

INSTITUT FRANCAIS DU PETROLE

Direction de Recherche Gisements

CONFIDENTIAL

WETTABILITY STUDY OF SAMPLES FROM WELL 34/10-3
(STATOIL)

L. CUIEC

1 2 MARS 1980

**REGISTRERT
OLJEDIREKTORATET**

Project : B 77/79 012

number of copies : 20

LC/JT

February 1980

TABLE OF CONTENTS

ABSTRACT AND CONCLUSIONS

1 - FOREWORD

2 - DESCRIPTION OF POROUS MEDIUM AND FLUIDS USED

2.1.- Porous medium

2.2.- Fluids

3 - EVALUATION OF SURFACE PROPERTIES OF SAMPLES FROM WELL 34/10-3 UPON RECEPTION

4 - STUDY OF CLEANING OF SAMPLES FROM WELL 34/10-3

5 - EVALUATION OF THE WETTABILITY OF RESTORED SAMPLES, i.e. AFTER CLEANING,
SATURATION WITH RESERVOIR FLUIDS AND AGING

6 - CONCLUSIONS AND RECOMMENDATIONS

APPENDIX I - DESCRIPTION OF THE WETTABILITY TEST

APPENDIX II - PREPARATION OF SAMPLES

MICROPHOTOGRAPHS

TABLES

FIGURES

WETTABILITY STUDY OF SAMPLES FROM WELL 34/10-3

(STATOIL)

ABSTRACT AND CONCLUSIONS

This note is the final report on the first step of the research project concerning samples from well 34/10-3 (Statoil).

This first step deals with determining their wettability by the I.F.P. procedure on enough samples to establish a trend in the preserved, cleaned and "aged" state.

This study was made with porous medium and storage-tank oil from well 34/10-3 and synthetic water.

The following problems were examined :

- evaluation of surface properties of samples from the reservoir considered as they were when they reached us;
- study of the cleaning procedure of these samples;
- evaluation of the wettability of restored samples, i.e. after cleaning, saturation with reservoir fluids and aging.

This study shows that :

- the wettability of the samples upon reception is clearly water preferential for rock of the "ness" and "etive" types. We did not analyze the rock of the "rannoch" type. However it has a very good chance of also being water wet.
- no matter what procedure is used, the cleaning of these samples has little or no influence on the preceding property.

- after "restoration" the rock remains water wet, and the result obtained is close to that obtained upon reception.
- the rock has a very good chance of being water wet in situ.
- with regard to the surface properties, the samples received may be considered as sufficiently representative.
- if we have to respect the "initial saturations" parameter, it is preferable to use the restoration procedure described at the end of the present report.

WETTABILITY STUDY OF SAMPLES FROM WELL 34/10-3
(STATOIL)

I - FOREWORD

The surface properties of a porous medium, in particular the preferential affinity of its solid surface for water or oil, have considerable influence on the behavior of a rock/fluid system during experiments such as the displacement of one fluid by another, the determination of relative permeability curves, etc.

Therefore, when performing such experiments in the laboratory for any given reservoir, it is of prime importance to use a rock/fluid system that is as representative as possible of the one existing in situ.

When unpreserved core samples are available, modifications of the surface state may have occurred, with mud filtrate penetration, during contact with the atmosphere (oxidation), drying and handling. Even when "preserved" rock samples are used, the representativity of the surface state is doubtful. Indeed, it is not possible to prevent a temperature and pressure drop when the sample is being brought up. This drop may change the distribution of fluids on the surface of pores and be accompanied by the deposition of asphaltenes or other heavy fractions. Moreover a modification of the initial surface properties may occur with mud products or with oxidation in the atmosphere before the samples can be preserved by packing. Consequently there is always a risk in assuming that preserved cores have a representative surface state, and unfortunately it is not possible, a priori to estimate the extent of the modification of the surface properties of the rock during and after coring.

This evaluation may be made by the following three steps :

- 1 - Evaluation of the wettability of available samples from the reservoir.
- 2 - Working out a cleaning procedure leading to a rock which is as water-wet as possible. The role of this cleaning procedure is the extraction of a maximum of adsorbed compounds, whether they come from crude oil or from mud. It is not possible to separate compounds which were adsorbed in situ from those fixed afterwards.

3 - Evaluation of the wettability of samples after "restoration" of the original surface state. This means, after cleaning, saturation with reservoir fluids and aging under reservoir temperature and pressure conditions during the time required for the adsorption equilibria to be correctly established.

Comparison of results obtained in steps 1 and 3 makes possible the evaluation of modifications which took place in surface properties during and after coring.

These modifications may appear of so little importance that we can recommend the utilization of received samples without any restoration procedure. But in other cases the modifications appear to be very great, and the samples need to have their original surface state restored.

For samples from well 34/10-3, the study was performed in this way.

The wettability, or preferential affinity of the rock for water or oil was evaluated by a wettability test based on spontaneous and forced displacement experiments. This is described in Appendix I.

Before taking up the previous three points, let us look at the porous medium and fluids used.

2 - DESCRIPTION OF POROUS MEDIUM AND FLUIDS USED

2.1.- Porous Medium

The core fragments arrived "preserved" from drying and oxidation from the atmosphere.

From each piece of reservoir rock we received of the "ness" or "etive" type we cut one sample in the horizontal direction. The sample was always cut at the top end of each piece of rock.

The cutting out of samples was performed as described in Appendix 2.

Table 1 gives the list of the prepared samples.

For the rock of the "rannoch" type, at the time of the present investigation it was not possible to prepare consolidated samples using the technique described in Appendix 2.

Some analyses were performed on the porous medium.

Thin section analysis

- "ness" = 2 - 1936.60 - 36.75 m

Fine sandstone with micaceous and argillaceous beds.

The thin section shows a bed 5 mm thick made of micas, clay and siderite.

Average composition :

. angular quartz, average classification (75 to 150 μ)	45%
. phyllitic minerals	10%
. clay, siderite, organic matter	17%
: intergranular porosity	28%

- "etive" 1989.39 - 89.59 m =

Mean sandstone fairly well classified.

. angular quartz, highly corroded, about 300 μ	68%
. iron oxide, clay, altered micas, siderite	5%
. phyllitic minerals	traces
. good intergranular porosity	27%

- "rannoch" - 2017.20 - 2017.53 m :

Fine sandstone, well classified, micaceous.

. angular quartz, more or less corroded (about 120 μ)	50%
--	-----

- . phyllitic minerals dispersed in the rock but with preferential orientation (bedding) 10%
- . clays, iron oxide, highly altered micas, siderite 15%
- . good intergranular porosity 30%

X-Ray diffraction analysis

- "ness" = 3 - 1921.25 - 21.35 m :

Aspect : compact with fine grains.

Containing a great amount of oil (during cleaning of the powder we gathered a very small amount of a black compound probably organic, which was supernatant on the chloroform. Amorphous to X-rays.

Mineralogical analysis :

- . about 60% quartz
- . about 30% siderite (FeCO_3)
- . very little feldspar
- . argillaceous fraction : mainly kaolinite plus illite.

- "etive" = 1 - 1994.41 - 94.60 m :

Aspect : course-grained sand (containing a bit of oil)

Mineralogical analysis :

- . 90 to 95% quartz
- . a bit of feldspar
- . traces of dolomite. ($\text{CaMg}(\text{CO}_3)_2$)
- . argillaceous phase : mainly kaolinite, traces of illite.

- "rannoch" 2017.20 - 17.53 m

Aspect : course little-compacted material, course grains

Minerological analysis :

- . about 80 % quartz
- . about 5% siderite (FeCO_3)
- . a bit of feldspar
- . argillaceous fraction mainly composed of kaolinite but also containing a complex mixture of illite, traces of chlorite, vermicullite and interstratified materials (probably illite and vermicullite?).

Scanning electron microscopy analysis

In addition, a scanning electron microscopy analysis was performed on pieces of rock of the "ness" and "rannoch" type. Some photomicrographs are given at the end of this report. Interesting views of the porous system were obtained especially for the "rannoch" type piece of rock.

2.2.- Fluids

The fluids used in the present investigation were as follows :

- Synthetic brine :

- . composition (in distilled water)

NaCl	35.6 g/l
KCl	0.4 g/l
$\text{CaCl}_2, 2\text{H}_2\text{O}$	4.68 g/l
$\text{Mg Cl}_2, 6 \text{H}_2\text{O}$	2.78 g/l

- . the pH of this brine was 6.2 at 20°C .

It was adjusted to about 7.05 by the addition of NaOH.

. specific gravity - viscosity

Temperature °C	20	40	73
Specific gravity, g/cm ³	1.026	1.022	1.006
viscosity, cP	1.083	0.713	0.413

: stock-tank oil

We used the stock-tank oil we received.

. specific gravity - viscosity

Temperature °C	20	50	73
Specific gravity, g/cm ³	0.882	0.861	0.845
viscosity, cP	17.29	6.22	3.55

. refined oils . . .

Two refined oils were used :

Soltrol 130 (supplied by Phillips Petroleum)

Albelf (supplied by Elf-Aquitaine)

Their specific gravities and viscosities are given in the following table :

Nature of oil	Temperature, °C	20°	73°
		specific gravity, g/cm ³	0.756
	viscosity, cP	1.62	0.70
Albelf	specific gravity, g/cm ³	0.88	0.847
	viscosity, cP	206	15

Soltrol 130 was used for the wettability test. It is virtually composed of isoparaffinic hydrocarbons.

3 - EVALUATION OF SURFACE PROPERTIES OF SAMPLES FROM WELL 34/10-3 UPON RECEPTION

In order to determine the preferential affinity of the solid surface for water and oil, in the state it was received, the following operations were performed with samples 3 to 8 :

- saturation of the gaseous part of the pore volume with Soltrol 130 (the mean value of the gaseous content of the samples was 49% PV) ;
- flooding with Soltrol at 20°C;
- temperature adjusted to 73°C with sample submerged in Soltrol;
- wettability test at 73°C (reservoir temperature).

Table 2 gives the results obtained.

Since the nature of the fluids contained in the porous medium upon reception is unknown, it is not possible to give the precise oil saturation at the beginning of the wettability test. But because of the previously described procedure, we are sure of having only oil and brine in the pores, the latter being with a residual saturation.

Porosity and absolute permeability were evaluated after a cleaning procedure performed at the end of the test. Unfortunately, this evaluation was not possible for samples 5, 7 and 8 (see comments in Table 1).

During flooding with Soltrol, before imbibition in brine, high values of k_{ro} to S_{wr} were obtained, i.e. an average of 1.34 for samples 3, 4 and 6 for which the permeability was evaluated after cleaning.

During the wettability tests, high oil recoveries were obtained by spontaneous displacement (2.85 to 8.25 cm³, or 16 to 21% PV for samples for which the porosity was determined). Figure 1 shows the oil recovery curves in cm³ as a function of time.

