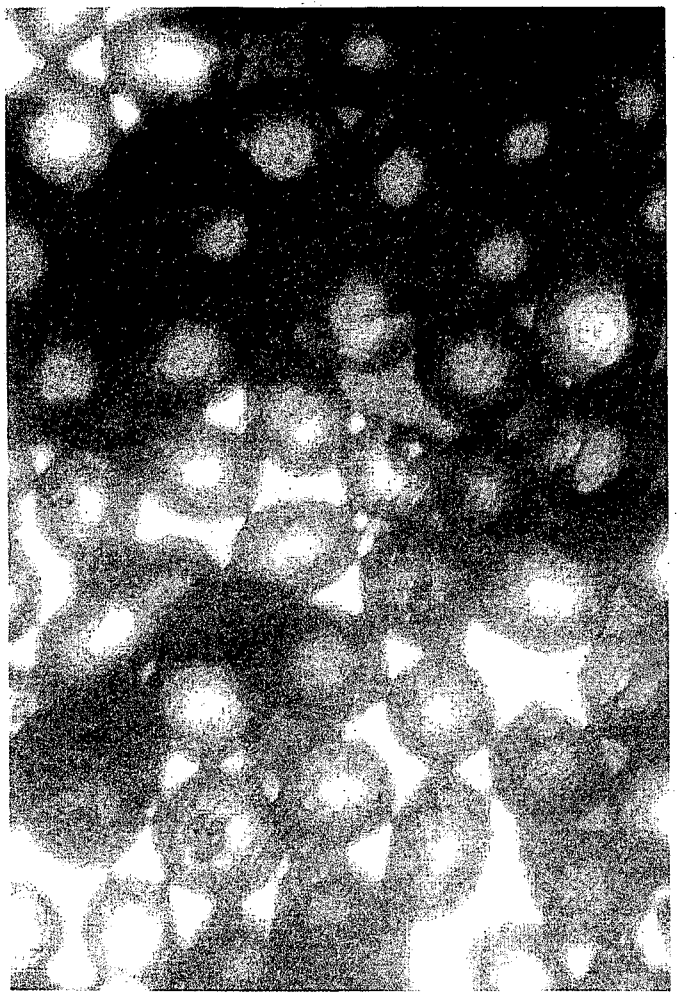


14-b
20x
Oil Bank

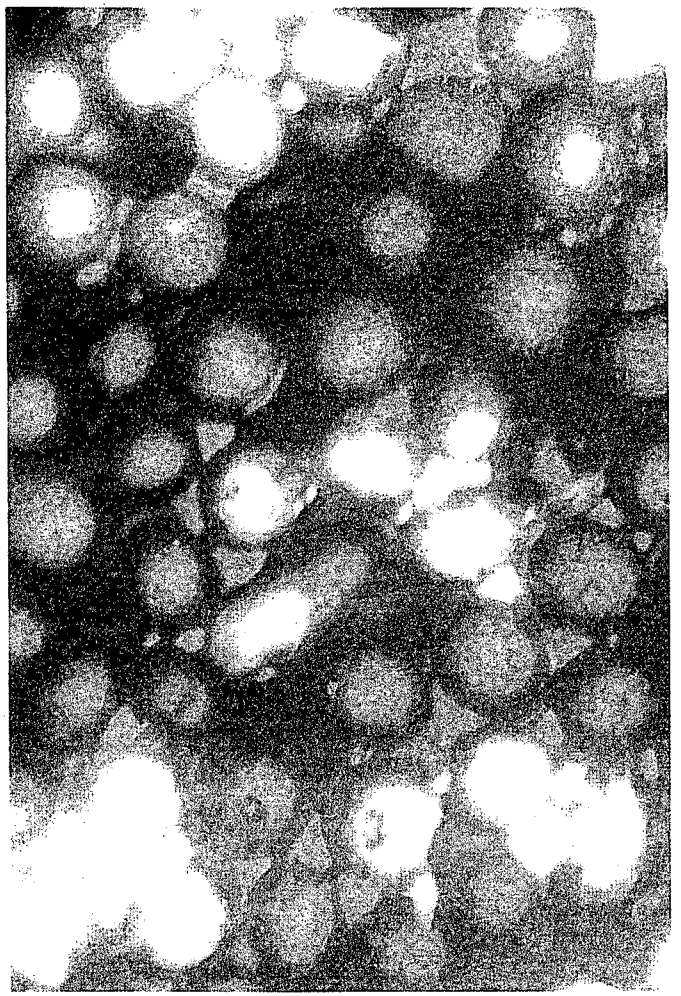


14-c
20x
