

A Topical Report

**BLOCKING OF HIGH PERMEABILITY ZONES IN
STEAMFLOODING BY EMULSION**

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BLOCKING OF HIGH PERMEABILITY ZONES IN STEAMFLOODING BY EMULSIONS

by

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Abstract

It is well-known that the economics of many steamflood EOR technologies are strongly affected by steam override (gravity segregation) and steam channeling. The economic performance of many steamfloods can be improved if an effective method of plugging the steam override or channeling zones is utilized. Traditionally, "foam" blocking techniques have been utilized with variable success to achieve these goals.

In this paper, the first-phase of laboratory results in the development of a new emulsion blocking technique for the correction and control of steam override and channeling are presented. Coreflood experiments were performed to demonstrate the effectiveness of emulsion blocking at temperatures ranging from ambient to 160° C. The permeability reduction of various types of cores using externally produced emulsions and in situ generated emulsions was measured for light and heavy crude oils. Externally produced emulsions injected into oil-free cores reduced the permeability by 86 percent at 110° and 77 percent at 160° C. Emulsions generated in situ using a caustic emulsifier with Wilmington crude oil achieved a 43 percent reduction in effective permeability with the absolute permeability of this core being 1,400 md. In both cases of injected and in situ emulsions the emulsion block was thermally stable and no time degradation of the block was observed.

The strong possibility that "foamblocks" as heretofore applied may have an emulsion blocking component to the overall plugging mechanism is discussed. Foams created with known foaming agents were contacted with various crude oils and the authors observed emulsified droplets within the liquid interfaces.

INTRODUCTION

The efficiency of many steamflood enhanced oil recovery (EOR) technologies is severely affected by the gravity segregation of the low-density injected steam and the high-density displaced reservoir fluids resulting in steam override near the top of the permeable reservoir layer where the oil saturation has been depleted. Cores taken from heavy oil reservoirs at the termination of a steam drive clearly show this upward migration of the steam and consequent segregation of the displacing (steam) and displaced (oil and water) fluids. An effective method to increase the efficiency of a steam drive is to plug the high permeability steam-swept override zone in order to direct steam to zones of higher oil saturation and thus improve the ratio of oil produced to steam injected.

Some heavy oil producers utilizing steamflood techniques have adopted the method of foam injection or in situ foam creation as a mobility buffer to effect such a diversion of steam from the steam-swept zone. These so-called

"foamblocks" are created by injecting a relatively concentrated solution of foaming agent (surfactant and perhaps a noncondensable gas) with the flowing high-temperature, high-pressure steam. The noncondensable gas allegedly supports the supposed foam structure, insofar as the steam would condense due to heat loss and the foam would collapse if the inert gases were not present.

To date there is no conclusive theoretical or laboratory analysis of exactly how a foam would increase the resistance to flow of steam in the steam-swept zone, and further there is no direct or indirect evidence of actual foam formation in the porous structure of the reservoir medium. Work performed at New Mexico State University (Holbrook, et al., 1981), however showed that foam viscosities are generally an inverse function of foam density. Thus, in areas where gas (steam) fingering is pronounced, these researchers postulated that foam viscosity would increase with gas saturation and thereby tend to mitigate steam channeling. Although foam is pseudoplastic (non-Newtonian) in character, the theoretical rheology of foam flow in a porous medium is not consistent with the mobility buffering concept for steam because of low yield points and high inherent compressibilities of the foam structure.

In early 1979, it was hypothesized by one of the authors that in effect what may be the actual mechanism for steam diversion in these "foamblocks" is the accidental creation of plugging macroemulsions since thermally stable surfactant agents such as alkyl-aryl benzene sulfonates were being utilized as successful "foamers". Thus, it was conjectured that macroemulsion plugging may be an effective method for correcting steam override and may further be a dominant or adjunct mechanism in the heretofore perceived foamblocking techniques.

LITERATURE BACKGROUND

A review of the literature resulted in several references relating to the use of emulsions as agents for causing permeability reduction. McAuliffe demonstrated that injection of externally produced oil-in-water emulsions at 75° F effectively reduces the water permeabilities of sandstone cores (1973). These laboratory findings were later substantiated by a successful field test of emulsion injection followed by waterflooding in the Midway-Sunset Field (McAuliffe, 1973).

Several waterflood recovery process patents assigned to Texaco, Inc., describe laboratory core studies in which blocking emulsions were produced in situ by injection of surfactant mixtures (Varnon, et al., 1979, Schievelbein, 1979, and Schievelbein, et al., 1979). The conditions under which these experiments were performed (relatively high salinity, presence of divalent ions, crushed limestone cores) allow us to infer the creation of water-in-oil emulsions. Cooke, in laboratory experiments, has observed that a viscous oil-external emulsion may be responsible for the large increase in pressure gradient that is observed immediately behind the displacement front during alkaline waterflooding under saline conditions (1974). A recent waterflood patent assigned to Mobil Oil Corporation relates to the creation of a plugging emulsion within a high salinity stratified reservoir (Hurd, 1984). The surfactant/water/oil emulsion described in this patent is likely oil external.

Special Requirements

Special requirements must be met in order to extend the use of emulsions as mobility buffers to steamflood conditions. The chemicals used and the emulsions produced must be stable for long periods of time at steamflood temperatures. Some chemical surfactants such as sulfates would be expected to hydrolyze too rapidly under such conditions.

Since many steamfloods are performed in the laboratory and in the field with steam generated from fresh water, the level of salinity in the steamflooded channel is expected to be low. Consequently, the emulsion must be stable under low salinity conditions.

Conditions within the steamflooded channel are likely to be more favorable to the presence of oil-in-water emulsion than water-in-oil emulsion. Fresh water systems are generally associated with highly stable oil-in-water emulsions (Mayer, et al., 1982). Entrapment of oil droplets, a mechanism for reducing permeability to water, is associated with alkaline floods performed using fresh water.

Emulsion Formation

Oil-in-water emulsions can be produced by mixing oil with an aqueous solution of emulsifier (agent-in-water method) or by utilizing the naturally occurring surfactants already present in some oils (agent-in-oil method). Either method is suitable for creation of the emulsion above ground and then injection of that emulsion into the reservoir.

In situ formation of oil-in-water emulsions adds the requirement that the emulsification proceed spontaneously or at least with very little energy input due to mixing. Most such systems are associated with the agent-in-oil procedure and spontaneous emulsification to oil-in-water emulsions does often occur when mixing aqueous caustic and petroleum oils containing naphthenic acids. Some researchers propose that diffusion of the naturally occurring surfactants across the interface is the mechanism that causes this phenomena (Becher, 1983).