A certain classification of the curves can be seen as a function of the permeability of the samples. It can be seen that the permeability of samples No.8 is abnormally high. This is due to the deconsolidation of the sample under consideration during its assembly before the fusible alloy was set in place. In this case gravity must have played an important role during the imbibition experiment.

During imbibition in oil, no brine recovery was obtained.

During the ensuing oil flooding, the amount of brine displaced was always less than the total amount of oil displaced during the first two stages of the test, despite the use of a high-viscosity oil.

This means that residual water saturation was greater at the end of the test than at the beginning of the test. This does not change the value of the oil wettability index but affects the value of k_{ro} to S_{wr} which is underestimated.

The $\frac{k_{ro} \text{ to } S_{wr}}{k_{rw} \text{ to } S_{or}}$ ratio determined from the wettability test is between 1.1 and 11.6.

The water wettability index is finally between 0.43 and 0.59 for the "ness" formation rock, and it is 1 for the "etive" formation rock. The oil wettability index is always zero.

Consequently the rock had a clearly water wettability behavior upon reception. The "etive" formation appeared to have more preferential affinity for water (even though the interpretation of the test performed on sample No.8 must be done with care because of the possible role of gravity). This can be compared with the fact that the samples from the "etive" formation were underneath the water-oil contact level (situated at 1971m).

The generally high values of the extreme relative permeability ratio are in agreement with the preceding conclusion (if we refer to what is generally found in the literature on this subject).

Although we have not examined the case of samples from the "rannoch" type formation, because this rock has a similar mineralogical composition to that of the two preceding rocks as well as being situated in the water zone, it is probable that the rock from the "rannoch" type formation is also preferably water wet.

4 - STUDY OF CLEANING OF SAMPLES FROM WELL 34/10-3

Cleaning was done at 73°C, using the forced displacement technique. It was performed at the end of the previous test (evaluation of the wettability upon reception).

All cleaning procedures began by a circulation of toluene which is a good solvent for heavy compounds from crude oils that might be deposited on the solid surface. After this initial flood, various possibilities were attempted :

- a) A mixture of solvents containing an acid-type solvent (methanol), a basic-type solvent (acetone) and an aromatic (toluene) in proportions 15-15-70, followed by a mixture of alcohols.
- b) A solvent which can be classified as of the "acid" type, chloroform, preceded by cyclohexane and followed by a mixture of alcohols.
- c) A solvent having a "basic" nature, dioxane, followed by a mixture of alcohols (in some cases dioxane was preceded by a circulation of methanol-acetone-toluene).

A total solvent volume of about 100 times pore volume was circulated through the sample for about five days.

After cleaning, the porous medium was dried at 80°C for two days in a partial vacuum. The samples were then saturated with brine, placed in S_{wf} by a refined-oil flood (Albelf followed by Soltrol 130) at 20°C, equilibrated at 73°C and subjected to the wettability test at 73°C.

The results obtained are given in Table 3.

During brine saturation and measurement of the intrinsic permeability to brine, losses of sand occurred for samples 5 and 7. Therefore abnormally high values were obtained for porosity (38 and 45%) and absolute permeability to brine (5600 to 6000 mD). We think that this was due to a technical defect in adjusting the temperature of the oven during drying, and this cause depart of the SnBi alloy

to melt and the samples to become deconsolidated.

These two samples could not be used after this. They were replaced by two other samples, Nos 10 and 11, taken from the same cores as samples 5 and 7 in the immediate vicinity of the preceding samples.

These two new samples were cleaned after the gas phase had been replaced by Soltrol (39.9 and 13% PV respectively).

After cleaning, drying and saturation by brine, the initial oil saturation was established by an Albelf viscous oil flood at 20°C. Despite this precaution, residual saturation remained high (20 to 43% PV) compared with the in-situ S_{wi} value ($\sim 10\%$). However this parameter is only of secondary importance for the analysis performed. The value of the relative permeability to oil at the end of flooding was quite high on the whole (0.75 to 1.57).

This flood was followed by flooding by Soltrol 130 oil which changed almost nothing in the saturation state and hardly changed the values of permeability to oil (0.69 to 1.17).

After bringing the samples to equilibrium at 73°C, the wettability was evaluated.

High oil recovery was always obtained during imbibition in brine (23 to 37% PV or 39 to 53% of the oil in place) (cf. curves in Figure 2).

Likewise for each sample the same curve was plotted in Figures 3 to 7. On these figures for samples 3, 4 and 6, the result obtained during the first test (upon reception) was also included. For samples 10 and 11, oil recovery by imbibition in brine after cleaning was compared with that obtained at the reception of samples 5 and 7 which were respectively similar to the two preceding ones. To maintain a homogeneous presentation in % PV, it was assumed that the porosity of samples 5 and 7 (which could not be measured) was exactly the same as samples 10 and 11 respectively.

A comparison of the oil recovery curves during imbibition in brine upon reception and after cleaning showed that the kinetics of the phenomenon was always faster after cleaning. That might be due to an increase in the preferential affinity of the rock for water, but this is difficult to affirm. Indeed the S_{oi} values at the beginning of the experiment were probably different in the two cases, and furthermore the microscopic distribution of the two fluids, i.e. water and oil, was perhaps also different.

Imbibition time was deliberately limited so as not to make the experiment too long. There is no certainty that maximum recovery was attained, in particular in the state at the time of reception. It is therefore difficult to make any comparisons on this point.

With regard to brine recovery during imbibition in oil, once again it is still nil.

After imbibition in oil, an Albelf viscous oil flood followed by a Soltrol oil flood was performed. The k_{ro} to S_{wr}/k_{rw} to S_{or} ratio evaluated during the wettability test was between 6 and 19.6.

The value of r_w is always equal to one and that of r_o always equal to 0. But in the light of that has just been said concerning imbibition time, care should be taken in interpreting this increase in the water wettability index.

It can be seen that the same result is obtained no matter what cleaning procedure is used.

Considering the clearcut preferential water wettability of the rocks upon reception, it is not surprising that cleaning did not increase or only moderately increased the affinity for water, and that all cleaning procedures gave approximately the same result.

In the light of what has just been said, any procedure can be chosen despite the fact that for further experiments we have recommended a procedure using a methanol-acetone-toluene mixture coming after toluene.

Table 4 gives the porosity values determined from logs (from Statoil) and from gravimetric evaluation in the laboratory. It can be seen that the agreement is entirely correct.

5 - EVALUATION OF THE WETTABILITY OF "RESTORED" SAMPLES, i.e. AFTER CLEANING, SATURATION WITH RESERVOIR FLUIDS AND AGING

For samples 3, 4, 10 and 11 after the preceding test, the following series of experiments was performed.:

- . Flooding by storage oil at 73°C with a counterpressure downstream from the porous medium so as to prevent any degassing of the oil.
- . Maintaining a temperature of 73°C under pressure for a determined length of time for the "aging" of the rock-fluid system.
- . Flooding by 1.5 PV Soltrol to change the nature of the oil present in the pores. This flood was done quickly to limit any eventual desorption of products fixed on the solid (this is indispensable because the wettability test was performed at atmospheric pressure and at 73°C).
- . Wettability test at 73°C.

Since sample No. 6 had a low oil saturation at the end of the wettability test performed after cleaning (cf. Table 3), the decision was taken to clean it again, dry it and then to put the brine-oil pair back in place with a more realistic S_{oi} value. It was then subjected to the same treatment as the other samples (cf. above).

With regard to aging time, four different values were chosen for the "ness" type rock. To assign an aging time to the different samples, the results of the previous section were used so that the shortest aging times would be applied inasmuch as possible to the samples for which spontaneous oil displacement by imbibition in brine was the fastest after cleaning.

For the "etive" type rock, since only one sample was available, the influence of aging time was not investigated, and the sample under consideration underwent aging during three weeks, the maximum time for the preceding series.

The results of this part are gathered in Table 5. The curves for oil recovery by imbibition are plotted in Figure 8.

Likewise, so as to facilitate a comparison of spontaneous oil recoveries per sample no matter what the state of this sample may be, the recovery curves are also plotted in Figures 3 to 7.

With regard to oil recovery with imbibition in brine compared to the behavior of the cleaned rock, a displacement of the curves toward the right in figures 3 to 7 was noted in all cases. The kinetics of the phenomenon became of the same order of magnitude as during imbibition performed on samples upon their reception in the case of samples 3, 4, 5/10. Differences were observed for examples 6 and 7/11.

No spontaneous brine displacement was observed during imbibition in oil.

Figure 9 shows the variation in the $WI = r_w - r_o$ difference as a function of the aging time of the rock-fluid system.

Compared with the cleaned rock ($WI = 1$) a drop in the WI was observed. After several hours of aging the effect was approximately the same as after three weeks.

Concerning the results obtained with samples No. 3 for which $WI = 0.39$ when it was received, it was found that this sample also had the lowest WI value (0.43). Since this sample was less permeable, we think that this is the explanation.

Table 6 shows the value of the WI term upon reception, after cleaning and after aging, for all the samples examined.

According to the value of the WI index this table shows that the wettability of the rock after aging is finally similar to what it was upon reception.

6 - CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The following conclusions can be drawn from this investigation :

- . The wettability of the samples upon reception is clearly preferential to water for the rocks of the "ness" and "etive" types. The preferential affinity for water is more marked for the latter.
- . No matter what procedure is used for cleaning these samples, cleaning does not affect or hardly affects the preceding property. This is not surprising considering the strong affinity the rock has for water at the time of reception.
- . After cleaning, establishing saturation with reservoir fluids and aging under reservoir temperature and pressure, the rock remains preferentially water wet in the case of "ness" and "etive" formations.
- . Compared with the cleaned rock, aging reduces the kinetics of the spontaneous displacement of oil by water. The effect is more or less the same no matter whether aging lasts for a few hours or a few weeks. The behavior of the "restored" rock is finally similar to that of the rock at the time it is received. With regard to the rock from the "rannoch" formation, we did not take any consolidated samples for the present investigation. However considering the preceding results and the fact that the rock from this formation is considerably underneath the water/oil contact, it can be supposed that this rock would have led to results similar to the preceding ones.

Considering that various reasons during and after sampling may have caused the wettability of the rock to evolve toward a lower affinity for water (or a stronger affinity for oil), we feel that the rock from the formation investigated has a strong chance of being preferentially water wet in situ.

RECOMMENDATIONS

From the sole standpoint of "surface properties", we feel that the samples received can be used directly.

On the other hand for experiments in which the initial saturation state must be respected (i.e. $S_{wi} \sim 10\%$ PV), either the core samples must be taken with an oil mud (to preserve S_{wi} but care must be taken to see that this mud does not contain any additives capable of influencing the surface properties of the rock). or, care must be taken to establish initial saturations during the so-called restoration procedure.