Oil Bank Disintegrated

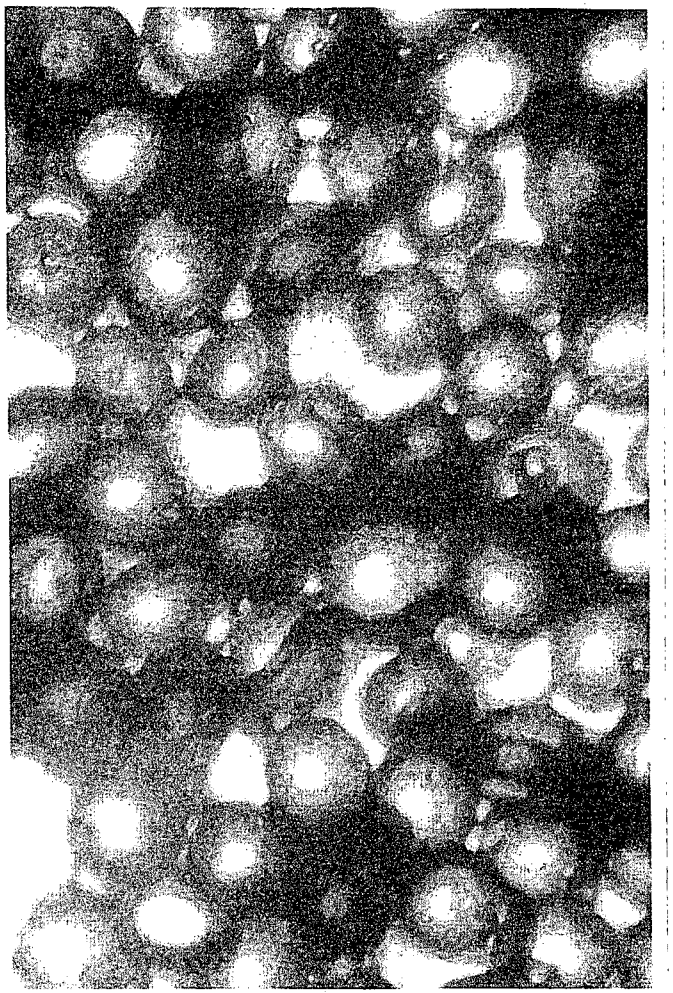
Photo 15. Residual Oil After Tertiary CO_2 Flooding
SACROC Crude Oil



