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**EVALUATION OF THE EFFECTS OF LONG-TERM-STORAGE IN SALT
CAVERNS ON THE PHYSICAL AND CHEMICAL PROPERTIES OF CERTAIN
CRUDE OILS AND DISTILLATE FUEL OILS**

Final Report

July 1979

Work Performed Under Contract No. EL-78-C-01-7151

**Kavernen Bau-und-Betriebs—GmbH
Hannover, Germany
and
KBB Incorporated
Harahan, Louisiana**

MASTER

U. S. DEPARTMENT OF ENERGY

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FINAL REPORT

prepared for

U.S. DEPARTMENT OF ENERGY STRATEGIC PETROLEUM RESERVE OFFICE

Contract No. EL-78-C-01-7151

Evaluation of the Effects of Long-Term-Storage in Salt Caverns on the Physical and Chemical Properties of Certain Crude Oils and Distillate Fuel Oils

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Contents

- Acknowledgements
- 1 Introduction
- 2 Summary
- 3 Background and historical data
 - 3.1 Etzel site - background data
 - 3.1.1 Oil storage
 - 3.1.2 Crude oil landing
 - 3.1.3 Oil storage in K 117
 - 3.2 Lesum site - background data
 - 3.2.1 Crude and fuel oil landing
 - 3.2.2 Crude oil storage in L 103
 - 3.2.3 Fuel oil storage in L 104
 - 3.3 Etzel site - historical data
 - 3.3.1 Leaching/oil fill/brine displacement operations
 - 3.3.2 Preventing the brine from being contaminated with oil
 - 3.4 Lesum site - historical data
 - 3.4.1 Determination of the oil/brine interface level and temperature logs
 - 3.4.2 Separating systems
- 4 Sampling at the Etzel and Lesum site
 - 4.1 Preparatory work at Etzel
 - 4.1.1 Depressurization
 - 4.1.2 Workover operations
 - 4.2 Sampling at Etzel

- 4.3 Preparatory work at Lesum
 - 4.3.1 Depressurization
 - 4.3.2 Workover operations
- 4.4 Sampling at Lesum
- 5 Chemical analysis program
 - 5.1 Analyses
 - 5.2 Special analyses procedures
 - 5.2.1 True boiling point distillation
 - 5.2.2 Atomic absorption spectrophotometry
 - 5.2.3 Determination of total amount of nitrogen in crude oil
- 6 Evaluation of results
 - 6.1 Comparison of present and historical data
 - 6.2 Mixing and stratification of crude oil and fuel oil in the caverns
 - 6.3 Extent and stability of emulsions
 - 6.4 Chemical contaminations of the brine below the oil/brine interface layer
- 7 Conclusions

Final Report

Enclosures

Cavern No.

1	Map of Northern Germany with sites Etzel and Lesum		
2	Amounts of crude oil filled intoK 117	T*	
3	IVG Pool delivery NWO and Etzel	T	
4	Oil shipment to NWO	T	
5	Amounts and types of crude oil pumped intoK 117	T	
6	Cavity caliper ofK 117	T	
7	Theoretical oil layer thickness of cavernK 117	T	
8	Theoretical cavern configurationK 117	G**	
9	Sonar surveyK 117	G	
10	Amounts of crude oil stored inL 103	T	
11	Historical data of Arabian Light	T	
12	Sonar surveyL 103	G	
13	Amounts of fuel oil stored inL 104	T	
14	Sonar surveyL 104	G	
15	Specifications of fuel oil	T	
16	Temperature logs from.....K 117 (historical)	G	
17	Flow sheet Etzel	G	
18	Temperature logs fromL 103 (historical)	G	
19	Temperature logs fromL 104 (historical)	G	
20	Depressurization of cavernK 117	T	

* Table

** Graph

		<u>Cavern No.</u>	
21	Completion of	K 117	G
22	Prakla probe		G
23	Filling procedure of sample receptacles		G
24	Time required and notes on sampling	K 117	T
25	Sampling depths	K 117	G
26	Depressurization of	L 103	T
	and	L 104	
27	Completion of	L 103	G
28	Completion of	L 104	G
29	Time required and notes on sampling	L 103	T
30	Time required and notes on sampling	L 104	T
31	Sampling depths	L 103	G
32	Sampling depths	L 104	G
33	Water and sediment	K 117	T
34	Density	K 117	T
35	Mercaptan, H ₂ S and total sulfur	K 117	T
36	Pour point	K 117	T
37	Salt content	K 117	T
38	Kinematic viscosity	K 117	T
39	Neutralization number	K 117	T
40	Vapor pressure	K 117	T
41	Optical density color	K 117	T
42	Nitrogen content	K 117	T
43	Trace metal-copper	K 117	T
44	Trace metal-iron	K 117	T
45	Trace metal-vanadium	K 117	T
46	Trace metal-nickel	K 117	T

