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REPORT

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DAMAGE TO POROUS MEDIA DUE TO THE DEGRADATION OF FLOCCULANTS

MASTER

PREPARED BY

DR. O. J. VETTER AND
DR. V. KANDARPA

VETTER RESEARCH
3189C AIRWAY AVE. - COSTA MESA - CALIFORNIA 92626

SUBMITTED TO

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Ottomar Vetter

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PREAMBLE

The United States Department of Energy, Division of Geothermal Energy (DOE/DGE) awarded Vetter Research (VR) a contract to perform research related to injection and reinjection problems in geothermal operations. This contract (No. DE-AC03- 79ET27146) is entitled: "Injection, Injectivity and Injectibility". The present report is one of the deliverables under this contract and it deals with the subject of the damage to porous formation by the degradation of flocculants. The report summarizes the results of flow studies involving the flow of some commercial organic flocculant solutions through sandstone cores at high temperatures.

1.0 ABSTRACT

The heat-depleted brines from high temperature geothermal reservoirs must be treated to remove particles prior to reinjection. Filtration and/or sedimentation with and without chemical aids such as flocculants is considered to be the most suitable method for this geothermal application. Most of the flocculants suitable as an additives to aid the particle removal are organic chemicals. By nature, they tend to degrade to carbonaceous material. Thus, it is conceivable that the degradation residues from the flocculants can enter the porous formation and can cause reduction in permeability. This in turn can reduce the injectivity of the formation rock. In addition, a change of the rock wettability (contact angle) through adsorbing flocculants may also cause a reduction of reservoir injectivity.

The present report summarizes the results of laboratory experiments involving the flow of two commercial flocculant solutions through Berea sandstone core plugs at 225° C. The two flocculants used are TFL-398 and TFL-362 (commercial flocculants supplied by Tretolite Division of Petrolite Corporation). The flow of brine containing these flocculants through these sandstone cores resulted in considerable permeability reductions during the flow experiments even when used at concentrations of only 5 ppm. The reduction in permeability is attributed to the plugging of pore spaces by the degradation products.

The results of these experiments demonstrated that the use of a flocculant can cause severe formation damage. It is, therefore recommended, to always conduct laboratory tests prior to the use of any specific flocculant for geothermal applications to avoid any damage to injection wells.

2.0 SUMMARY AND CONCLUSIONS

1. Prior to the brine reinjection, the heat-depleted brines have to be treated to remove potentially damaging materials. In particular, suspended solids must be removed from the brines to be reinjected to avoid severe damage to the injection wells and reservoirs.

2. Particle removal prior to reinjection through filtration and/or sedimentation process with and without chemical aids such as flocculants is potentially the most suitable method for a problem-free geothermal reinjection operation.

3. Geothermal brines are normally self-floccing, but the process is rather slow. There seems to be an upper temperature for the self-floccing process. For example, the Mercer 2 brines did not self-floc above approximately 92° C.

4. Flocculants added to the geothermal brine may (a) increase the floccing temperature and (b) improve the floccing process at any temperature.

5. An improvement of the floccing process by (a) increasing the maximum floccing temperature and (b) increasing the flocculation rate at any temperature provides many economical and technical incentives for an operator of a high temperature geothermal field.

6. The desired improvements of the flocculating process could be achieved by the addition of flocculants to the heat-depleted brine upstream of the required particle removal.

7. The major benefit of this chemically induced or aided flocculating process would be a drastically decreased size of the solid removal facilities and with that a drastically decreased investment cost for the required solid removal facilities.

8. Apparently, some commercial flocculants have been successfully used for increasing the rate of formation of floccs (i.e., accelerated the self-floccing) in geothermal operations. However, these flocculants were not successful in increasing the effective flocculant temperature.

9. Flocculants added to the heat-depleted geothermal brine may not only show benefits but may also cause detrimental effects for a given geothermal field operation.

10. The major disadvantage of a flocculant additive may be a slow reduction of the injectivity of the geothermal injection wells.

11. An injectivity reduction of the injection wells through the use of flocculants can be caused by two mechanisms.

12. Most of the organic chemicals degrade to carbonaceous materials at the high temperatures encountered in geothermal environment. Therefore, it might be suspected that the solid degradation products can damage porous formation by plugging of the pores.

13. There is another possible mechanism by which the flocculants may cause damage to the formation. This damage is through change in wettability of the rock matrix due to adsorbed flocculant on the rock surface. This wettability change can decrease the relative rock permeability and, thus, can decrease the injectivity.