If this procedure is used, we recommend the following operations :

Cleaning :

- . at 70 to 80°C,
- . using toluene followed by a methanol-acetone-toluene mixture in proportions of 15-15-70 (or a methanol-toluene mixture if resistance problems for Hassler membranes arise), followed by an ethanol-methanol mixture.

Drying :

- . At 70 to 80°C in a partial vacuum (water blast) during two days (or by circulation of hot nitrogen).

Establishing initial saturations :

- . To attain low S_{wi} values (of approximately 10%), the evaporation procedure is the only one that can be used for obtaining the result in a reasonable space of time. It consists in displacing a certain amount of brine saturating the sample by means of hot methane circulation, and then replacing the methane phase by decane followed by reservoir oil.

Aging

- . under reservoir and temperature conditions
- . a time of several days seems sufficient.

APPENDIX I

DESCRIPTION OF THE WETTABILITY TEST

To evaluate the preferential affinity of the rock for oil and water, a wettability test based on spontaneous and forced displacement experiments was chosen (1-3). This test is always performed with the same system of fluids, i.e. Soltrol 130 (refined oil) and synthetic brine.

No matter what prior treatment the sample had undergone before the wettability test, it had to be saturated with Soltrol oil and water, with the latter being in residual saturation.

With the samples in their initial surface state (when we received them), this was done by replacing gas from samples with Soltrol oil and then flooding them with this oil.

For samples having been cleaned and dried, we performed a 100% saturation with field brine followed by a flood with a viscous refined oil (Albelf). This flood was followed by a circulation of a less viscous refined oil (Soltrol 130).

For samples whose original surface properties had been restored by cleaning, saturation with reservoir fluids and aging under reservoir conditions, a short flood was performed with Soltrol oil so as to change the nature of the oil contained in the porous medium with minimum desorption of any polar products possibly adsorbed on the rock.

After the fluids had been injected, the following operations were successively performed :

- imbibition in brine,
- displacement by brine,
- imbibition in Soltrol oil,
- displacement by Albelf, sometimes followed by Soltrol 130.

The displacements were performed in special core holders. A fluid-volume circulation of approximately 10 times pore volume was adopted.

For all the samples, the test was performed at 73°C.

During the test, various parameters were determined (saturation, extreme relative permeabilities). A water wettability index, r_w , can be evaluated and is defined as follows (3), (1) :

$$r_w = \frac{\text{amount of oil displaced by brine by imbibition}}{\text{amount of oil displaced by brine by imbibition and displacement}}$$

Likewise, an oil wettability index, r_o , was defined as :

$$r_o = \frac{\text{amount of brine displaced by oil by imbibition}}{\text{amount of brine displaced by oil by imbibition and displacement}}$$

On the basis of a great many experiments (References 1, 2 and 3), we know that, for a preferentially water wet solid, r_o is nil and r_w has a positive value which is all the closer to one as its affinity for water is strong. For preferential oil wettability, the values of the two indices are inverted. Lastly, for intermediate or neutral wettability, both ratios have zero or very low values.

We use also the difference $WI = r_w - r_o$. The value of WI is between +1 and -1.

REFERENCES OF APPENDIX I

- 1 - CUIEC, L.E.- "Restoration of the Natural State of Core Samples",
SPE Paper 5634, 50th Annual Fall Meeting of SPE of AIME,
Dallas, Texas, 28 September - 1 October 1975.

- 2 - CUIEC, L.E.- "Méthode de restauration de l'état de surface des échantillons",
Proceedings of the International Symposium on Hydrocarbon
Exploration, Drilling and Production Techniques, Paris,
Editions Technip, 10-12 December 1975.

- 3 - AMOTT, E.- "Observations Relating to the Wettability of Porous Rock",
Trans. AIME, 216, 156, 1959.

APPENDIX II

PREPARATION OF SAMPLES

Before sampling, the core was frozen at -15°C during one night. Then after the core package was opened, the cutting out of samples 4cm in diameter and 6.4cm in length was performed under a gaseous nitrogen circulation, at a low rotation speed (700 rpm), and with a maximum of care. Then the samples were placed in a special device so as to introduce a Sn-Bi alloy between the cylindrical face of the sample and a metal piece making up the central part of the displacement cell.

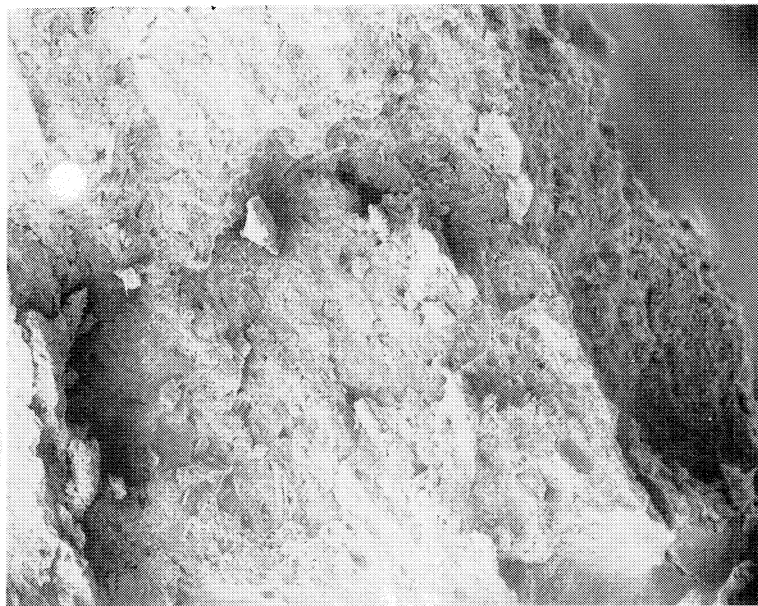
This alloy has a melting point of 138°C . The metal was cast at 150°C under nitrogen pressure to keep the fluids in the pores and to prevent their oxidation.

After cooling and slow decompression, the length of the core was adjusted with a special tool without any lubrication to avoid pollution of the faces of the samples. The final length of the samples was close to 6cm.

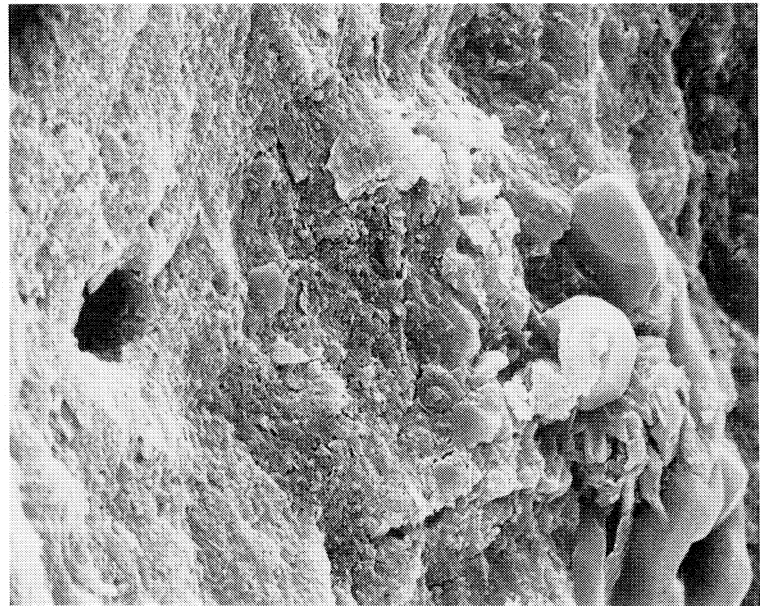
SCANNING ELECTRON MICROSCOPY ANALYSIS

(well 34/10-3 , ness 3 , 1921.25 - 1921.35 m)