			<u>Cavern No.</u>	
47	TBP distillation-sample	2	K 117	T
48	TBP distillation-sample	3	K 117	T
49	TBP distillation-sample	8	K 117	T
50	TBP distillation-sample	12	K 117	T
51	TBP distillation-sample	14	K 117	T
52	TBP distillation-sample	15	K 117	T
53	TBP distillation-sample	20	K 117	T
54	TBP distillation-sample	21	K 117	T
55	TBP distillation-sample	22	K 117	T
56-A	TBP distillation-samples 2, 3, 14, 15, 20, 21, 22	K 117		G
56-B	TBP distillation-samples 2, 3, 8, 12, 14, 15, 20, 21, 22	K 117		G
57	Light hydrocarbon analysis fraction 2	K 117		T
58	Light hydrocarbon analysis fraction 4	K 117		T
59	Light hydrocarbon analysis fractions 2 and 4	K 117		G
60	Crude oil analyses Lesum	L 103		T
61	Crude oil analyses Lesum	L 103		T
62	Crude oil analyses Lesum	L 103		T
63	Trace metals Lesum	L 103		T
64	TBP distillation-sample	1	L 103	T
65	TBP distillation-sample	2	L 103	T
66	TBP distillation-sample	4	L 103	T
67	TBP distillation-sample	6	L 103	T
68	TBP distillation-sample	10	L 103	T
69	Light hydrocarbon analysis fractions 2 and 4	L 103		T
70	Light hydrocarbon analysis fractions 2 and 4	L 103		G

		<u>Cavern No.</u>	
71	Fuel oil analyses Lesum	L 104	T
72	Fuel oil analyses Lesum	L 104	T
73	Fuel oil analyses Lesum	L 104	T
74	Engler distillation Lesum	L 104	T
75	Brine analyses Etzel and Lesum		T
76	Test distillation	K 117	G
77	Comparison - density	K 117	G
78	Comparison - viscosity	K 117	G
79	Comparison - vapor pressure	K 117	G
80	Comparison - neutralization number	K 117	G
81	Comparison - density	K 117	T
82	Comparison - viscosity	K 117	T
83	Comparison - vapor pressure	K 117	T
84	Comparison - neutralization number	K 117	T
85	Comparison - water and sediment	K 117	T
86	Brine sample analysis - Etzel cavern	K 101/111	T
87	Brine sample analysis - Etzel cavern	K 101/111	T
88	Brine sample analysis	K 117	T

Photographs follow enclosures

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Mr. Harry N. Giles, DOE's technical project officer for this contract assisted in reviewing the various reports, evaluating and interpreting the data, and preparing this Final Report.

Mr. Peter Niederhoff of KBB GmbH served as project manager for this contract.

1 Introduction

In the United States, the Energy Policy and Conservation Act of 1975 established the requirement for a Strategic Petroleum Reserve (SPR) of up to one billion (10^9) barrels of oil. The Federal Energy Administration, now a part of the Department of Energy (DOE) was tasked with implementing the SPR. The majority of the oil comprising the SPR will be stored in solution-mined caverns in salt, and in conventionally dry-mined chambers in salt and in other rocks.

Because there was very little information available on the effects of long-term storage on the physical, chemical, refining and/or marketing characteristics of crude oil and fuel oil, DOE contracted KBB Inc. to perform pertinent studies. Five Interim Reports dealing with various phases of the studies were prepared. This Final Report presents a comprehensive evaluation of the effects of long-term storage in salt-solution caverns on the properties of certain crude and distillate fuel oils.

In the northern part of West Germany, near Wilhelmshaven (see enclosure 1), 33 caverns, leached in the Etzel saltdome, are used to store crude oil.

The caverns are designed for an initial capacity of $500\,000\text{ m}^3$ ($3.15 \cdot 10^6$ bbl). When needed, this amount can be withdrawn, and the caverns refilled up to four times. The caverns will then have reached their maximum permissible volume of $1\,000\,000\text{ m}^3$ ($6.30 \cdot 10^6$ bbl).

In 1971, the IVG (Industrieverwaltungsgesellschaft mbH), Bonn, awarded a turnkey contract to KBB (Kavernen Bau- und Betriebs-GmbH), Hannover, to design and construct the cavern project. KBB executed the entire underground storage project, including solution mining and oil storage operations, sonar surveys, monitoring, and analyses.