14. Some laboratory experiments are performed by degrading two commercial flocculants, TFL-398 and TFL-362 in Berea sandstone cores. These experiments showed that a severe damage to the permeability of a clean porous medium can be created by the flocculant addition to the brine.

15. The damage caused by the injection of brine containing flocculant is not completely eliminated by simple back flushing.

16. TFL-362 flocculant caused more damage than TFL-398 flocculant. This demonstrates that different flocculants can result in different degrees of damage to porous media.

3.0 RECOMMENDATIONS

1. The results of the experiments involving flocculant degradation in porous media clearly demonstrated that they can cause formation damage. Thus, specific potential flocculants suitable for a given brine should be tested through core flow tests in the laboratory prior to their use in the field.

2. The optimum quantity of any specific flocculant needed for a given brine to effectively remove the particles should be determined to avoid excess flocculants from entering the formation.

3. Effective analytical methods should be developed to determine low concentrations of flocculants in brines. This will help in properly monitoring the brines for the presence of excess concentrations of flocculants prior to their entrance into the injection wells.

4. Radioactively labelled flocculants can be used for laboratory and field studies to overcome the analytical sensitivity problems normally encountered in this type of work.

4.0 INTRODUCTION

The need to reinject the heat-depleted brines from geothermal operations and the problems associated with this reinjection have been discussed previously [1]. Prior to the brine reinjection, the heat-depleted geothermal brines have to be treated to remove potentially damaging solid materials. In particular, suspended solids must be removed from the brines to be reinjected to avoid severe damage to the injection wells and reservoirs.

Various treatment methods for geothermal brines have been discussed in the literature [2 through 10]. One of the main constituents of the geothermal brines which can cause injection problems is silica. Considerable attention has been paid by various investigators to the removal of silica and other suspended particles from brines [6,7,11 through 16]. In spite of the extensive number of methods available for water treatment, the actual treatment of a geothermal brine from a specific resource would require testing prior to its reinjection [1,13,15]. Some unforeseen precipitation and reinjection problems often arise as experienced during Mercer 2 well test in the Imperial Valley, California [15] and can create various types and degrees of well and reservoir damage. Such problems would require site specific testing in the laboratory and in the field.

It has been the experience of many investigators (including that of the authors) that particle removal prior to reinjection through filtration and/or sedimentation with and without chemical aids such as flocculants is the potentially most suitable method for a problem-free geothermal reinjection operation [13,17 through 20]. The purpose of flocculant additions is to aid in the formation and growth of particle agglomerates of such size and density that effective solid-liquid separation can be accomplished. Some recent field studies [21] have claimed great success in forming large flocs of silica through use of organic chemical flocculants and the subsequent removal of the floccs from the brine.

It has been outlined in previous reports [15,26] that the geothermal brines are normally self-floccing, thus creating a better chance to effectively utilize the gravity forces in particle sedimendation. These gravity forces acting on the relatively large floccs provide the basis for the required particle removal prior to reinjection through settling tanks or through the settling (or clarification) zone of a reactor/clarification system [13].

Unfortunately, the natural flocculation (self-flocculation) process is temperature dependent [26,27] and rather time consuming. For example, the Mercer 2 brine [26] will not self-flocculate within a reasonable time above 92° C. Even below 92° C, the self-flocculation as determined by rather complex particle characterizations in the field [26,27] will proceed fairly slowly, thus requiring rather large and expensive brine treatment facilities (e.g., a reactor/clarification system).

It was hoped that aiding the self-flocculating tendency of a geothermal brine by the addition of flocculants will not only increase the reaction rate of the flocculation process (i.e., by decreasing the required flocculation time) but may also aid the flocculation at higher temperatures by starting the flocculation process at temperatures above the self-flocculation temperature.

The first flocculating experiments by the authors were performed during some field test work in the Imperial Valley, California [14]. These experiments were rather successful as far as an improvement of the flocculation reaction rate is concerned. However, these experiments were unsuccessful regarding an increase of the effective flocculation temperature through flocculants above the self-flocculating temperature.

5.0 SUSPECTED DAMAGE MECHANISM THROUGH FLOCCULANT UTILIZATION

The damage mechanism through flocculant utilization is not quite clear. It was suspected that there exists at least two different damage mechanisms:

1. The flocculant will get adsorbed on the internal solid surface of the rock matrix thus creating detrimental changes in the rock wettability (contact angle). This, in turn, could have a considerable and detrimental impact on the permeability and with that on the injectivity.