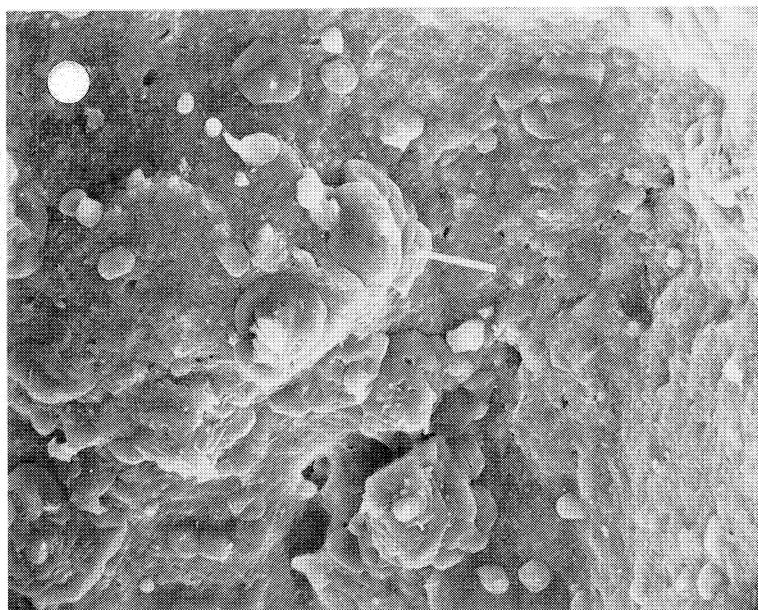
G x 100



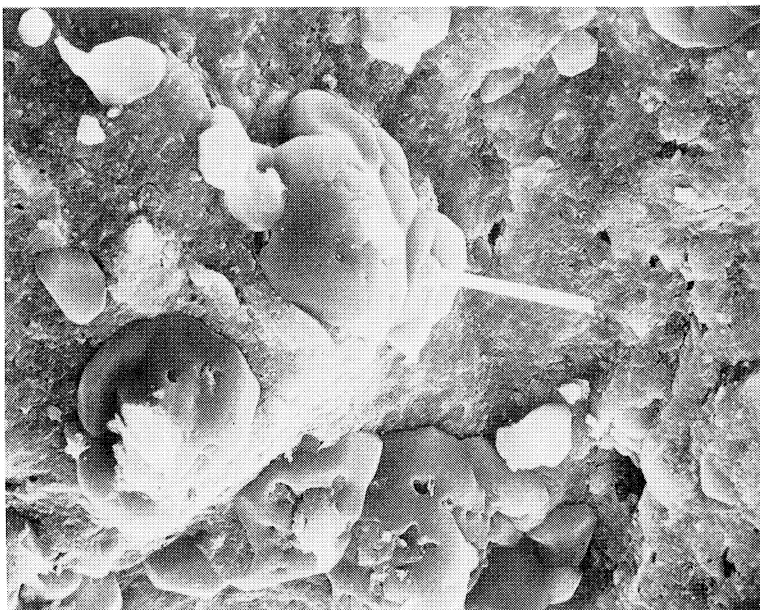
G x 4000



G X 1000



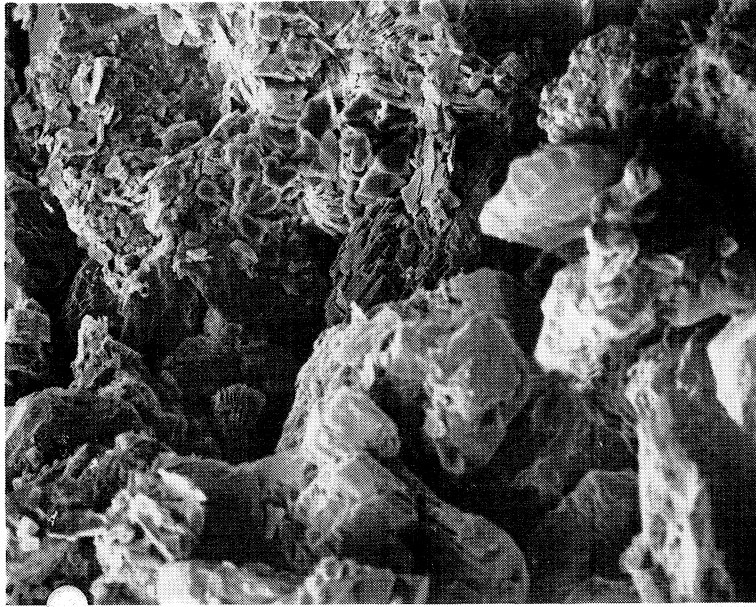
G x 2000



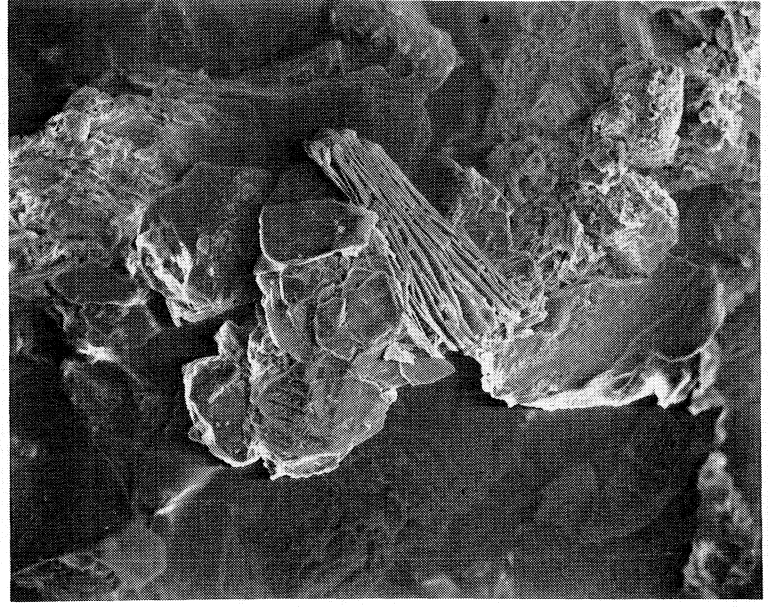
SCANNING ELECTRON MICROSCOPY ANALYSIS

(well 34/10-3 , rannoch, 2009.80 - 2010.17 m)

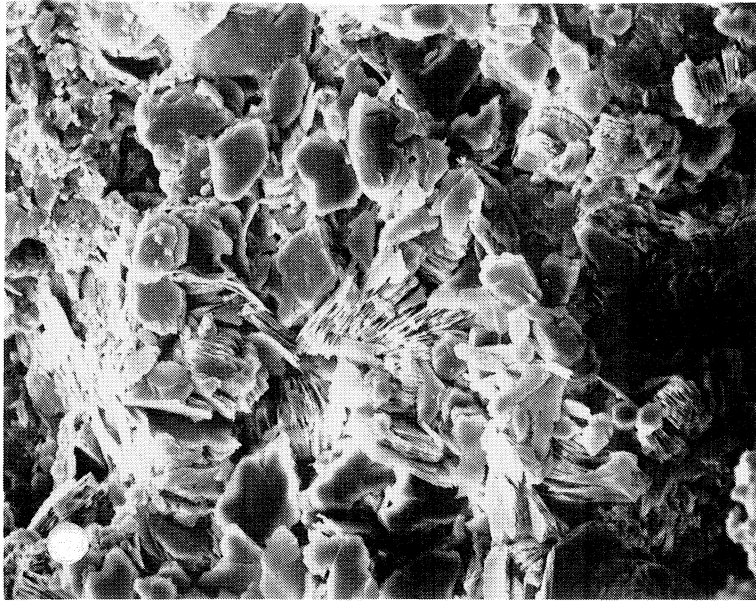
G x 200



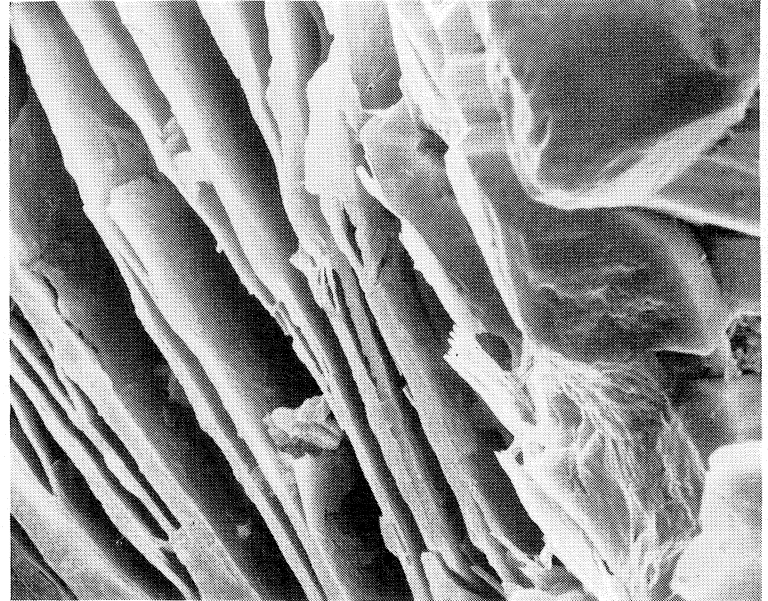
G x 200



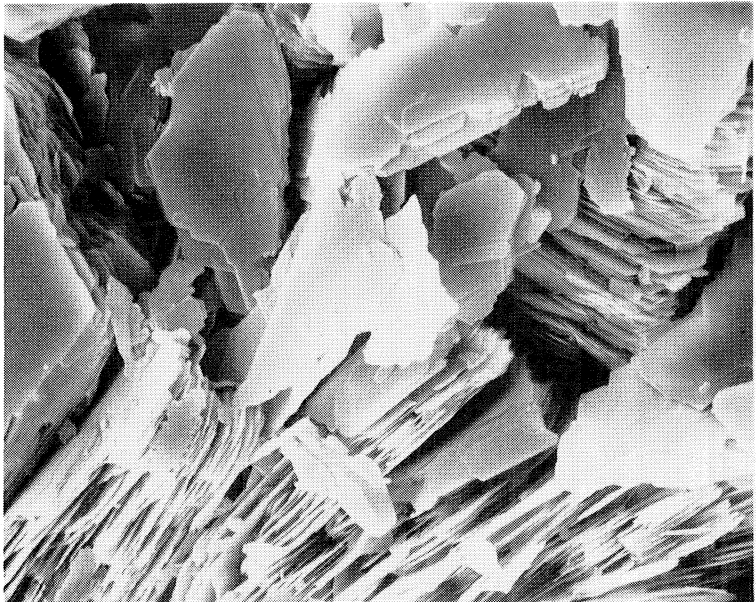
G x 400



G x 2000



G x 2000



G x 2000

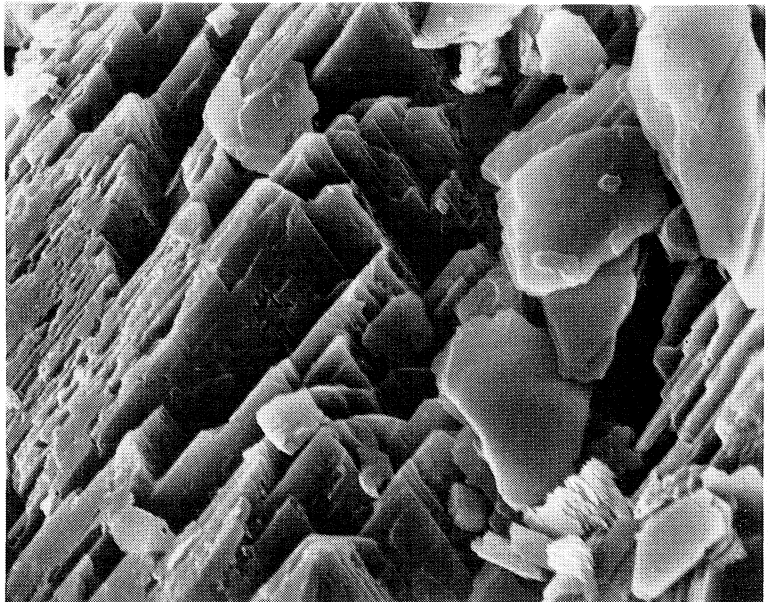


TABLE 1

IDENTIFICATION OF SAMPLES

Formation type and equiv. DST depth.	Core depth interval (m)	Our reference	Comments
"ness" = 3	{ 1919.55 - 19.75 1921.25 - 21.35	3 4	Important loss of sand after the first wet. test
= 2	1936.60 - 36.75	{ 5 10	
	1938.35 - 38.55	6	
"etive" = 1	1989.39 - 89.59	{ 7 11	Important loss of sand after the first wettability test.
	1994.41 - 94.60	8	Important loss of sand during the first wettability test.
"rannoch"*	2009.80 - 10.17	(1) (2)	Inconsolidated sand after sampling
	2017.20 - 17.53	(9)	Inconsolidated sand after sampling

* Samples 1, 2 and 9 were not used in this study.

TABLE - EVALUATION OF THE WETTABILITY OF SAMPLE UPON RECEPTION
(Well 34/10-3 - STATOIL)

WETTABILITY TEST AT 73°C															
Our reference (see Table 1)	Replacement of the gaseous phase by Soltrol 130		Soltrol flood at 20°C		Imbibition in brine		Displacement by brine			Imbibition in Soltrol	Displacement by Albelf			Wettability indices	
	Soltrol introduced cm ³	% PV*	Brine displaced % PV	k _{eff} md	Oil displaced cm ³	% PV*	Oil displaced cm ³	% PV*	k _w at S _{or} md	brine displaced % PV*	Brine displaced cm ³	% PV*	k _o at S _{wr} md	to water r _w	to oil r _o
ness 3	8.8	58.6	0	40.9 (k _r =1.42)	2.85	19.0	3.8	25.3	8.75	0	2.75	18.3	13.4	0.43	0
ness 4	12.2	48.2	0	283.7 (k _r =1.25)	5.25	20.7	4.0	15.8	23.5	0	7.0	27.7	111	0.57	0
ness 5	21.1	non avail.	0	215.6	7.7	non avail.	5.35	non avail.	15	0	3.2	non avail.	174	0.59	0
ness 6	9.55	39.7	0	54.1 (k _r =1.35)	3.85	16.0	3.4	14.1	7.5	0	4.7	19.5	28.9	0.53	0
etive 7	5.2	non avail.	0	266	3.85	non avail.	0	non avail.	57.5	0	0.4	non avail.	65	1	0
etive 8	12.9	non avail.	0	5300**	8.25	non avail.	0	non avail.	-	Important loss of sand	-	-	-	1	(0)

* from PV value obtained after cleaning, at the end of the present test.