Emulsification with caustic is possible with oils that have a total acid number (TAN) greater than 1.5 mg KOH/gm oil. Below 1.5, the oils will either not emulsify or will form water-in-oil emulsions. The rate of emulsification with caustic is much faster than emulsification with surfactant mixtures, which is a characteristic property for emulsions generated via the agent-in-oil procedure (Becher, 1983).

Although much literature exists regarding the spontaneous formation of thermodynamically stable microemulsions, which do not contain droplets large enough to cause permeability reductions, little information is available regarding the spontaneous formation of macroemulsions which contain larger oil droplets and are not thermodynamically stable. However, review of the literature does indicate that some aqueous surfactant mixtures (agent-in-water) may promote the spontaneous formation of macroemulsions. Researchers at the University of Texas have concluded that spontaneous emulsification - distinctly different from the low tension processes - between oil and an aqueous phase containing petroleum sulfonates occurs with specific

hydrocarbons and not with others (Cash, et al., 1975 and Schechter and Wade, 1976). The spontaneous emulsification observed occurred only in a narrow region at the interface and was not observed to occur for paraffins or for crude oil.

Emulsion Properties

In most cases, the type of an emulsion (oil-in-water or water-in-oil) can be predicted by the appearance of the emulsion. In general, oil-in-water emulsions will appear to be chocolate or brown in color and dilute easily with water, although creaming will occur eventually if agitation ceases. An important property of the emulsion is the droplet size distribution. If the droplets are too small, they may tend to slowly solubilize into the continuous phase or not block at all, and if they are too large, creaming and coalescence may become problems. The oil droplets in macroemulsions normally are between 1 and 50 microns in diameter and are easily visible with an optical microscope. Emulsions produced via the agent-in-oil procedure usually have more uniform droplet sizes and are quite stable (Wasan, 1976). The viscosity of oil-in-water emulsions remains low enough to pump easily. The electrical conductivity of oil-in-water emulsions tends to be that of the aqueous phase.

Water-in-oil emulsions, on the other hand, generally appear to be black in color, do not dilute with water, and have electrical conductivity lower than that of the brine. The viscosities may be very high and thixotropic.

Blockage Mechanisms

McAuliffe's concept of the mechanism by which an oil-in-water emulsion can cause a permeability reduction is shown in figure 1. In this case, the oil droplet is large enough to cause blockage by lodging within the pore throat. For this situation, the flow of a dilute, stable emulsion in a porous media is similar to a filtration process. If the pressure gradient across the drop becomes great enough, the drop may be forced on through the pore throat. Another process for reducing permeability has been observed by Soo. When emulsions are injected into a porous media micro-model, drops not only block pores of throat sizes smaller than their own, but they are also observed to capture on pore walls and in crevices (Soo, et al., 1984).

It is important to observe that a reduction in permeability from emulsion plugging may not necessitate that the median droplet size equal or exceed the median pore throat diameter. Competition from an ensemble of smaller droplets "crowding" a single pore throat would have the same effect in blocking a pore throat as would one large droplet as shown in figure 1. Another important (but speculative) mechanism of emulsion plugging to consider, is the decrease in relative permeability of the gas (steam) phase due to the presence of an additional competing emulsion phase. Here again, emulsion droplets smaller than the median pore throat size in the porous structure would possibly play a role in the overall blocking mechanism.

Finally, permeability reductions attributed in the literature to the formation of water-in-oil emulsions are evidently due to the high viscosity of those emulsions or to the formation of an oil film (lamella) across the pore throat (Cooke, 1974).

EXPERIMENTAL

Study of Emulsification

The crude oils selected for the study are a 19° API California crude from the Wilmington Field and a 33° API mid-continent crude from the Delaware-Childers Field in Oklahoma.

Emulsions were tested by mixing an emulsifier (either caustic or commercial surfactants dissolved in water) and the crude oil, then heating the sealed container to 110° C in an oven. After heating, the sample was removed from the oven and placed in a mechanical shaker for 15-20 minutes, then returned to the oven. This procedure was repeated three times before the sample was left in the oven for observation.

Emulsification of the Wilmington oil with caustic proceeds almost spontaneously. The stability of 50 percent oil-in-water emulsions produced with Wilmington oil and caustic is given in table 1. These oil-in-water emulsions are quite stable at 110° C for long periods of time. The optimal concentration of sodium hydroxide occurs at 0.42 percent NaOH where a uniform single-layer oil-in-water emulsion is produced. At higher NaOH concentrations, the increasing ionic strength of the solution results in formation of upper layer water-in-oil emulsions.

Droplet size distributions for oil-in-water emulsions were determined with a Model TA II Coulter Counter. The quantitative results obtained with the Coulter Counter were verified by qualitative observations with an optical microscope. The droplet size distribution for the Wilmington oil at optimal NaOH concentration is given in figure 2 along with the size distributions for some other oil systems. The pore size distribution for a 300 md Berea core is also given for comparison.

The total acid numbers (TAN) and the experimentally determined optimal NaOH concentrations for Wilmington and other viscous, asphaltic crude oils are given in table 2.

Oils with low TAN such as the Delaware-Childers crude cannot be emulsified with caustic. Attempts were made to produce an oil-in-water emulsion which is stable at 110° C by using petroleum sulfonates of different average weights as the emulsifying agent. The Delaware-Childers oil was mixed with the aqueous emulsifier at a ratio of 1:6 and tested according to the procedure previously described. If all of the oil emulsified, this would correspond to a 14.3 percent oil-in-water emulsion. Results of emulsification tests performed with petroleum sulfonates at 3.75 percent active concentration and Delaware-Childers oil are given in table 3. Also given are results for mixtures of 3.75 percent petroleum sulfonates with 2.5 percent SE-463, a water soluble ethoxylated sulfonate furnished by GAF Chemical Company. The volumes of the layers were observed after 16 hours at 110° C. The largest volume of stable oil-in-water emulsion was obtained for the mixture of 3.75 percent Petrostep 420, a medium equivalent weight petroleum sulfonate, and 2.5 percent SE-463 at 4.25 percent NaCl.

All of these systems, after shaking, separated into two distinct layers. At low salinities, the lower layer consists of oil-in-water emulsion which is stable for some period of time, depending on the particular system. At higher salinities inversion occurs, and the lower layer separates as a clear liquid with no oil droplets - the upper layer then becoming a water-in-oil-emulsion.