15-a
20x
Residual Oil After
Waterflooding

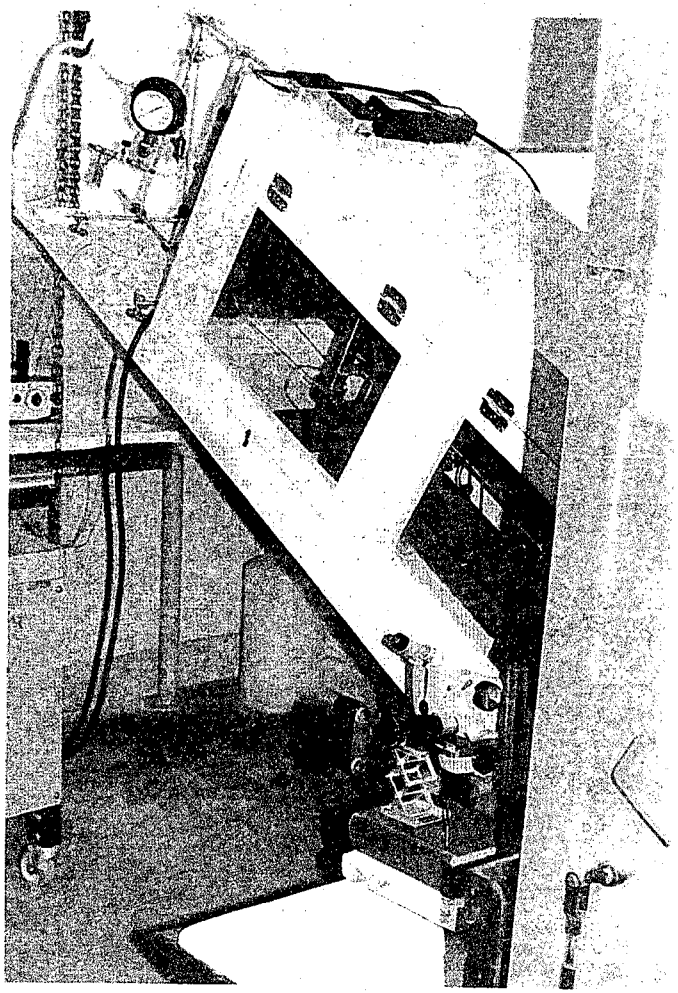


15-b
20x
Oil Bank

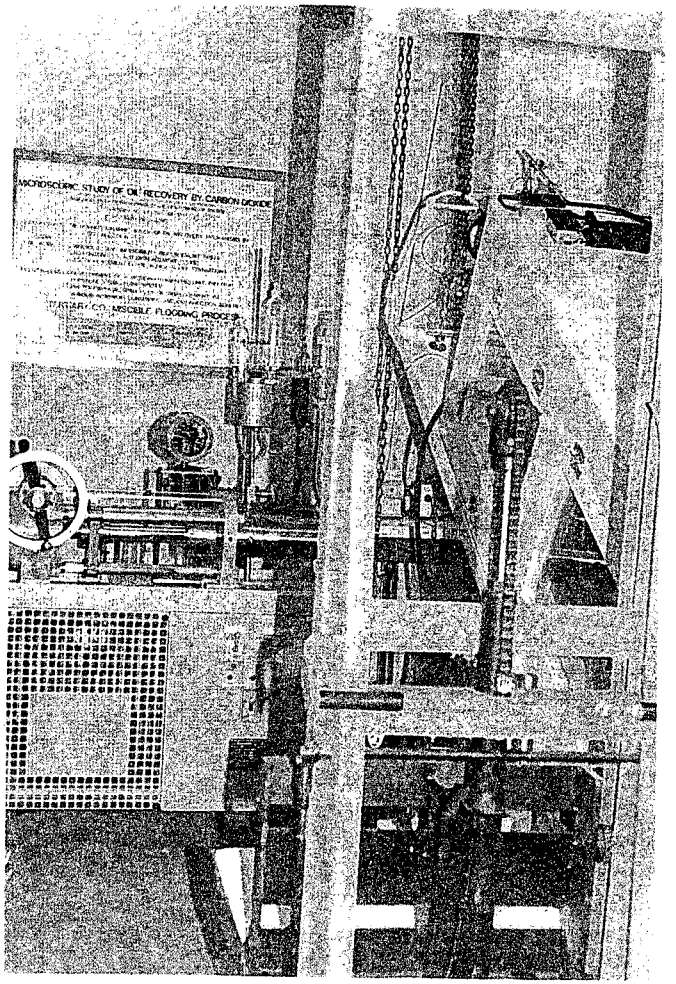


15-c
20x
Residual Oil After
CO_2 Flooding

Photo 16. Down-Dip Displacement Assembly