At Lesum, near Bremen, MOBIL AG Germany, has six caverns filled with crude or distillate fuel oils.

KBB performed solution mining of several caverns at this site, and planned the filling process of other caverns.

Although KBB's engagement at both sites was essentially completed, KBB was permitted to collect and analyze oil samples from three caverns on behalf of DOE.

Following award of the DOE contract in June 1978, preparatory work began. During August and September 1978, 50 samples were taken from 3 caverns, at different depths.

Laboratory analyses and evaluations of these samples were conducted between October 1978 and June 1979.

In order to evaluate the long-term behaviour of crude and fuel oils stored in salt caverns, KBB Inc., USA, together with KBB GmbH, West Germany, were commissioned to carry out this investigation on three caverns in West Germany, which have contained either crude or fuel oils for several years.

The steps of the investigation are detailed in the flow chart on page 7.

From Etzel cavern K 117 and Lesum caverns L 103 and L 104 a total of fifty oil and brine samples were taken and subsequently analyzed in a comprehensive program.

Etzel cavern K 117 contains a variety of light and heavy crude oils, thus it is especially suitable for studies of stratification and mixing. The results show that

- a) mixing has taken place to a certain degree - variations in properties, such as density and viscosity are negligible
- b) some primary theoretical layers are still recognizable, based on the concentration of sulfur, mercaptans, salt, as well as by neutralization number, and
- c) mixing occurs, more readily, when a heavy crude oil is stored above light crude.

In Lesum cavern L 103 only one type of crude oil, Arabian Light, is stored. Here no evidence of stratification of the individual crude oil batches could be found. Variations in quality of the individual crude oil batches are minimized during storage.

Since 1971 Lesum cavern L 104 has been filled with distillate fuel oil, although various amounts have been withdrawn and refilled at times. Investigations indicate that no deterioration of the fuel oil has occurred relative to its purchase specifications. Slight variations in the physical and chemical properties of the various fuel oil batches pumped into storage are still recognizable. Mixing has not taken place completely, apparently due to negligible convection currents.

Investigation of samples obtained from within or just above a viscous brine/oil interface layer in the two crude oil caverns, indicate that its properties are dependent on the quantity of oil stored, the quality of the individual batches of oil prior to storage, and the period of storage. Studies indicate that the layers in both caverns account for approximately 0.5 % of the amount of crude oil stored. The layers contain sedimented components of the stored crude oil; namely water, sediment, salt, trace metals and mercaptans. It is believed that the layers also contain relatively large contents of asphaltenes and n-alkane waxes.

In the fuel oil cavern, no emulsion stratum was found. Only small quantities of the components which are necessary to decrease surface tensions, and promote emulsion formations are found in fuel oils. Furthermore, there is insufficient water in the fuel oil for the development of an interfacial emulsion layer.

Oil contamination of the brine below the oil/brine interface layer is not significant. The values of oil-in-brine were determined to be less than 0.1 ppm. It should be noted that this value is even less than those values determined for the brine withdrawn during the solution-mining process.

It is impossible to make exact statements about changes in the properties of the various crude and fuel oils stored in the three caverns studied, because of the scarcity of historical and baseline data. However, it is possible to say that the crude and fuel oils have not undergone any deleterious changes. The crude oils could be withdrawn and processed using existing refining technology. Similarly, the fuel oils have not undergone any changes which would affect their marketability.

Evaluation of the Effects of Long-Term-Storage in Salt Caverns Properties of Certain Crude Oils on the Physical and Chemical and Distillate Fuel Oils

3 Background and historical data

In this report, background and historical data include data concerning crude-oil storage, solution-mining processes, cavern-shape development, leaching/oil filling, brine displacement operations, monitoring systems, and analyses of brine and oil at the Etzel and Lesum sites.

3.1 Etzel site - background data

3.1.1 Oil storage

At Etzel, one of the 33 caverns, namely K 117, was selected for sample collection. The leaching of this cavern started in July 1974, and filling with crude oil in September 1975. The last batch of oil was pumped in in January 1977 with a resulting total volume of 460 000 m³ ($2.89 \cdot 10^6$ US bbl). The volumes, dates, and generic types of crude oil stored are given in enclosure 2.

As common to all Etzel caverns, a variety of light and heavy oils were stored in cavern K 117.