The optimal system consisting of 3.75 percent Petrostep 420 and 2.5 percent SE-463 was particularly stable and emulsions up to 33 percent Delaware-Childers oil were easily prepared. The size distribution for this emulsion is given in figure 2 and is broader than the size distribution for other emulsions - typical for an emulsion prepared via the agent-in-water procedure.

Since the systems with Delaware-Childers oil did not result in spontaneous emulsification - desirable for performing emulsification in situ - to macroemulsions, a procedure similar to an "emulsifiable concentrate" was tested. The system tested resembles dispersant mixtures used for treatment of oil spills (Blanchard and Dudley, 1976). These mixtures consist of surfactants dissolved in an oil compatible solvent. The dispersant formulated consisted of sorbitan partial fatty ester, dodecylbenzene sulfonate, and isobutyl alcohol in methylisobutyl ketone. Upon contact with the Delaware-Childers oil and water, a macroemulsion forms spontaneously. Although the quantitative size distribution has not yet been determined, observation with the optical microscope revealed droplets in the 5-10 micron range.

Coreflood Test Procedure

Laboratory coreflood experiments were performed to test the effectiveness of emulsion blocking in improving sweep efficiency at elevated temperatures. The emulsions, prepared as previously detailed, were diluted to 0.5 volume percent oil before injection into the cores. The emulsion reservoir was stirred slowly to prevent the dispersed oil droplets from creaming. Creaming was more a problem with the light oil emulsion than with the heavy oil emulsion.

Berea cores (10 in. x 1.5 in.) used in the experiments were fired at 800° F to minimize the effects of clay-water reactions. After firing, the cores were saturated with brine, mounted in a Hassler type core holder, and placed in a temperature controlled box. After determining initial absolute permeability, the cores were either left oil-free or saturated with oil and waterflooded to residual oil saturation.

Fluid injection, pressure monitoring, and temperature were controlled by an HP85 microcomputer system. Injections were done at constant flow rate with a Constametric III metering pump, from which the filters were removed.

Coreflood Tests with Oil-Free Cores

The coreflood experiments were at first performed at ambient temperature and then extended to hot water conditions at 110° C as an approach to saturated steam conditions. Pilot experiments with the light mid-continent crude were extended to the heavier California crude oil, with an actual steamflood at saturated steam conditions (160° C) to test the steam stability

of an "emulsion block" created with the heavier oil. The data for these coreflood tests are summarized in table 4.

Figures 3 and 4 illustrate the effects on effective permeability to water of injecting the 0.5 percent oil-in-water emulsions created from Delaware-Childers oil. At 25° C, a 68 percent reduction in permeability occurred after injecting 9.5 PV of emulsion. At 90° C, 10 PV of emulsion resulted in a 95.2 percent reduction in permeability, with most of the reduction occurring within 3 pore volumes.

Figures 5, 6, and 7 are the permeability reductions that resulted when injecting 0.5 percent emulsions produced from the Wilmington oil and caustic. The temperatures are, respectively, 25, 90, and 110° C. The reductions in permeability were from 84 to 88 percent, with the major part of the reduction occurring within one pore volume of emulsion injection.

These experiments were conducted at constant flow rate. Blocking effects at constant pressure (more similar to field conditions) would probably show more dramatic effect. In all of the experiments with injected emulsions, the effective permeability to water was decreased far more than an equivalent amount of residual oil would have reduced the permeability. The emulsion droplets are more efficient at reducing the effective permeability of a core than is the same amount of oil that is not emulsified.

Similar results were obtained when injecting an externally produced emulsion into a core which would be steamflooded. The results (table 4) show that the emulsion block (created with Wilmington oil) was stable at steamflood conditions. This experiment was conducted with a 25 in. core and saturated steam at 160° C. Before emulsion injection, permeabilities were measured before and after steam to make sure the steamflood itself did not cause a permeability reduction. A 77 percent reduction in permeability from external emulsion injection was observed under these steamflood conditions providing strong evidence for the development and utilization of this type of blocking procedure in the field.

Emulsion Injection into Cores Containing Residual Oil

These experiments were performed because of uncertainty about the effect of residual oil on an "emulsion block". In the case of residual oil remaining in the core, the effective permeability to water is much lower at the beginning of emulsion injection than with an oil-free core. The results are summarized in table 5.

In one experiment, emulsion injection was begun after waterflood and in the others emulsion injection was begun after tertiary recovery. The number of pore volumes of emulsion injected was 10, 7, and 8, respectively. The reductions in effective permeability, 52, 33, and 56 percent, were significant, but not as high as when using oil-free cores.

These experiments also illustrate a problem in performing corefloods in a one-dimensional coreflood apparatus - the situation of gravity override is difficult to simulate. The two-dimensional steamflood model now being installed at NIPER will allow a more realistic simulation of field steamfloods and the resultant channeling due to gravity override.

In Situ Emulsification

Coreflood experiments designed to cause permeability reductions by in situ creation of oil-in-water emulsions have been less successful than externally produced emulsions, but still show significant reductions in permeability. The data are summarized in table 6.

The first two tests were performed in the usual manner of saturating the core with Wilmington oil and then waterflooding to residual oil saturation, resulting in oil saturation of 45 and 49 percent, respectively, before caustic injection. After injection of caustic slugs, the effective permeabilities were reduced 27 and 25 percent, respectively.

In the third test, the core was not saturated with oil before waterflooding and the residual oil saturation was 34 percent before injection of caustic. Under this condition, the reduction in effective permeability increased to 43 percent. In all three tests, oil-in-water emulsions were produced from the core which had droplet size distributions appropriate to cause pore throat blockages. These three tests again illustrate that it is difficult to simulate in a one-dimensional model the conditions which exist in an actual reservoir after a steamflood, but that it is possible to create "emulsion blocks" in situ under appropriate conditions.

Another experiment was performed using Delaware-Childers oil and the dispersant mixture described previously. Sand (70 mesh) was packed into a 1.25 in. x 5 in. glass tube. The sandpack was placed in a vertical position and gravity flow (constant WP) was utilized to saturate with oil and waterflood to residual oil. The flow rate to water after waterflood was measured at 7.1 ml/minute.

After dispersant introduction, the flow rate was reduced to zero and would not resume until increased pressure caused the emulsion formed to be forced on through the sandpack. This experiment draws more attention to the probability that emulsion blocks will be more stable at constant pressure than at constant flow.