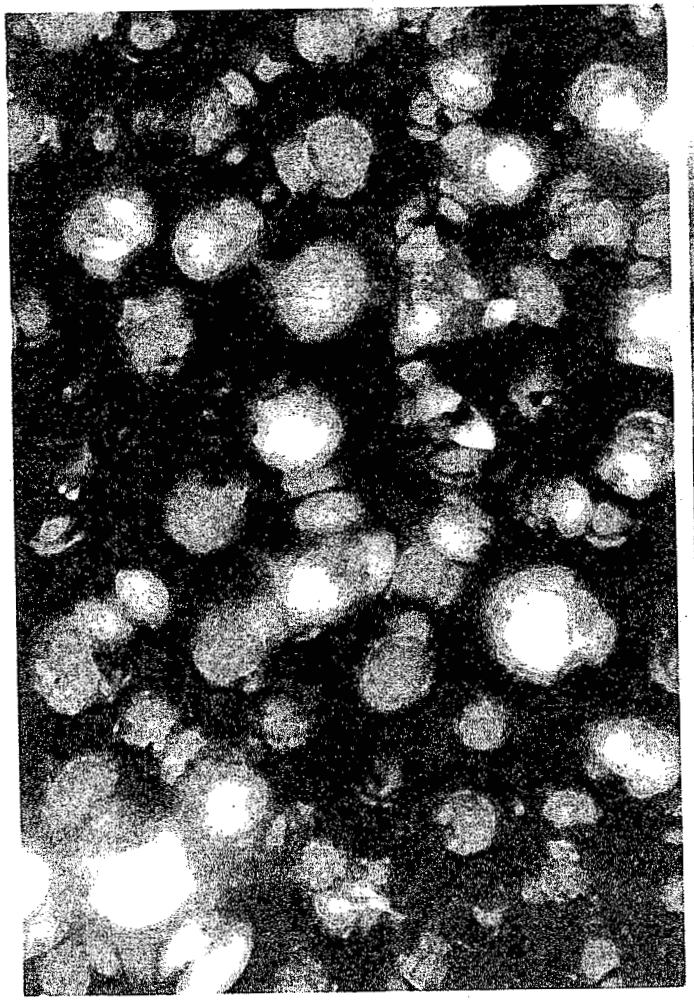


16-a



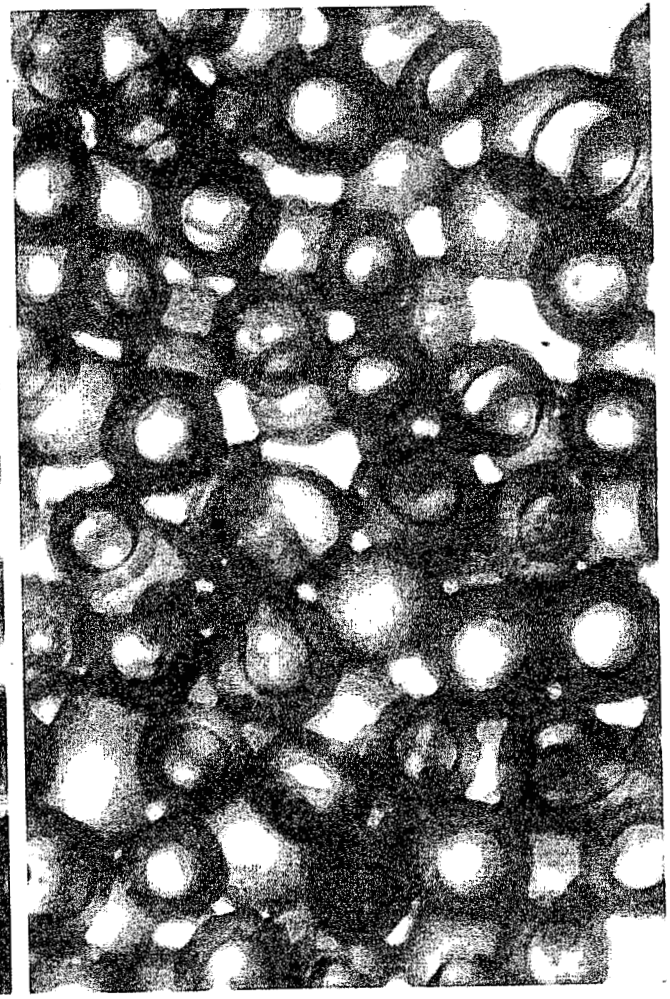
16-b

Photo 17. Down-Dip Displacement
Slaughter Estate Crude Oil
30°, 2000 psi



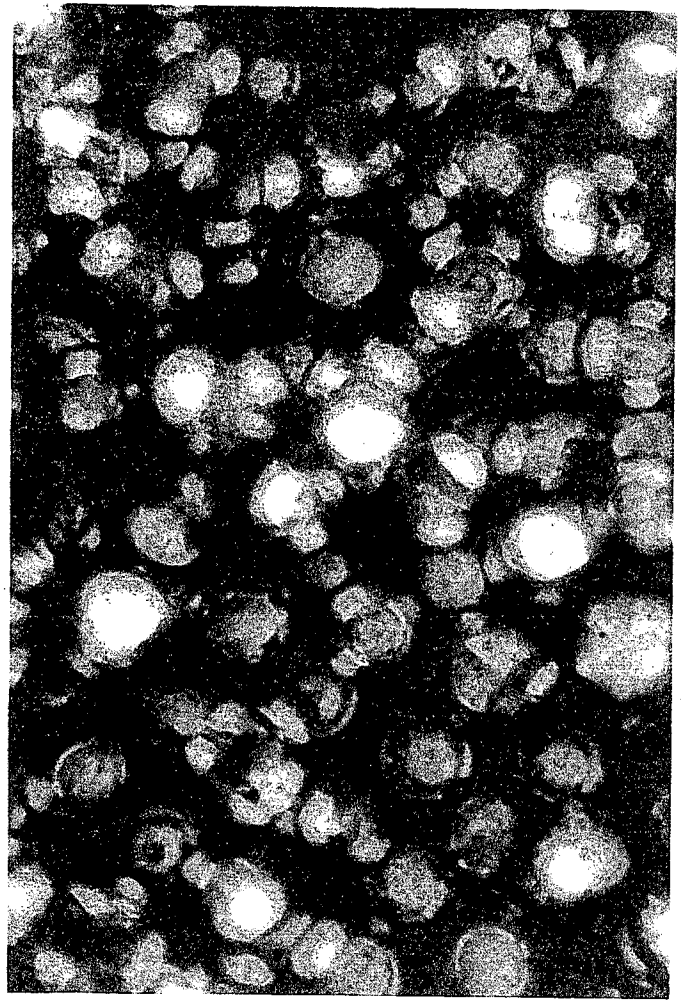
17-a
20x