** this sample has partially disaggregated before sealing with S-B1 alloy. After rolling in an alumina foil, we have performed the sealing. The value of k_{eff} is not representative of the original one.

TABLE 3 - EVALUATION OF THE WETTABILITY OF SAMPLES AFTER CLEANING
(Well 34/10-3 - STATOIL)

Our reference (see table 1)	Cleaning Procedure	WETTABILITY TEST AT 73°C														
		Saturation with brine.		Flood with Albelf at 20°C		Flood with Soltrol 130 at 20°C		Imbibi. in brine	Displacement by brine		Imbibi. in Soltrol	Displacement by refined oils		Wettability indices		
		porosity ϕ %	absol. perm. k_w mD	k_{ro} at S_{wr}	S_{oi} %	k_{ro} at S_{wr}	S_{oi} %	Oil displ. % PV	Oil displ. % PV	k_{rw} at S_{or}	S_{or} %	brine displaced % PV	brine displa. % PV	S_o % k_{ro} solt.	to water r_w	to oil r_o
3	toluene MAT* EtOH+MeOH**	19.9	28.7	0.75	60	0.77	60	23.3	0	0.11	36.7	0	15.7	52.4 (0.66)	1	0
4	toluene cyclohexane chloroform EtOH+MeOH	33.5	227	1.16	58.9	1.17	59.3	27.5	0	0.09	31.4	0	27.9	59.3 (0.66)	1	0
10	toluene MAT dioxane EtOH+MeOH	33.8	178	1.57	63.1	0.98	63.1	33.2	0	0.13	29.9	0	29.6	59.5 (1.22)	1	0
6	toluene dioxane EtOH+MeOH	32.1	40	0.81	56.9	0.95	56.9	23.4	0	0.075	33.5	0	12.7	46.2 (1.47)	1	0
11	toluene MAT dioxane EtOH+MeOH	29.5	1,330	0.78	71.1	0.69	71.1	37	0	0.03	34.1	0	36.1	70.2 (0.29)	1	0

* MAT = mixture methanol + acetone + toluene (in proportion 15/15/70)

** EtOH + MeOH = ethanol + methanol (50/50)

TABLE 4 - COMPARISON OF POROSITY DETERMINATIONS (Log. and gravimetric)
for Well 34/10-3 rock

Formation type and equiv. D S T deph	Core depth interval (m)	Approx. Ø log %	Ø - gravimetric determination
"ness" = 3	{ 1919.55 - 19.75 1921.25 - 21.35	24 27	19.9 (sample n° 3) 33.5 (sample n° 4)
= 2	{ 1936.60 - 36.75 1938.35 - 38.55	35 35	33.8 (sample n° 10) 32.1 (sample n° 6)
"etive" = 1	1989.39 - 89.59 { 1994.41 - 94.60	29 31	29.5 (sample n° 11) non available
"rannoch"	2009.80 - 10.17 2017.20 - 17.53	30 32	non available non available

TABLE 5 - EVALUATION OF THE WETTABILITY OF SAMPLES AFTER "RESTORATION"
 (well 34/10-3 - STATOIL)
 - INFLUENCE OF AGING TIME

WETTABILITY TEST AT 73°C															
Our refer. (see table 1)	Stock tank oil flood		Aging 73°C, duration hours	Soltrol 130 Flood		Imbibition in brine	Displacement with brine			Imbibi. in Soltrol	Displacement with refined oils			Wettability indices	
	brine displaced % PV	S _o %		k _o at S _{wr} md	S _o %		Oil displaced % PV	Oil displa. % PV	k _w at S _{or} md		S _{or} %	Brine displa. % PV	Brine displa. % PV	k _o (soltrol) at S _{wr} md	S _o %
10 "ness"	0	59.5	5	48.6	59.5	29.6	17.4	41	12.2	0	39.1	63.5	51.3	0.63	0
4 "ness"	3	62.3	46	31.5	62.3	19.8	13.4	7	29.1	0	31.2	117	60.3	0.59	0
6 * "nes"	0	73.8	161	33.5	73.8	35.0	11.7	10	27.1	0	33.8	50	60.9	0.75	0
3 "ness"	0	52.4	504	48.7	52.4	13.3	20.6	5.9	18.5	0	33.3	42	51.8	0.39	0
11 "etive"	0	70.2	504	134	70.2	41.6	14.3	17.5	14.3	1.5	49.6	560	65.4	0.74	0.03

* At the end of the wettability test performed after cleaning (see table 3), S_o was equal to 46.2. So as to study the "restoration" with a more realistic S_o, the sample was cleaned again, saturated with brine and flooded with Albelf and Soltrol before the Stock Tank oil flood, as indicated in the following table :

Cleaning as previously (see table 3)	Saturation with brine		Albelf flood		Soltrol flood	
	Porosity Ø, %	Abs.perm. k _w , md	k _o at S _{wr} md	S _{oi} %	k _o at S _{wr} md	S _{oi} %
	32.4	35.5	97	73.8	45	73.8

TABLE 6

VALUES OF $WI = r_w - r_o$ UPON RECEPTION,
 AFTER CLEANING AND AFTER "RESTORATION"
 FOR SAMPLES FROM WELL 34/10-3 (STATOIL)

Formation type	Sample number	WI upon reception	WI after cleaning	AFTER RESTORATION	
				WI	aging time hours
ness	3	+ 0.43	+ 1	+ 0.39	504
	"	+ 0.57	+ 1	+ 0.59	46
	"	+ 0.59	non available		
	"	+ 0.53	+ 1	+ 0.75	161
	"	10	non available	+ 1	+ 0.63
etive	7	+ 1	non available		
	"	+ 1	non available		
	"	11	non available	+ 0.71	504