Conclusions

After creating foam with known foaming agents and contacting that foam with crude oils, the authors have observed emulsified droplets of oil within the liquid interfaces between the gas bubbles of the foam; however, we have not contributed (yet) to the question of whether "foam" blocking is really emulsion blocking.

We have shown that emulsions can be formed that are stable at higher temperatures, and survive on dilution with fresh water. They have the theoretically assumed drop sizes to block pores in a porous medium at elevated temperatures and in the presence of saturated steam. Emulsion blocking occurs also in the presence of residual oil. In situ formation of emulsions by injecting the emulsifying agent was shown to cause blocking, but further research is needed to increase its effectiveness and to prepare the technique for commercialization.

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TABLE 1. Emulsification of Wilmington Crude Oil with Caustic

NaOH(%)	Vol (ml.)		Apparent emulsion		Room temperature		4 hrs. at 110° C		8 days at 110° C	
	Oil	Aqueous	Upper	Lower	Upper	Lower	Upper	Lower	Upper	Lower
0.00	3.0	3.0	w/o	w	5.0	1.0	3.3	2.7	3.1	2.9
0.03	3.0	3.0	o/w	o/w	5.5	0.5	5.3	0.7	3.6	2.4
0.08	3.0	3.0	o/w	o/w	4.5	1.5	4.1	1.9	3.5	2.5
0.17	3.0	3.0	o/w	o/w	5.1	0.9	4.7	1.3	3.8	2.2
0.25	3.0	3.0	o/w	o/w	4.8	1.2	4.4	1.6	0.4	5.6
0.42	3.0	3.0	o/w	o/w	5.7	0.3	6.0	0.0	0.1	5.9
0.58	3.0	3.0	w/o	o/w	2.7	3.3	2.8	3.2	2.8	3.2
0.75	3.0	3.0	w/o	o/w	3.5	2.5	3.2	2.8	1.7	4.3
1.00	3.0	3.0	w/o	o/w	3.5	2.5	3.2	2.8	3.0	3.0

TABLE 2. - Emulsification of Asphaltic Crude Oils with Caustic

Oil	Viscosity (cps)	Wt. Percent		Acid Number	Optimal NaOH Concentration (%)
		S	N		
Wilmington 5G	175			2.04	.42
Wilmington B66099	370	1.73	.72	2.31	.18
Midway-Sunset B76067	650	1.05	.73	4.15	.42
Hasley Canyon B77023	>1100	5.5	.96	.68	None

TABLE 3. - Static Emulsification Tests Performed with Surfactants
and Delaware-Childers Oil

Petroleum Sulfonate	Average eq. wt.	Ethoxylated sulfonate	NaCl (%)	Volume, oil-in-water emulsion 110° C (16 hours elapsed) (ml)
Witco-40 (3.75%)	335	None	0	5.9
			2.12	6.0
			4.25	5.9
Witco-40 (3.75%)	335	SE-463 (2.5%)	0	6.0
			2.12	6.0
			4.25	6.0
PetroStep-420 (3.75%)	420	None	0	5.9
			2.12	6.2
			4.25	0
Petrostep-420 (3.75%)	420	SE-463 (2.5%)	0	6.0
			2.12	6.1
			4.25	6.9 (optimum)
Witco-18 (3.75%)	495	None	0	0
			2.12	0
			4.25	0
Witco-18 (3.75%)	495	SE-463 (2.5%)	0	6.5
			2.12	0
			4.25	0

TABLE 4. - Injection of Externally Produced Emulsion
into Oil-Free Cores

Temp. (°C)	Emulsion Injected (PV)	Permeability			Flow rate (cc/min)
		Absolute (md)	Final (md)	Reduction (%)	
<u>Delaware-Childers Oil (Light)</u>					
25	9.5	219	71	68.0	3.4
90	10.0	148	7	95.2	3.4
<u>Wilmington Oil (Heavy)</u>					
25	8.9	266	31	88.3	3.4
90	9.9	74	12	84.3	3.4
110	8.5	187	26	86.1	3.4
160	6.5	90	21	76.7	5.0

TABLE 5. - Injection of Externally Produced Emulsions into
Cores Containing Residual Oil
Delaware-Childers Oil - 25° C

Emulsion Injected (PV)	Permeability				Flow rate (cc/min)
	Absolute (md)	Before emulsion (md)	Final (md)	Reduction (%)	
10.0	285	33	16	52	3.4
7.0	324	26	18	33	3.4
8.0	297	84	38	56	3.3

TABLE 6. - In Situ Emulsion Formation
Wilmington Oil - 50° C

Emulsion Injected (PV)	Permeability				Emulsifier	Flow rate (cc/min)
	Absolute (md)	Before emulsion (md)	Final (md)	Reduction (%)		
0.45	325	45	33	27	0.55% NaOH	3.4
0.47	257	24	18	25	1.06% NaOH	3.4
0.83	1,400	88	50	43	1.06% NaOH	3.4