Original Condition

17-b
20x
Miscible Mixture

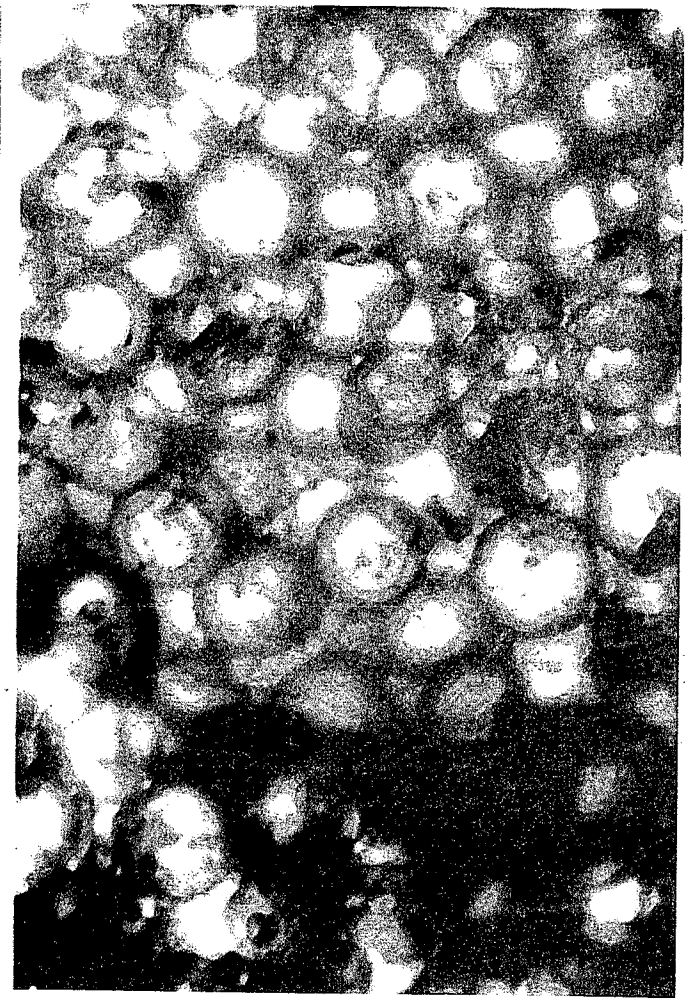


17-c
20x
Residual Oil

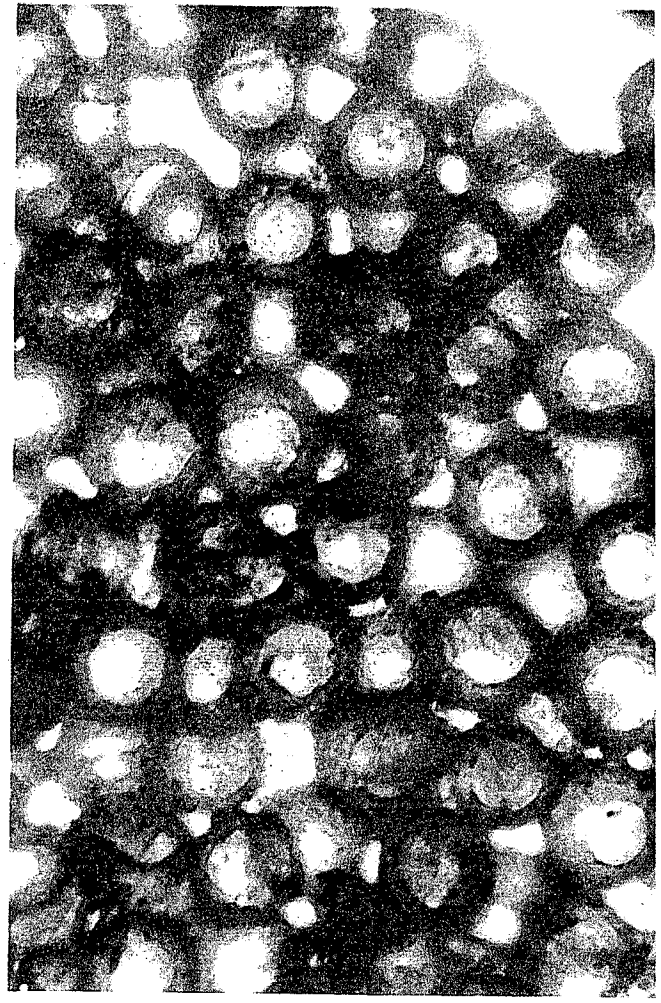
Photo 18. Down-Dip Displacement
Slaughter Estate Crude Oil
30°, 1500 psi