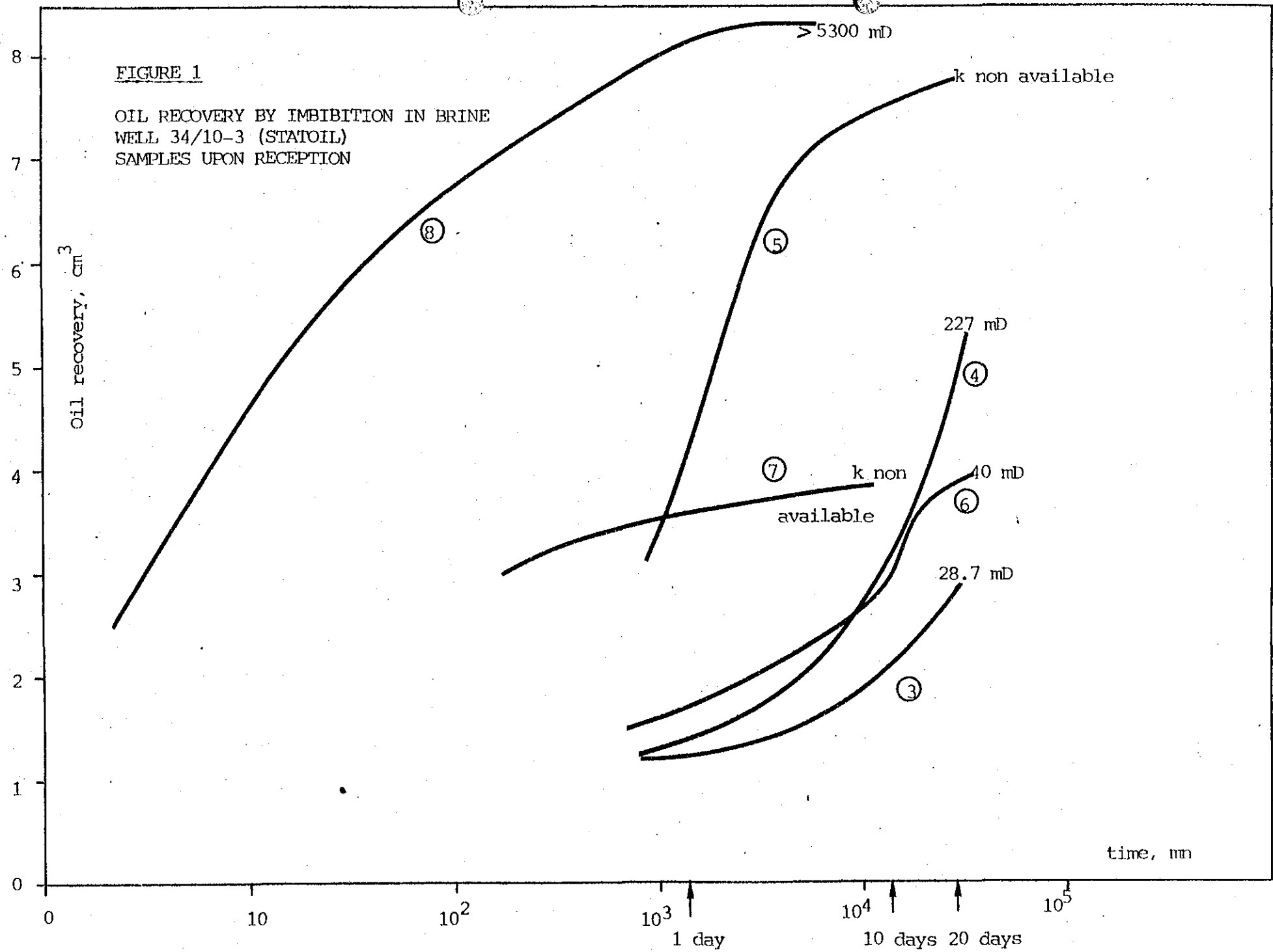


FIGURE 2

OIL RECOVERY BY IMBIBITION IN BRINE
WELL 34/10-3 (STATOIL)
SAMPLES AFTER CLEANING

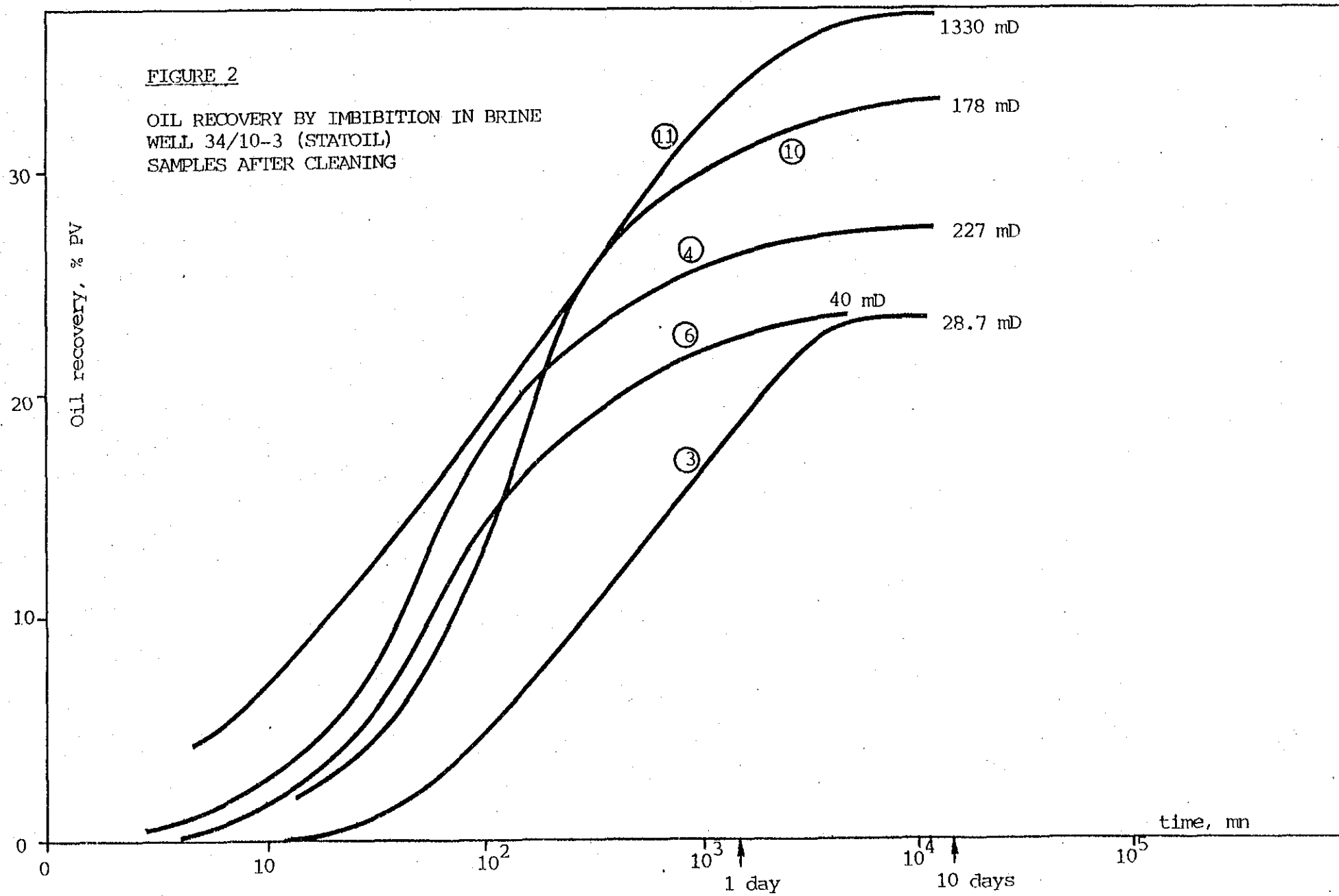


FIGURE 3

OIL RECOVERY BY IMBIBITION IN BRINE

WELL 34/10-3 (STATOIL)

SAMPLES ③ "NESS" = 3 , 1919.55 - 1919.75 m ($k_w = 28.7$ mD)

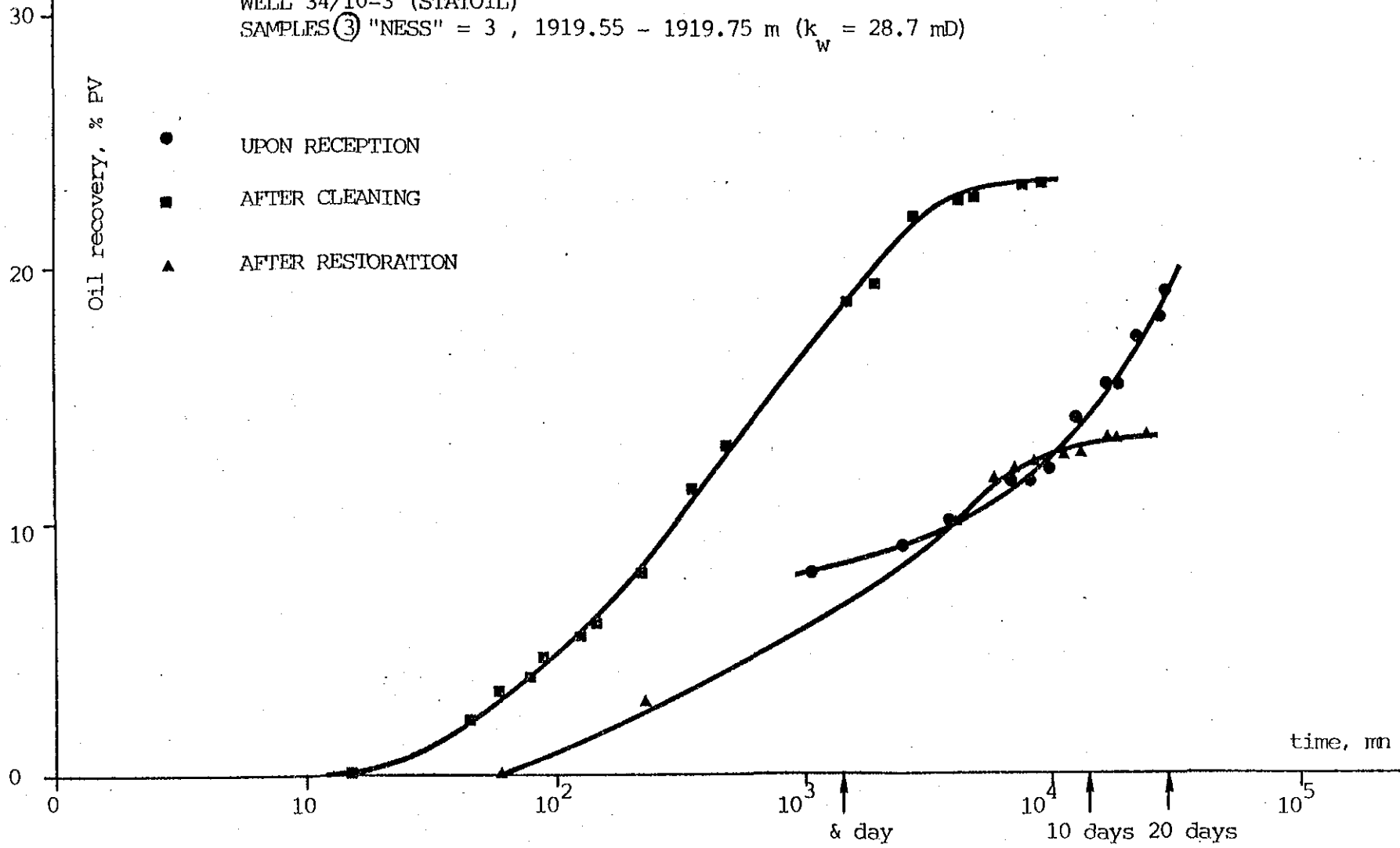


FIGURE 4

OIL RECOVERY BY IMBIBITION IN BRINE

WELL 34/10-3 (STATOIL)

SAMPLE ④ "NESS" = 3, 1921.25 - 1921.35 m ($k_w = 227$ mD)

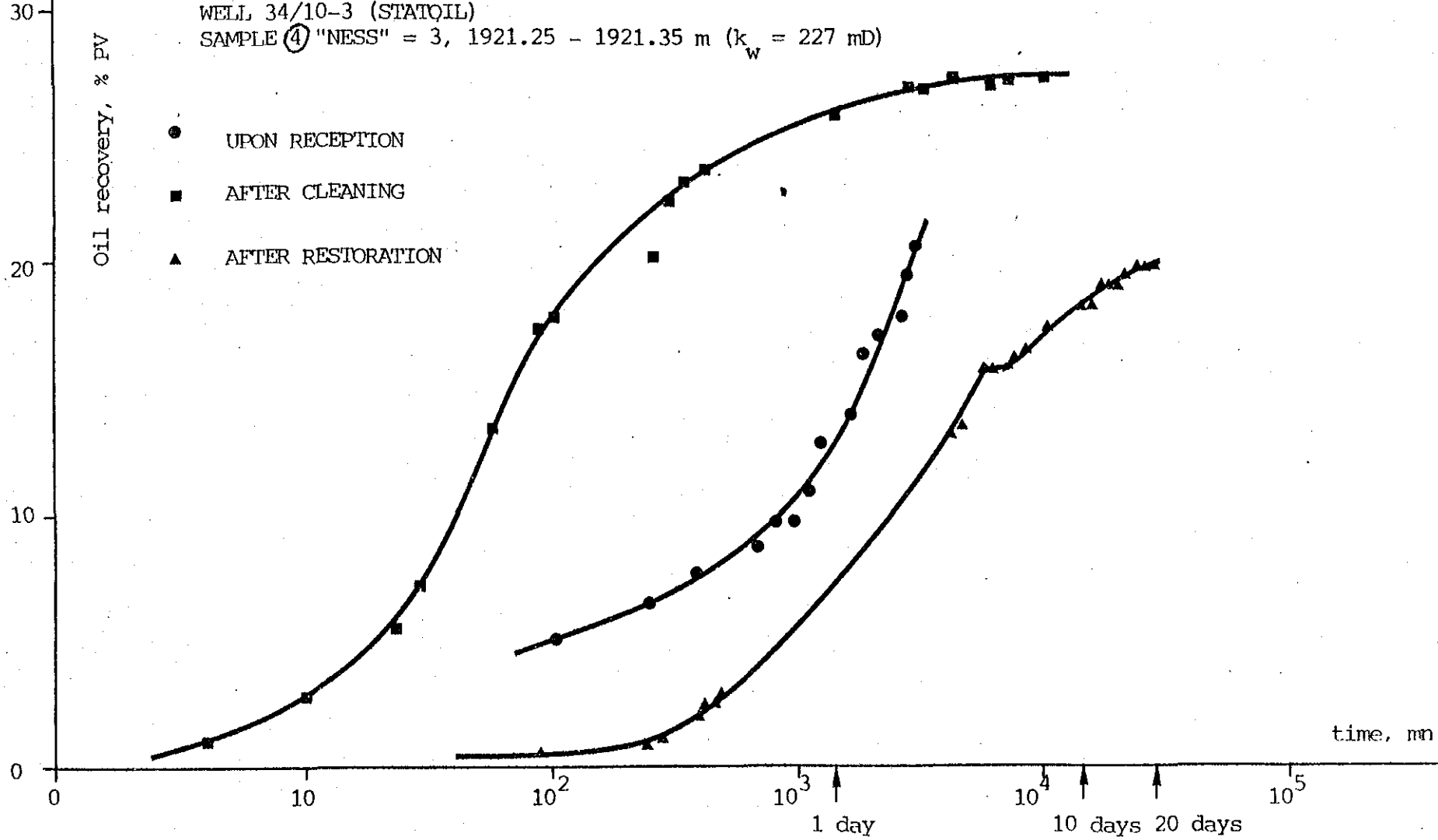


FIGURE 5

OIL RECOVERY BY IMBIBITION IN BRINE
WELL 34/10-3 (STATOIL)