2. The flocculant may undergo a chemically and/or physically induced degradation process and the solid degradation products may cause a plugging of the pores within the near wellbore region of the reservoir.

Both mechanisms could result in a permeability decrease of the rock matrix close to the injection wellbore (skin effect). Both potentially exisiting mechanisms are described briefly in the following two sections.

5.1 DAMAGE MECHANISM THROUGH FLOCCULANT ADSORPTION

Flocculants, by design, have a rather strong tendency to adsorb on solid surfaces. The theoretical relationship between flocculation and adsorption is not quite clear. However, most effective organic flocculants will strongly get adsorbed on the surface of rock materials. The adsorbed flocculant will change the contact angle on the solid face, thus possibly creating a large and detrimental effect on the relative permeabillity and, with that, on the well injectivity.

5.2 DAMAGE MECHANISM THROUGH FLOCCULANT DEGRADATION

Most of the organic chemicals degrade at high temperatures encountered in geothermal reservoirs. The degradation products (usually carbonaceous residues) can conceivably cause damage to the porous formation [22,23]. Thus , it is conceivable that excess amounts of flocculant chemicals can actually enter the porous reservoir rock and can result in a near well-bore damage of the injection wells.

A literature search has been made to find information related to the effect of flocculant degradation on the permeability of a porous media with little success.

6.0 OBJECTIVES OF THE REPORT

The present work is aimed at determining if addition of flocculants to a geothermal brine can damage the porous media and if so to what extent. The present report is a summary of the results of the laboratory experiments involving the flow of two commercial flocculant [21,24] solutions through Berea sandstone cores.

In order to pursue the main objective of this study, the following items are treated in this report:

1. Description of laboratory flow experiments to study the effect of flocculant addition on the permeability of porous cores.
2. The results of these laboratory experiments on the flow of two commercial flocculants through Berea sandstone cores at high temperature.

7.0 EXPERIMENTAL APPARATUS AND PROCEDURES

As mentioned in section 6.0, the main objective of the present work is to determine if the addition of flocculants to geothermal brines can cause damage to the porous media. To accomplish this objective some flow experiments are conducted in the laboratory. The materials, apparatus and procedures used for these experiments are described in this section.

7.1 MATERIALS USED

A recent study on the clarification of geothermal fluids using organic flocculants by Kochelek and Zenty [21] claimed the successful use of two commercial flocculants (TFL-362 and TFL-410), manufactured by the Tretolite Division of Petrolite Corporation). One of these flocculants (TF-362) and another (TFL-398) are used in the present laboratory study. Both of these flocculants are supplied by Tretolite [24]. Both of them are used at concentrations of 5 ppm and are mixed in deionized water. As a comparison, the flocculants in the field study were used at concentrations of 3 ppm [21].

To represent the porous media, Berea sandstone core plugs (1 inch diameter and 1.5 in length) are used. To avoid any damage by the swelling or dispersion of the clays, the core plugs are heated at 450°C for 4 hours to stabilize the clays [25]. The materials used in the present study are summarized in Table 1.

7.2 APPARATUS

The experimental apparatus used in the flow experiments of the present study is schematically shown in Figure 1. The main part of the apparatus consists of the Hassler sleeve core holder, which is mounted in a high temperature oil bath. The core itself is tied to two metal ends of a core holder (for a more detailed description of the core holder see reference [25]). The overburden pressure to the core is supplied by water which itself is pressurized by nitrogen gas from a nitrogen tank [25]. The flocculant solutions are stored in a flocculant tank. The flocculant solution is pushed into the core plugs by displacement using kerosene which itself is displaced by nitrogen gas from a gas tank. A back pressure (450 psi) in excess of the equilibrium water vapor pressure corresponding to the temperature used (225° C) was applied. The inlet and outlet pressures of the core are monitored by a differential pressure transducer. In addition, the inlet core pressure was monitored by a pressure transducer.

7.3 PROCEDURE

The major experimental procedures in the present work are described in this section.