18-a
20x
Original Condition

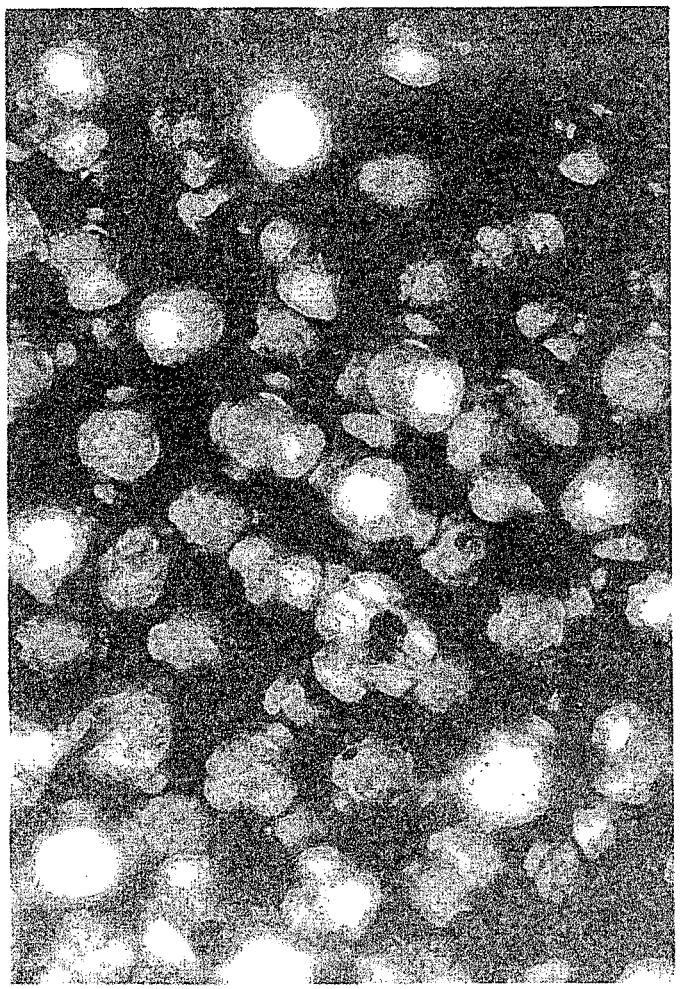


18-b
20x
Low Quality Miscibility

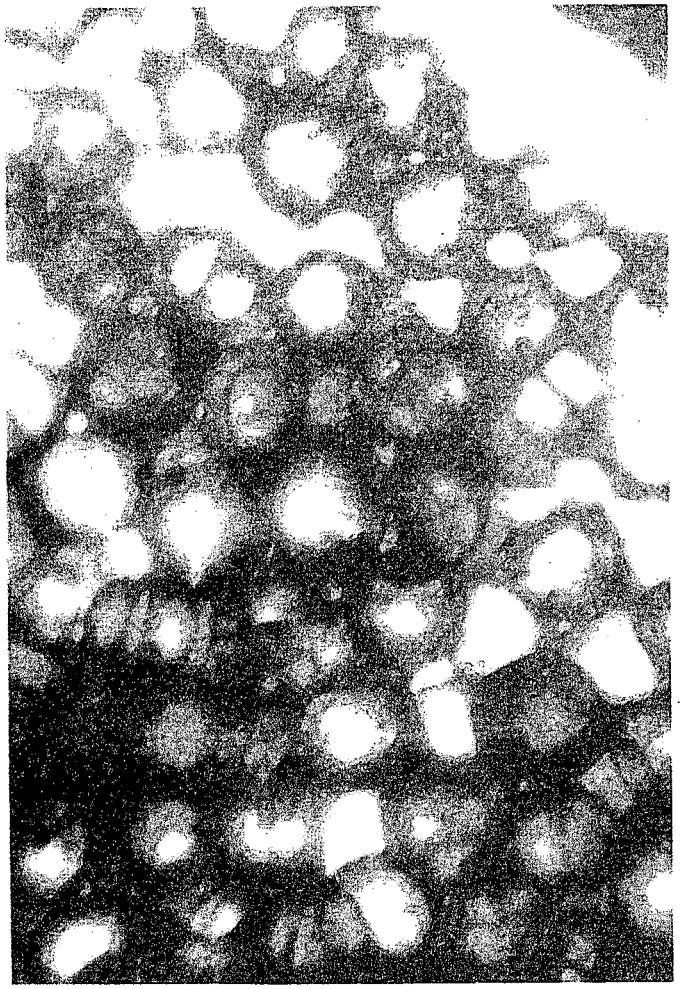


18-c
20x
Residual Oil

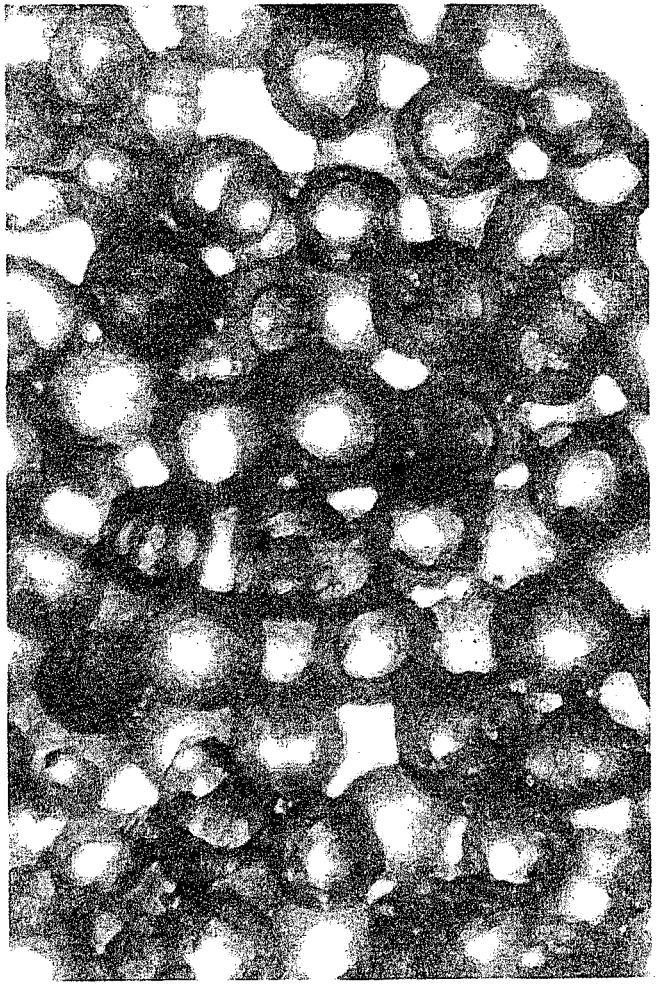
Photo 19. Down-Dip Displacement
Slaughter Estate Crude Oil
30°, 1000 psi