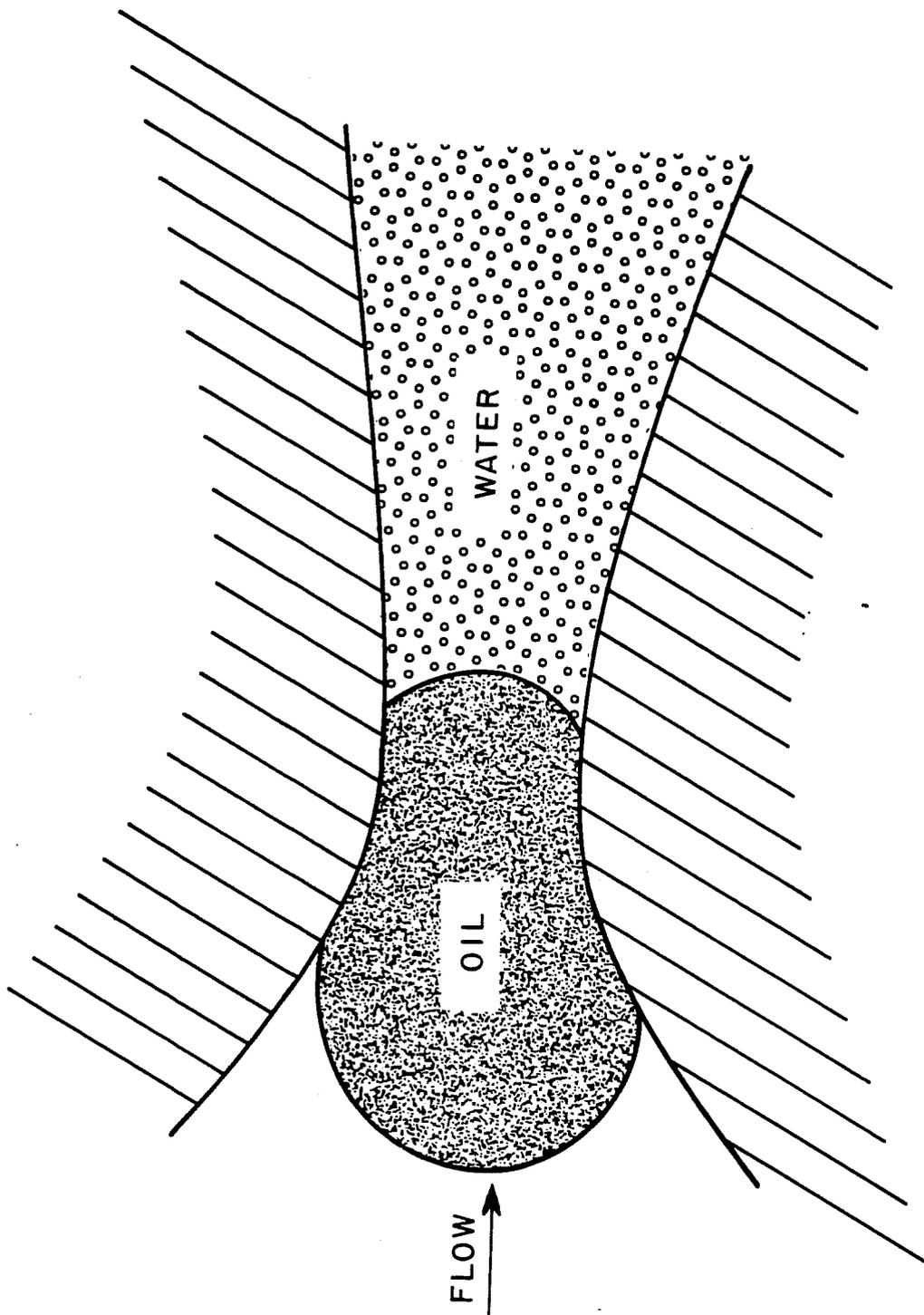


FIGURE 1. - Pore throat with oil droplet blocking water flow.

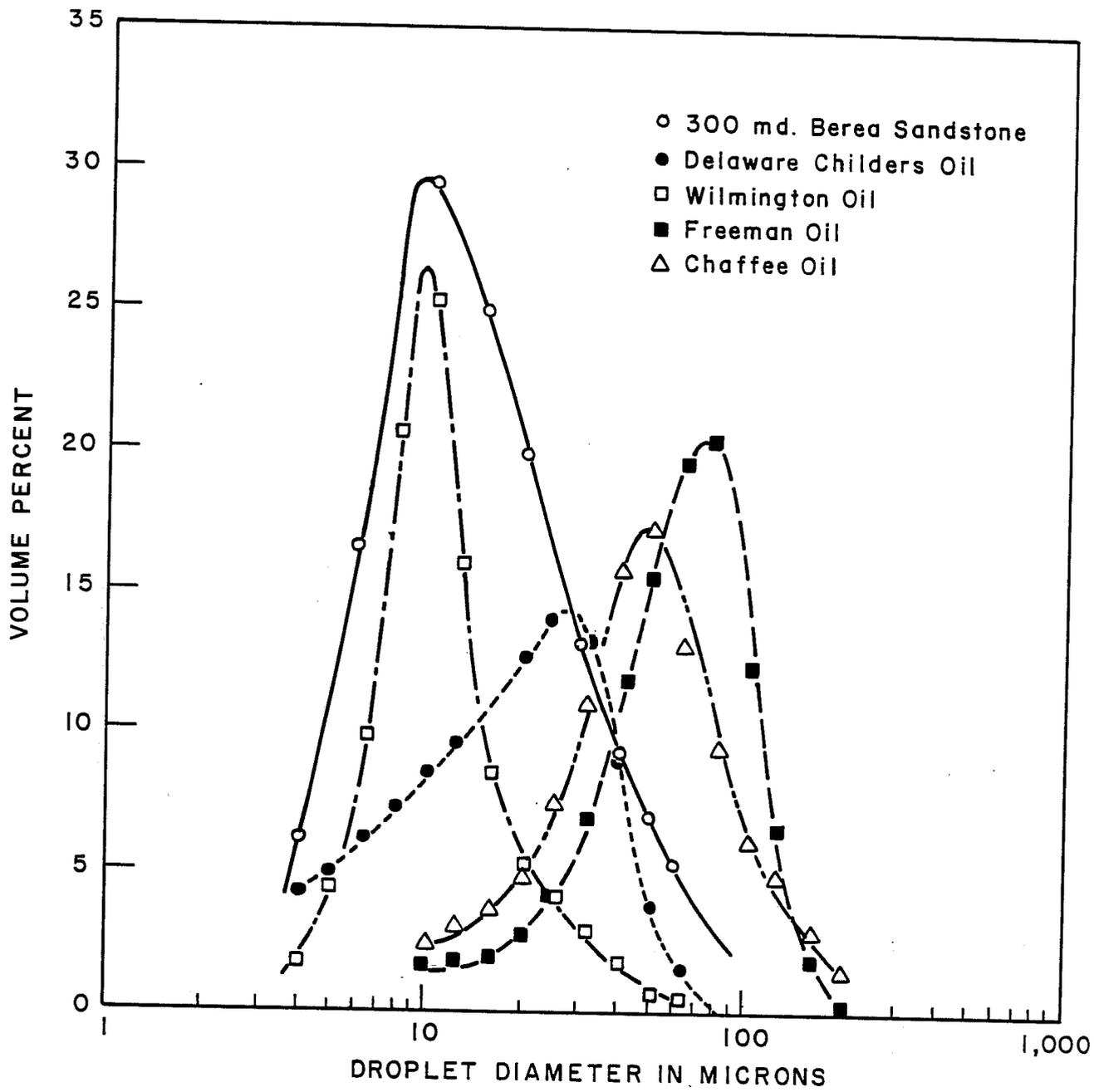


FIGURE 2. - Droplet size distribution for emulsions.