7.3.1 CORE PREPARATION AND PERMEABILITY MEASUREMENT

Core plugs are cut to 1.0 inch diameter and 1.5 inches long from Berea sandstone blocks. The core plugs are fired at 450° C for 4 hours to desynthesize the clays. A few initial flow tests with deionized water without the firing of the clays showed substantial variations in permeability with passage of deionized water through the unfired cores. After measuring the pososity, of the fired cores, a dry core is tied up to the two metal ends of a core holder [25]. This is accomplished, first, with a teflon tape , and then with a teflon shrink tube sleeve. The annulus of the core holder is filled with water and pressurized to about 1000 psi to simulate the overburden pressure. The core is evacuated for 1.0 hour (from the outlet end of the core holder) and then saturated with deaerated (under a pressure of 435 psi) and deionized water. Once the core is saturated, the base permeability of the core is measured at room temperature. After the high temperature flow of brine containing the flocculant through the core the oil bath is cooled to room temperature and, the permeability measurements are repeated.

7.3.2 PREPARATION OF FLOCCULANT SOLUTIONS

Both the flocculant solutions are prepared by mixing 5 ml of each of the flocculants (TFL-398 and TFL-362) in deionized water to make 1000 ml of the mixture and mixed at low speed in a laboratory blender. The flocculant solution is poured into the flocculant solution tank (see Figure 1).

7.3.3 FLOCCULANT FLOW EXPERIMENTS

Initially, the flow experiments are conducted by continuously pumping (at a flow rate of 0.3 ml/min) the flocculant solutions through the core (at 225° C) and monitoring the inlet pressure. These tests showed that the core permeability is decreasing slowly as more and more of the solution passes through the core. This is attributed to the dissolution of silica from the core material in the deionized water at 225° C. This type of procedure interfered with the main objective of the experiment, namely, the study of the core damage caused by flocculant addition to the brine. Therefore, this procedure was discontinued and an alternate procedure was followed.

The alternate procedure consists of saturating the core with the flocculant and measuring the permeability of the core at room temperature. The details of this procedure are given below. After the base permeability of the cores is measured, the back pressure regulator valve is set at 440 psi and the oil bath is heated to 225° C. Three (3) pore volumes of the flocculant (TFL-398) are injected into the core. This assures that the initially water saturated core is replaced by flocculant solution. The flocculant is allowed to degrade or to adsorb for 90 minutes. Then an additional three pore volumes of the flocculant (TFL-398) are injected into the core and allowed to degrade for 90 minutes. This process is repeated for 3 more times. The oil bath is allowed to cool to room temperature. The permeability of the core is measured using deionized water tank. At the end of the test, the core is back flushed by injecting deionized water and the permeability measurements are repeated. The complete flocculant degradation and adsorption experiments are repeated with a second flocculant, TFL-362, using the procedure similar to the one described above.

At the end of the tests, the cores from the two experiments are taken out of the core holder and dried. After examining the inlet surface of the cores with optical microscope to detect evidence of any damaging material, the cores were broken transversley. The broken surface was examined using optical microscope and scanning electron microscope (SEM) to detect evidence of damaging material. Damaging solids could not be detected by either observation.

8.0 RESULTS

The results of the experiments on flocculant degradation are shown in Tables 2 through 8. Tables 2 and 5 show the initial permeability measurements on core No.1 and 2, respectively. Tables 3 and 6 show the permeability measurements of the cores after the degradation of the flocculants TFL-398 and TFL-362, respectively. Tables 4 and 7 show the permeability measurements after back flushing the damaged cores. The results of all the tables are summarized in Table 8.

From Table 8, it is evident that core No.1 has an initial room temperature permeability of 89.6 md. After injecting the flocculant solution (TFL-398) into the core, its room temperature permeability decreased to 16.3 md, a decrease of about 82%. Back flushing the core with deionized water increased the permeability somewhat, but the original permeability could not be recovered. The final permeability of the core is still only 40% of the original permeability.

Table 8 also gives the results on core No.2 and TFL-362. The initial room temperature permeability of core No.2 is 76.2 md. After injecting the flocculant solution, TFL-362, its permeability decreased to a value of 1.0 md. This is a substantial decrease (a decrease of 99%). Back flushing the core increased the damaged permeability only slightly. The final permeability of core No.2 is still only 7% of the original permeability.

Thus, the results of this laboratory experiment clearly demonstrated the following:

1. The high temperature injection of organic flocculant solutions can cause damage to porous media.
2. TFL-362 flocculant caused more damage than TFL-398 flocculant.
3. The damage caused by the degradation products are not completely eliminated by back flushing.