19-a
Original Condition



19-b
Immiscible Phase Boundaries

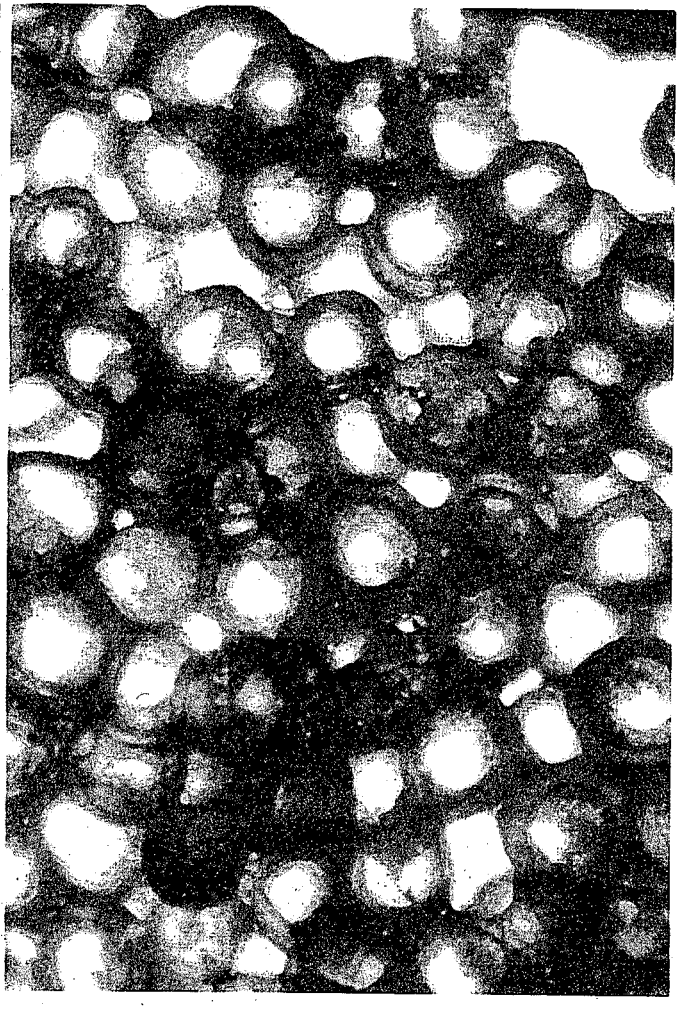


19-c
Residual Oil

Photo 20. Down-Dip Displacement
Slaughter Estate Crude Oil
15°, 2000 psi

20-a
20x
CO_2 Override

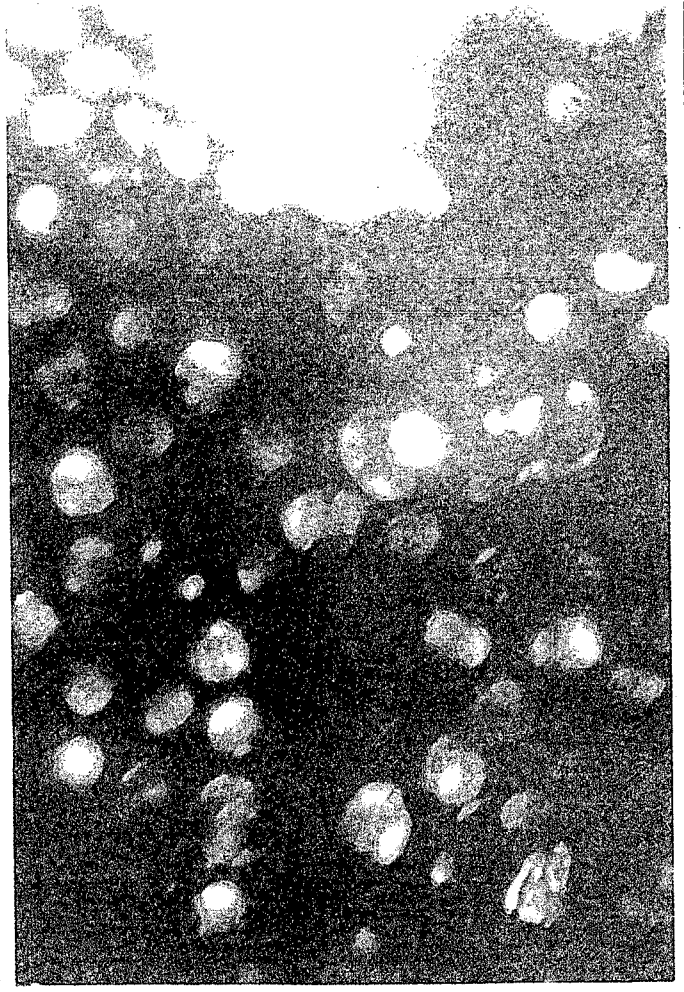
20-b
20x
Oil Expansion



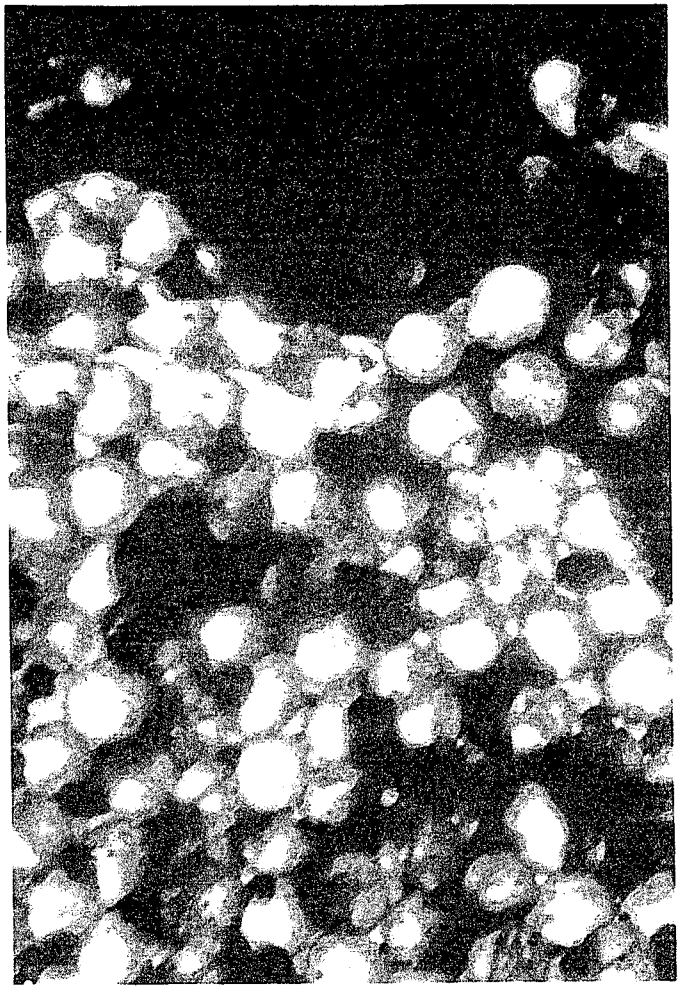
20-c
20x
Residual Oil

Photo 21. Tertiary CO_2 Flooding