"NESS" = 2, 1936.60 - 1936.75 m

Oil recovery, % PV

- UPON RECEPTION (sample ⑤) k_w non av.
 - AFTER CLEANING
 - ▲ AFTER RESTORATION
- } sample ⑩
 $k_w = 178$ mD

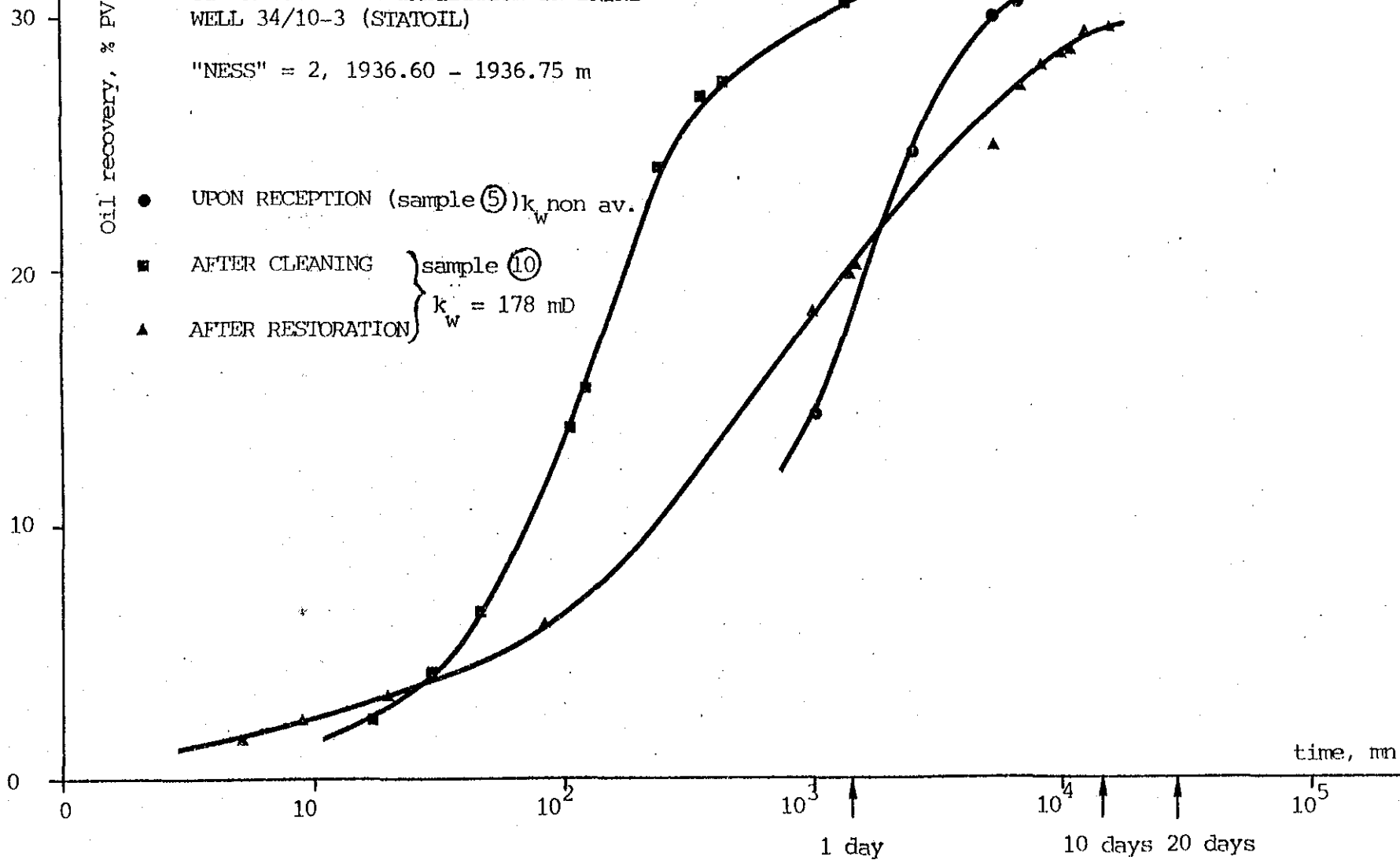


FIGURE 6

OIL RECOVERY BY IMBIBITION IN BRINE
WELL 34/10-3 (STATOIL)

SAMPLE 6 "NESS" 2, 1938.35 - 1938.55 m ($k_w = 40$ mD)

Oil recovery, % PV

- UPON RECEPTION
- AFTER CLEANING
- ▲ AFTER RESTORATION

20

10

0

0

10

10^2

10^3

10^4

10^5

time, mn

1 day

10 days 20 days

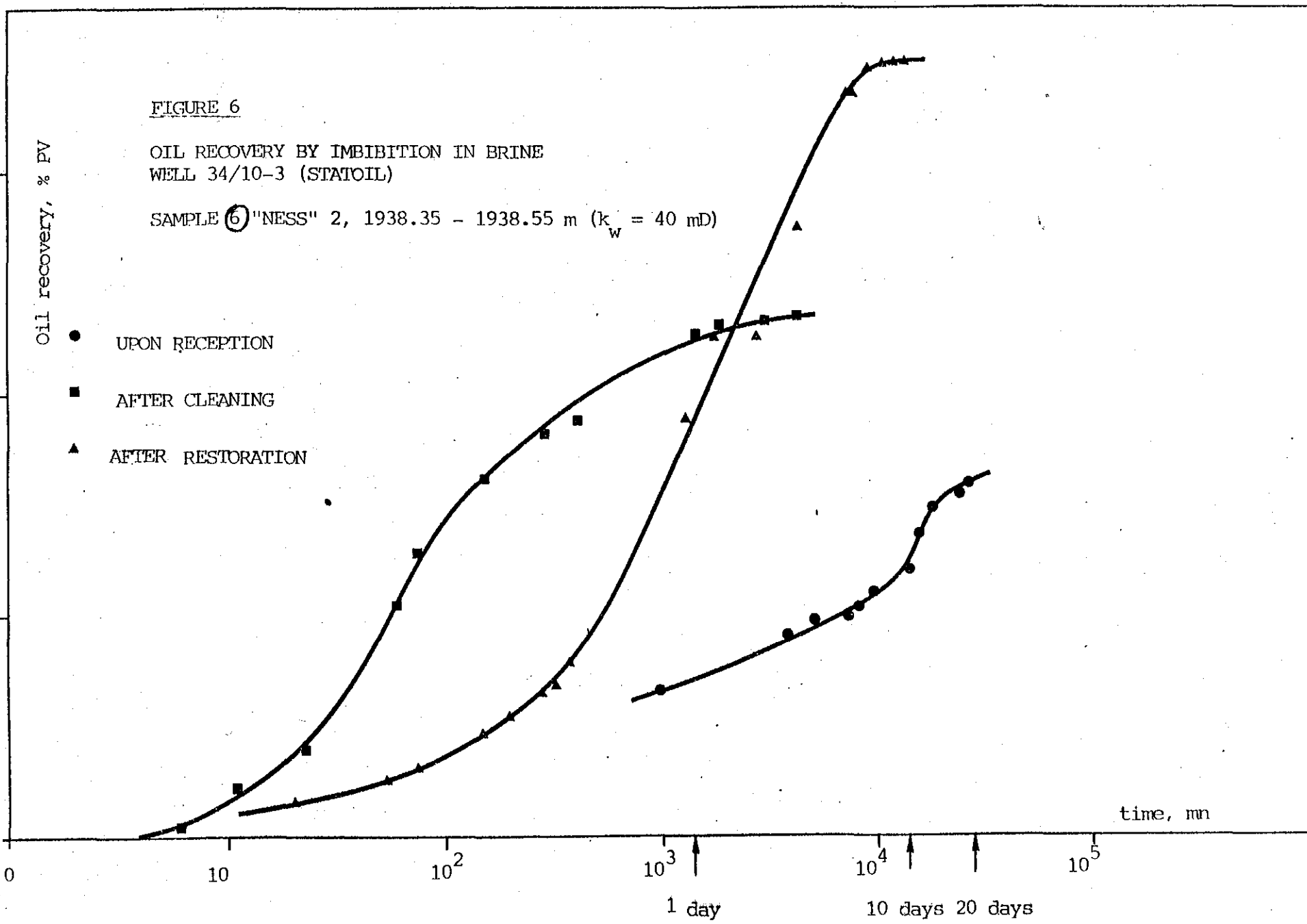


FIGURE 7

OIL RECOVERY BY IMBIBITION IN BRINE
WELL 34/10-3 (STATOIL)