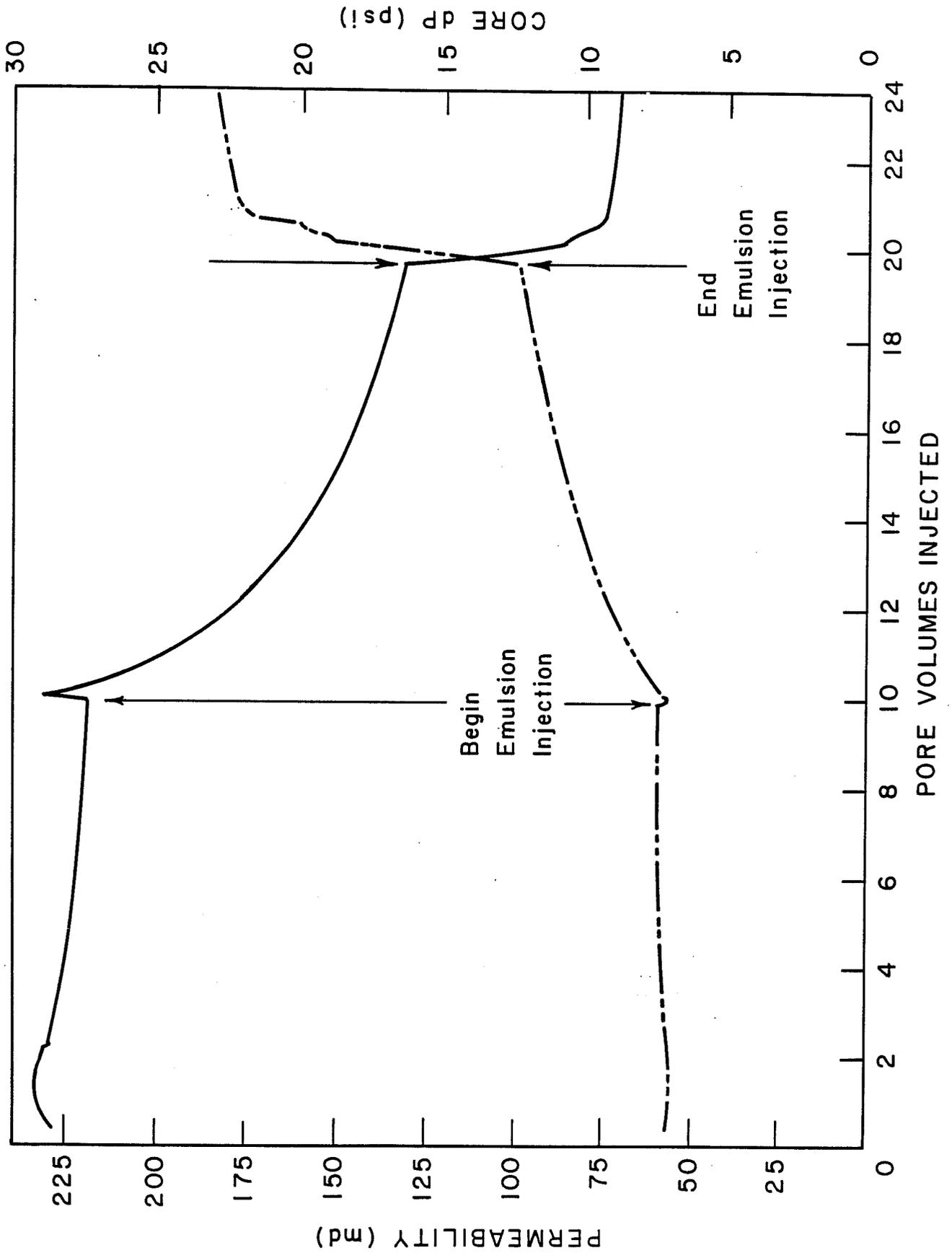


FIGURE 3. - Permeability reduction - 25° C Delaware-Childers oil.