Wilmington Crude Oil

21-a
20x
Original Condition
After Waterflooding

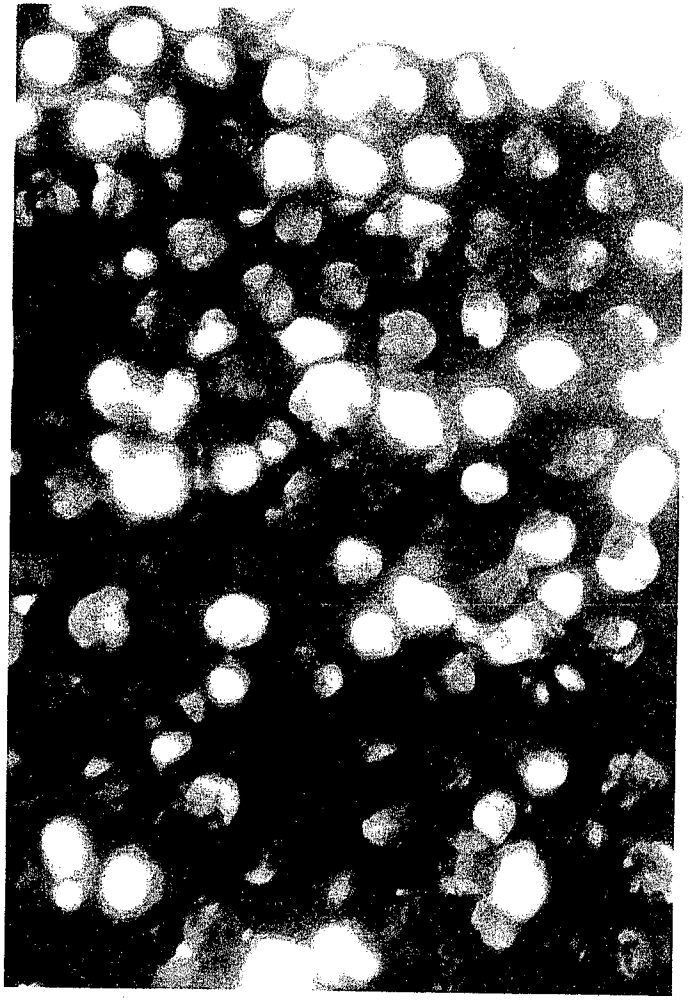


21-b
20x
Oil Expansion and
CO_2 Override

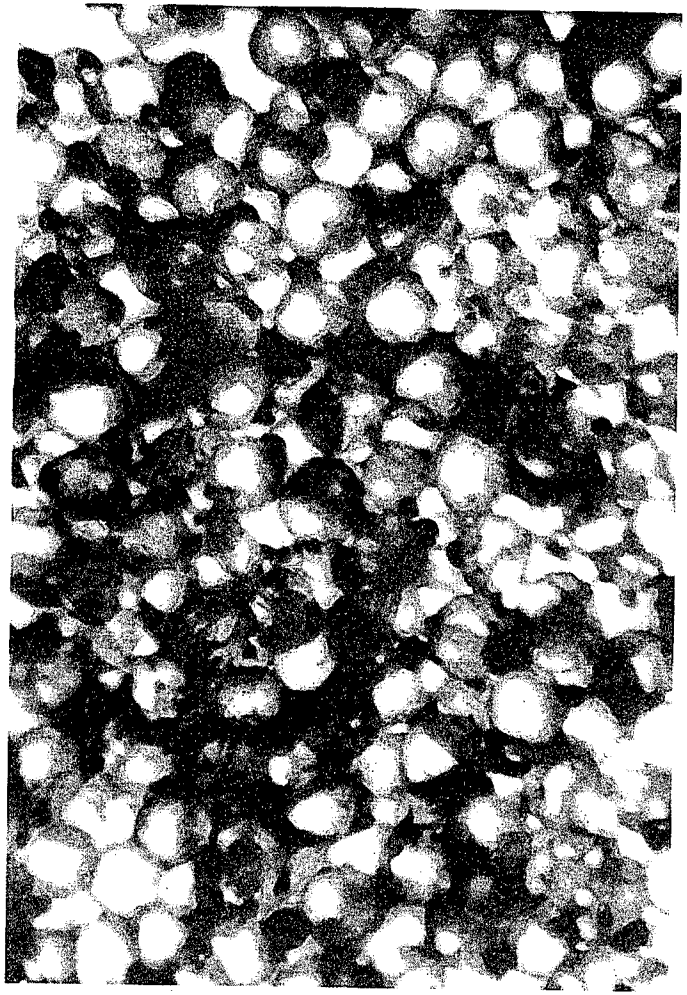


21-c
20x
After CO_2 Flooding

Photo 22. Tertiary CO_2-Foam Flooding
Wilmington CO_2 Crude Oil



22-a
20x
High Oil Saturation
Due to Oil Expansion



22-b
20x
Residual Oil After
Second Waterflooding