SAMPLE "ETIVE" 1989.39 - 1989.59 m

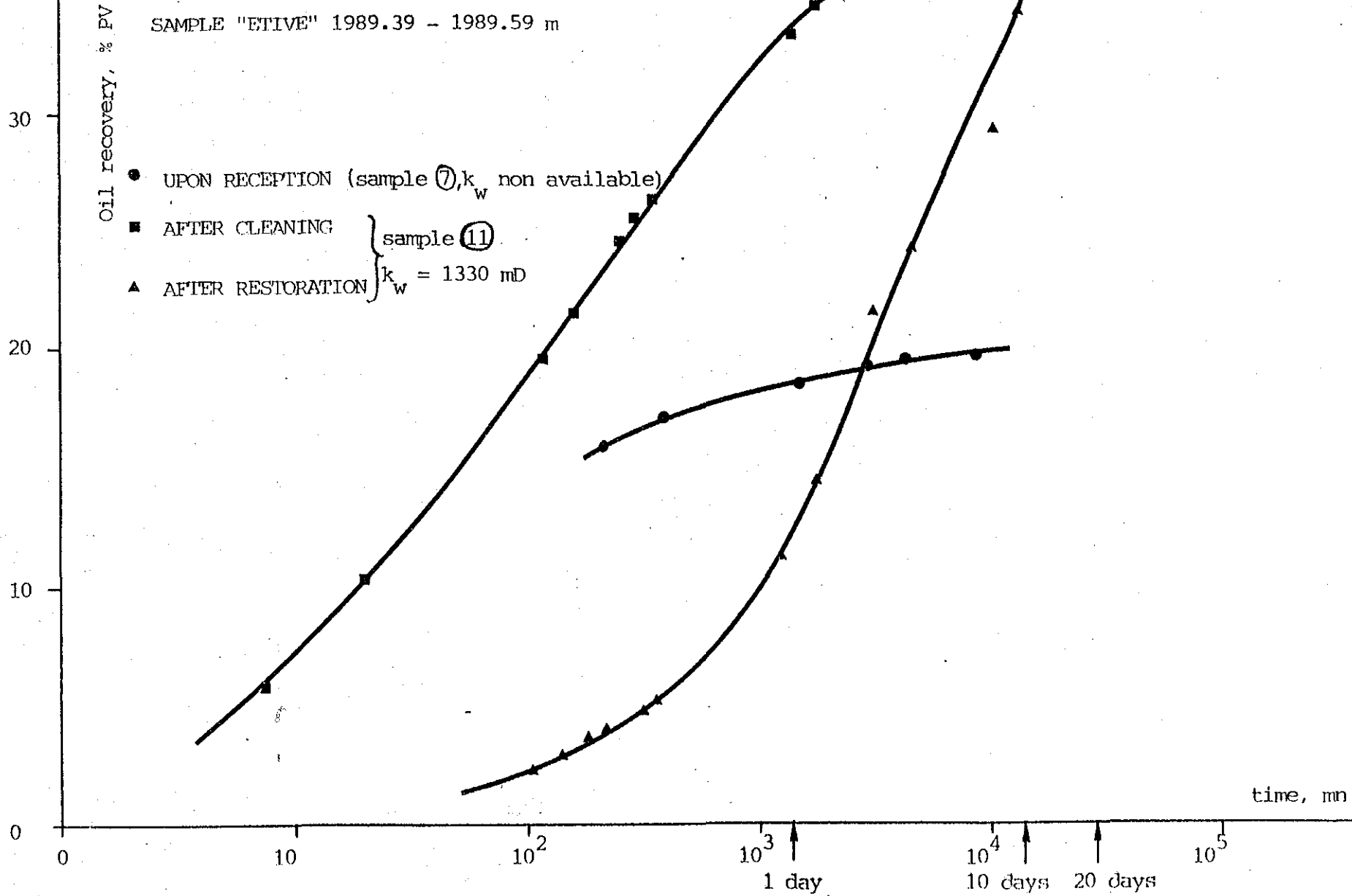
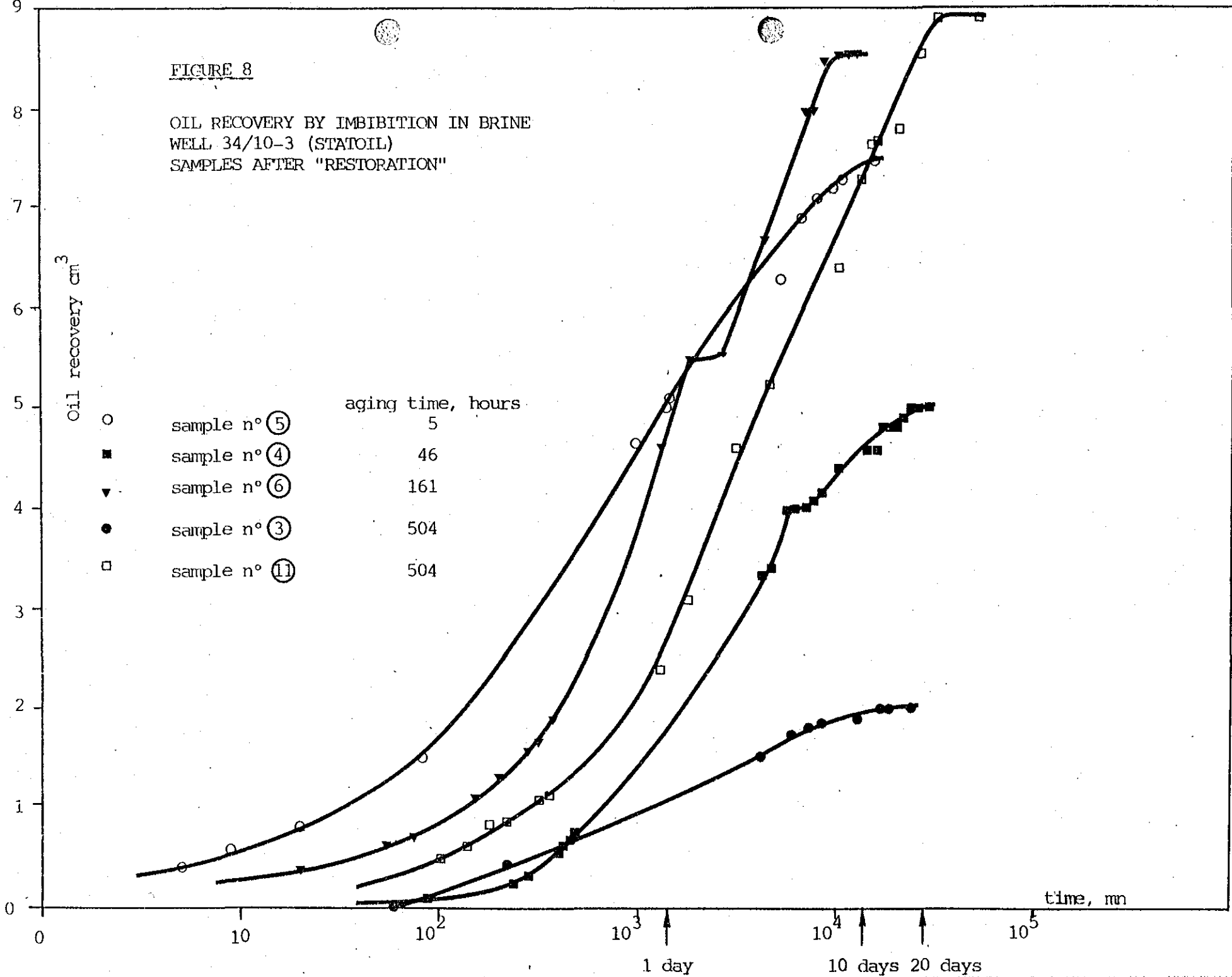


FIGURE 8

OIL RECOVERY BY IMBIBITION IN BRINE
 WELL 34/10-3 (STATOIL)
 SAMPLES AFTER "RESTORATION"

Oil recovery cm^3

○	sample n° ⑤	aging time, hours	5
■	sample n° ④		46
▼	sample n° ⑥		161
●	sample n° ③		504
□	sample n° ⑪		504



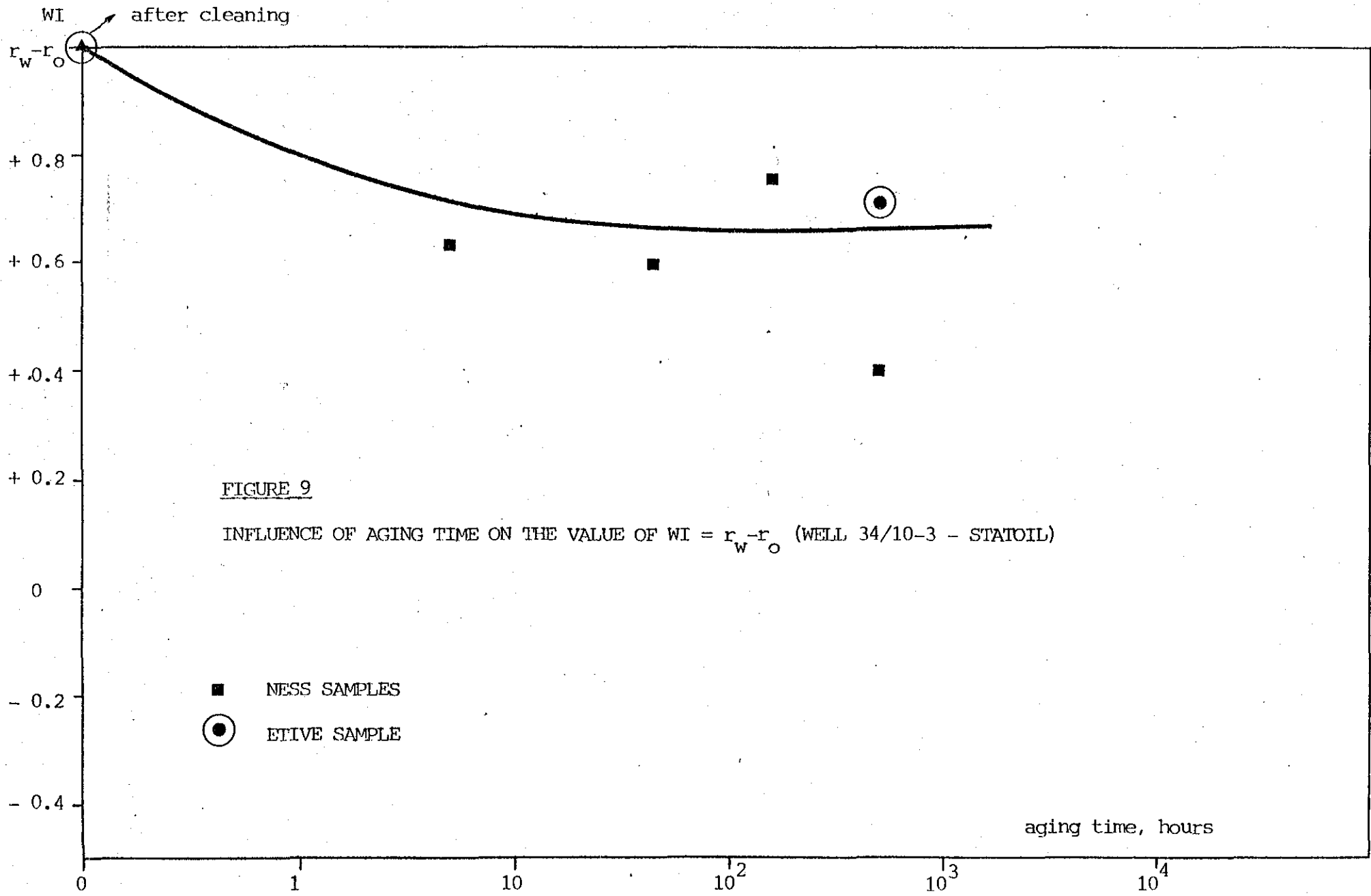


FIGURE 9

INFLUENCE OF AGING TIME ON THE VALUE OF $WI = r_w - r_o$ (WELL 34/10-3 - STATOIL)