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TABLE 1

SUMMARY OF THE MATERIALS USED IN STUDYING DAMAGE TO POROUS MEDIA BY FLOCCULANT DEGRADATION

1. FLOCCULANTS USED

A) TFL-362
B) TFL-398

BOTH SUPPLIED BY TRETOLITE
CONCENTRATIONS = 5 mg/l OF DI WATER

2. POROUS PLUGS USED

BEREA SANDSTONE CORE PLUGS (NO.1 & NO.2)
HEATED AT 450° C FOR 4 HOURS

3. CHARACTERISTICS OF THE CORES

A) CORE NO. 1 (USED WITH TFL-398)

DIAMETER = 2.54 cm
LENGTH = 3.90 cm
POROSITY = 20%
PORE VOLUME = 3.95 cc
AIR PERMEABILITY = 181 md

B) CORE NO. 2 (USED WITH TFL-362)

DIAMETER = 2.54 cm
LENGTH = 3.73 cm
POROSITY = 20%
PORE VOLUME = 3.78 cc
AIR PERMEABILITY = 135 md

TABLE 2

INITIAL ROOM TEMPERATURE PERMEABILITY MEASUREMENTS (CORE NO. 1)

NO.	DIFF. PRESSURE (psi)	MASS OF WATER COLLECTED (gm)	TIME (sec)	FLOW RATE ml/sec	PERMEABILITY (md)
1	11	8.628	89.45	0.096	91.5
2	11	10.981	119.82	0.091	87.0
3	11	9.886	105.45	0.094	89.0
4	11	9.044	94.70	0.095	90.6
5	11	8.490	90.08	0.094	89.4
6	11	9.073	95.14	0.095	90.5
7	11	8.511	90.30	0.094	89.4

TABLE 3

ROOM TEMPERATURE PERMEABILITY MEASUREMENTS AFTER DAMAGE BY FLOCCULANT (TFL-398) DEGRADATION

(CORE NO. 1)

NO.	DIFF. PRESSURE (psi)	MASS OF WATER COLLECTED (gm)	TIME (sec)	FLOW RATE ml/sec	PERMEABILITY (md)
1	125	8.611	50.54	0.171	14.6
2	125	10.023	55.06	0.182	15.6
3	125	11.642	59.51	0.196	16.8
4	125	11.966	59.60	0.201	17.2
5	125	11.057	54.67	0.203	17.4

TABLE 4

ROOM TEMPERATURE PERMEABILITY MEASUREMENTS AFTER BACK FLUSHING THE DAMAGED CORE
(CORE NO. 1)

NO.	DIFF. PRESSURE (psi)	MASS OF WATER COLLECTED (gm)	TIME (sec)	FLOW RATE ml/sec	PERMEABILITY (md)
1	30	11.616	109.31	0.106	39.3
2	30	8.764	87.55	0.100	36.7
3	30	7.285	77.36	0.094	34.5
4	30	10.041	113.05	0.089	32.6

TABLE 5

INITIAL ROOM TEMPERATURE PERMEABILITY MEASUREMENTS (CORE NO. 2)

NO.	DIFF. PRESSURE (psi)	MASS OF WATER COLLECTED (gm)	TIME (sec)	FLOW RATE ml/sec	PERMEABILITY (md)
1	11	7.377	90.35	0.081	74.7
2	11	7.507	90.29	0.083	76.1
3	11	7.514	89.30	0.084	77.0
4	11	7.606	90.37	0.084	77.0

TABLE 6

ROOM TEMPERATURE PERMEABILITY MEASUREMENTS AFTER DAMAGE BY FLOCCULANT (TFL-362) DEGRADATION
(CORE NO. 2)

NO.	DIFF. PRESSURE (psi)	MASS OF WATER COLLECTED (gm)	TIME (sec)	FLOW RATE ml/sec	PERMEABILITY (md)
1	200	3.024	182.06	0.017	0.86
2	200	2.582	140.67	0.018	0.95
3	200	3.075	152.36	0.020	1.0
4	200	3.251	151.35	0.022	1.1
5	200	2.793	126.57	0.022	1.1

TABLE 7

ROOM TEMPERATURE PERMEABILITY MEASUREMENTS AFTER BACK FLUSHING THE DAMAGED CORE

(CORE NO. 2)

NO.	DIFF. PRESSURE (psi)	MASS OF WATER COLLECTED (gm)	TIME (sec)	FLOW RATE ml/sec	PERMEABILITY (md)
1	200	11.116	120.48	0.092	4.8
2	200	15.378	156.57	0.098	5.3
3	200	7.099	68.37	0.104	5.4