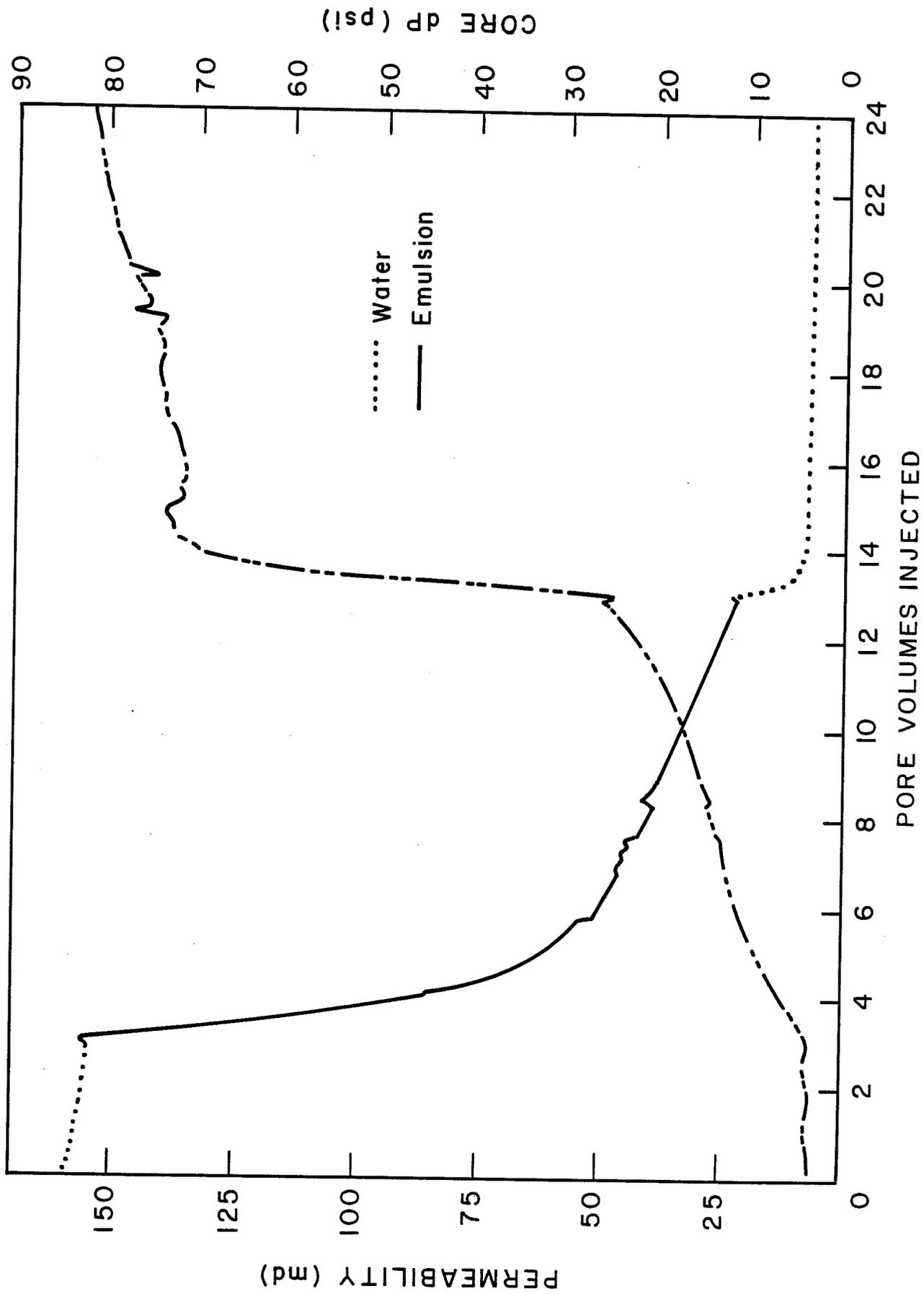


FIGURE 4. - Permeability reduction - 90° C Delaware-Childers oil.

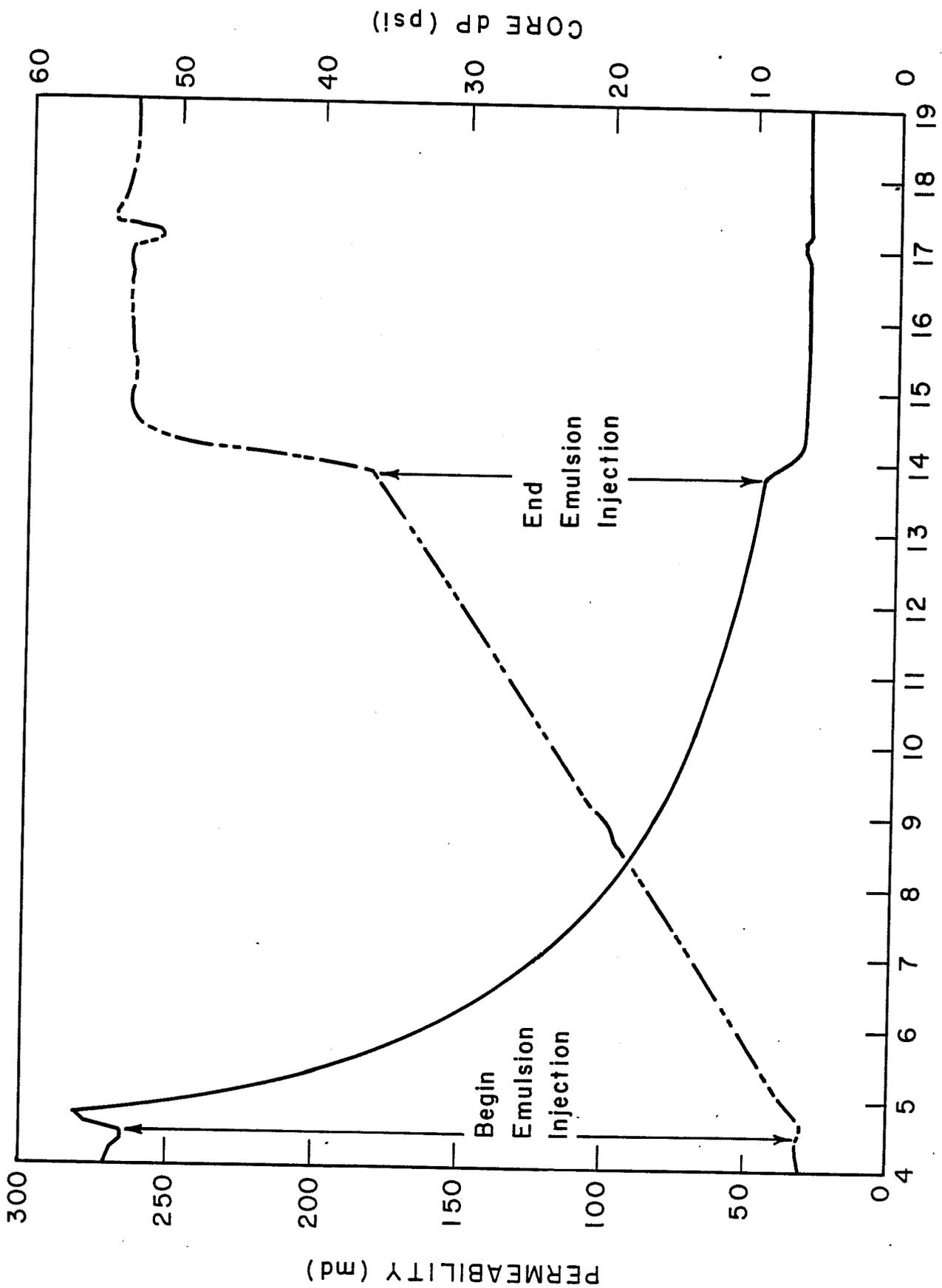


FIGURE 5. - Permeability reduction - 25° C Wilmington oil.