TABLE 8

SUMMARY OF THE RESULTS ON FLOCCULANTS DEGRADATION EXPERIMENTS

FLOCCULANT	CORE NO.	PERMABILITY (MD)		
		INITIAL	AFTER FLOCCULANT FLOW AT 225° C	AFTER BACK FLUSH
TFL-398	1	89.6	16.3	35.8
TFL-362	2	76.2	1.0	5.2

SCHEMATIC DIAGRAM OF THE FLOW APPARATUS

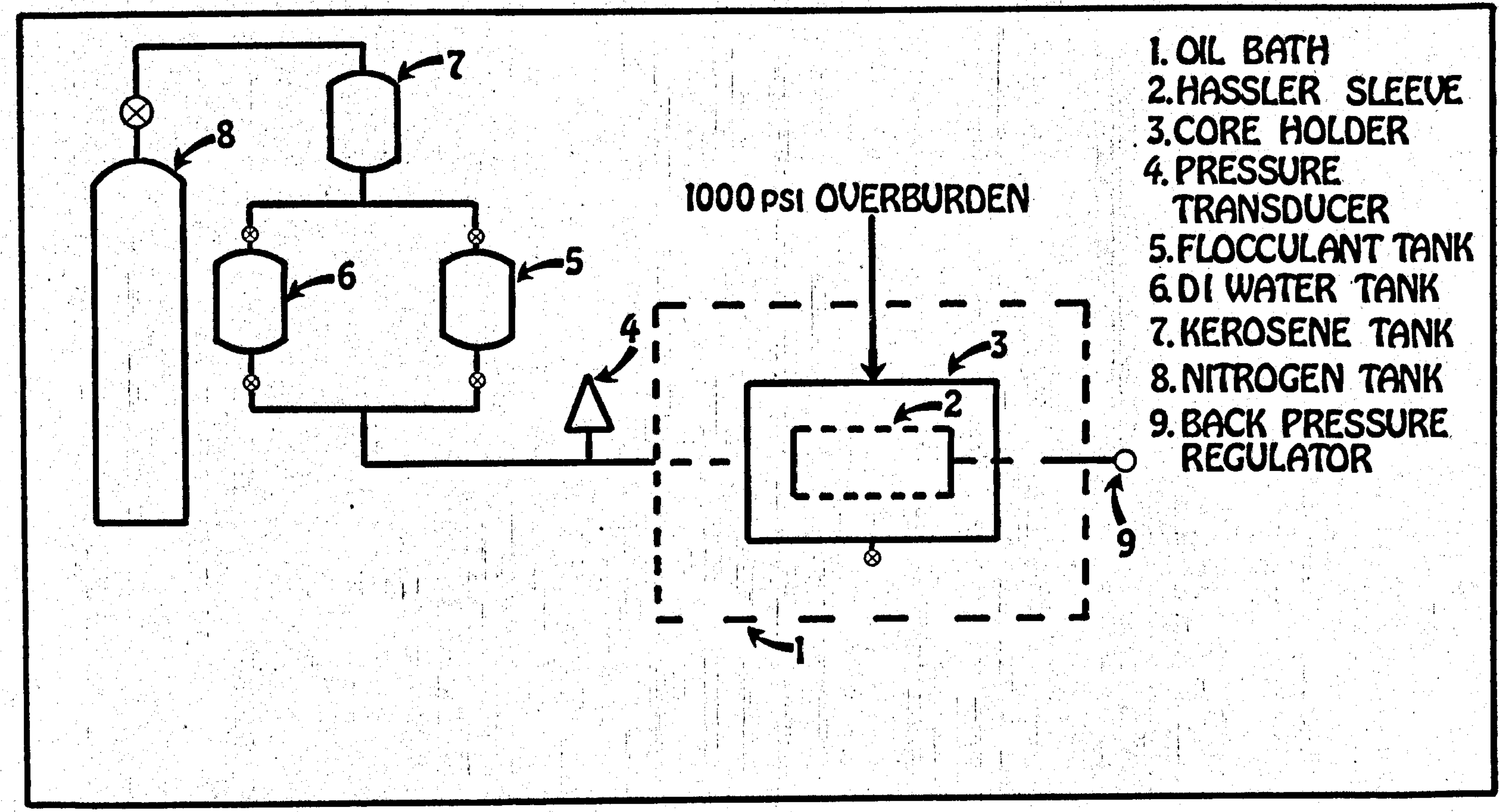

FIGURE 1