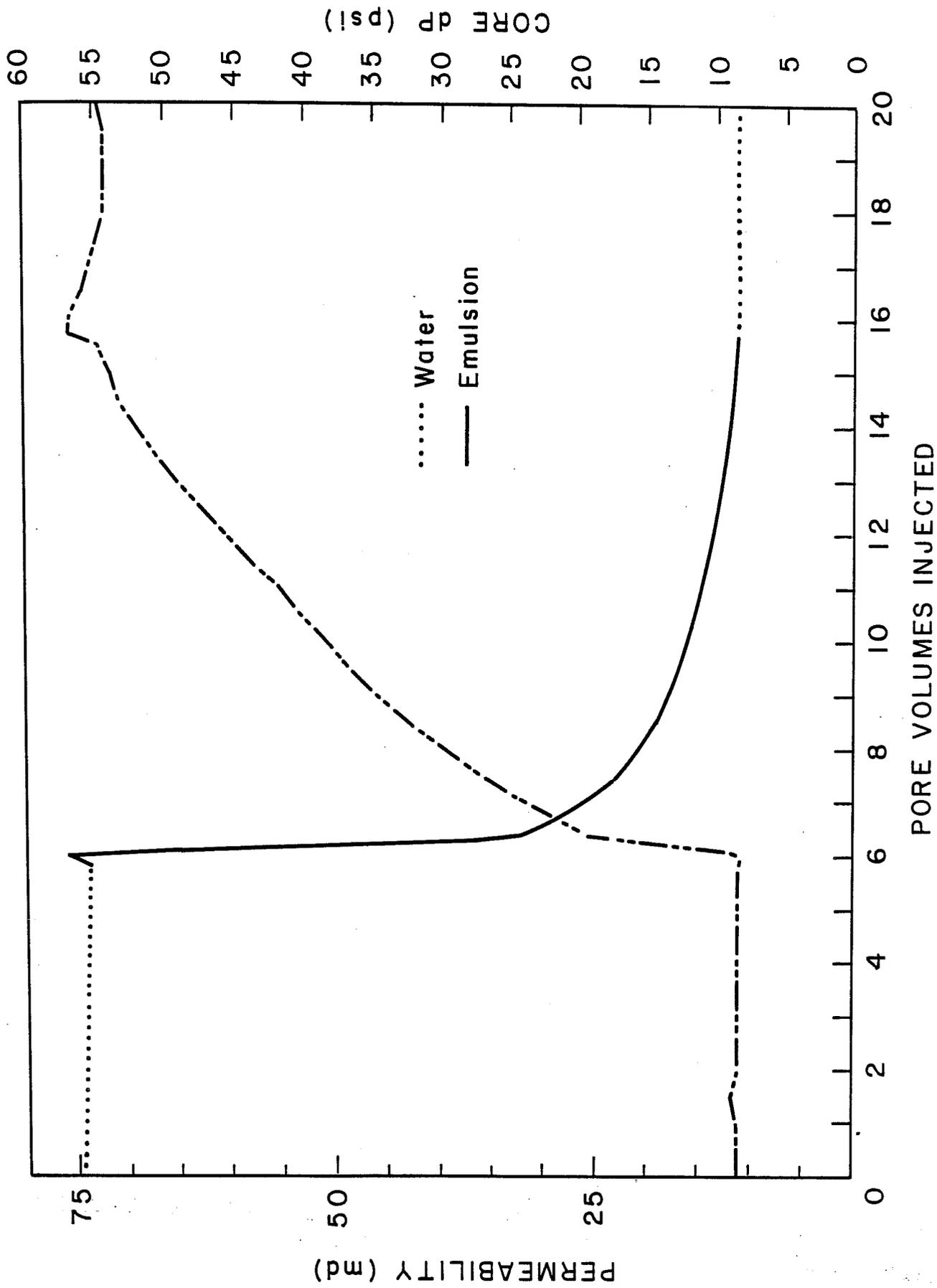


FIGURE 6. - Permeability reduction - 90° C Wilmington oil.

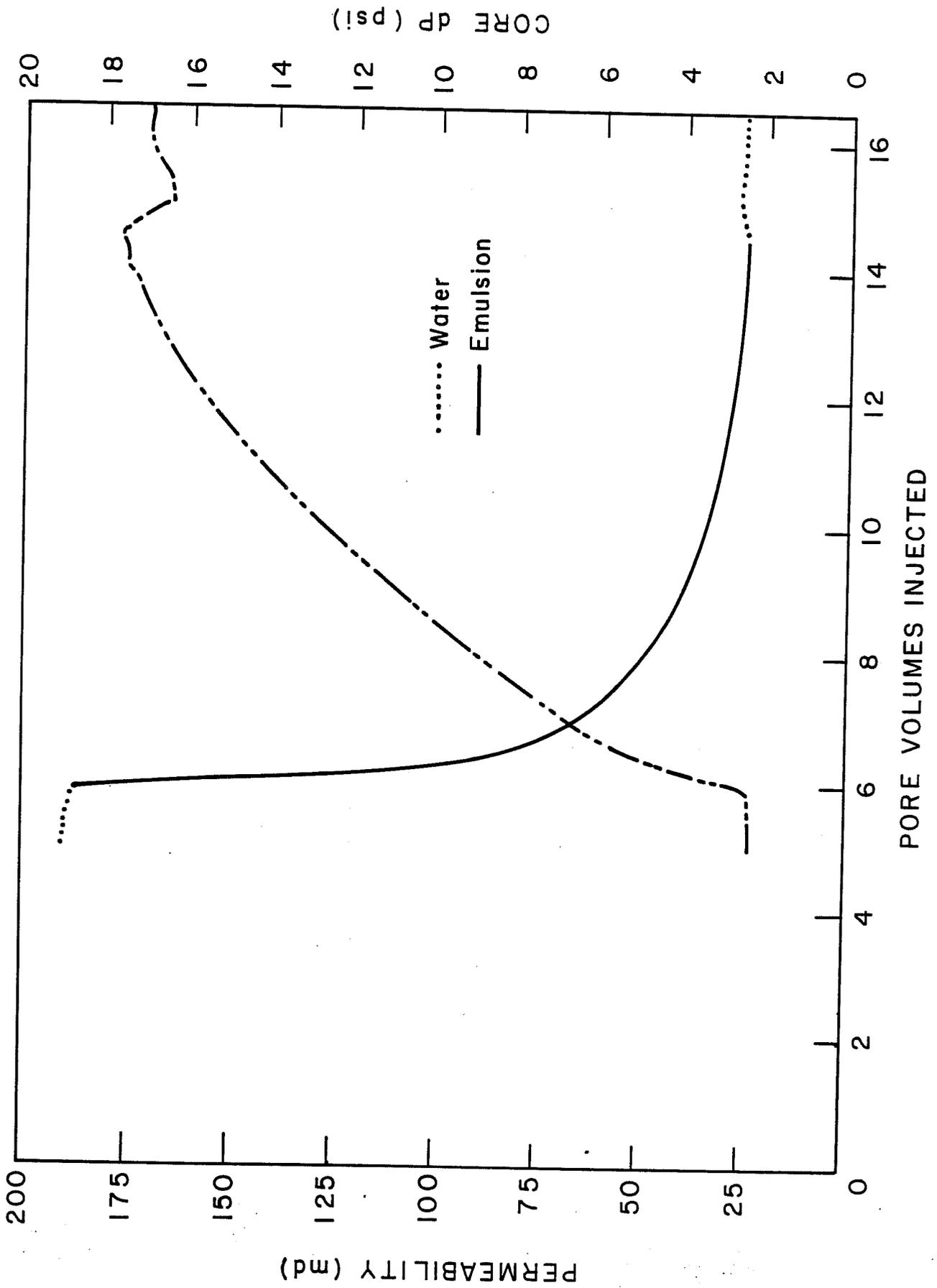


FIGURE 7. - Permeability reduction - 110° C Wilmington oil.