In addition to the fuel oil which was used as a blanket medium in the initial phase of solution mining, the following types of crude oils are stored:

Arabian Light (Saudi Arabia)

Iran Light (Iran)

Iran Heavy (Iran)

Basrah (Iraq)

IVG Pool.

When storing crude oils in Etzel no attempt was made to store one particular generic type in one particular cavern. This was impracticable for the following reasons:

- 1) The oil cargos from tankers were conveyed into an interim storage facility, and the oil was later pumped to Etzel at a high flow-rate.
- 2) The solution-mining concept is different for nearly every cavern, and consequently preset quantities of oil had to be pumped in at certain intervals to fill the predetermined cavern shapes.
- 3) The quantities which were pumped from the NWO storage farm to Etzel were too large for any one cavern, for the reasons mentioned under 2).

As an example, in September 1976, one tanker at NWO discharged 259 694 m³ of Iran Heavy and 23 974 m³ of Arabian Light, and then in November, another tanker discharged 254 439 m³ of Iran Heavy. All these quantities of oil were allocated to different Etzel caverns, after interim storage in the NWO facilities; thus nearly every type of oil is stored in every cavern.

The so-called IVG Pool Oil presents a special case. It is a composite of several crude oils which had been stored with NWKG *) over a period of more than 6 months, and had been pumped backwards and forwards between several of their tanks.

*) NWKG - tank farm near Wilhelmshaven

It consisted of the following generic crudes:

Arabian Light
Arabian Heavy
Iran Light
Iran Heavy
Nigeria Light
Basrah
Khafji.

A qualitative statement on this oil cannot be made, as the composition of each batch was different.

Temporary storage in the NWKG tanks occurred when either the official NWO (Nordwestdeutsche Olleitungsgesellschaft) tanks could not accept any more oil from the arriving tankers, or when Etzel was unable to receive oil for injection into the caverns. Enclosure 3 shows the amounts of oil pumped from NWKG to NWO, as well as from NWO at Wilhelmshaven to the Etzel cavern field, which is 26 km away.

The interim storage facilities of NWKG were only used between September 1976 and February 1977.

The unloading of oil for storage in the caverns began in November 1974.

Enclosure 4 shows the amounts of oil which were unloaded from tankers and stored by NWO. These amounts are further classified to show also the type of oil, the density at the time of unloading, the name of the tanker and the arrival date.

3.1.2 Crude oil landing

The crude oils which are stored at Etzel are composed entirely of shipments which were unloaded about 26 km away in Wilhelmshaven.

In Wilhelmshaven samples were taken and analyzed to provide the basis for the calculation of the net oil quantity.

Three checks were performed, each with subsequent analyses. These were:

- a) When the ships arrive, an independent inspector takes samples from the various ship compartments and examines them in a laboratory (pre-analysis).
- b) After the oil was pumped into the NWO tanks, one or two samples were taken from each tank, subsequently analyzed, and the results compared to the pre-analyses. These results then appeared in the Tanker Unloading Report.
- c) While the oil was being pumped from the NWO tanks to the Etzel cavern field, samples were collected from the pipeline at regular intervals, using an automatic sampler.

The samples represent a cross-section of the crude oil batch, and the analyses results served as the basis for the calculation from which "clean oil landed" data was computed. The water content was not allowed to exceed 1 %.

For tank analyses, tanker unloading reports, tanker time sheets, ship inspections, survey and ullage reports see enclosures in the original report "Background and Historical Data", August 1978.

3.1.3 Oil storage in K 117

At irregular intervals oil was pumped into the cavern, and the brine/blanket interface level was determined by gamma log. This was necessitated by the solution mining and fill concept for the Etzel caverns (see the historical data section).

During the initial solution mining phase fuel oil was used as a blanket (July 1974 to August 1975). This was followed by a partial filling with crude oil, taken from other caverns, as well as directly from the NWO storage tanks.

A breakdown of the generic types of crude oil taken from other caverns was extremely difficult, because some of these oils had been pumped from yet other caverns. The data in enclosure 2 was obtained through extensive research of documents at the cavern facilities. The data in enclosure 5 provides a chronological breakdown of the quantities and generic types of crude oil stored, and was compiled from enclosure 2.

Cavern shape development and determination of stratification

Due to irregular filling and interface level determinations required by the solution mining concept, cavity growth can only be calculated in terms of equivalent theoretical cavern diameter. The theoretical cavity diameters were computed using the amounts of oil pumped in and the interface level determinations. These formed a basis for the computation of the experimental cavern shape (see enclosure 6).

Using these cavity caliper computations and the crude oil fill and stratification data, the theoretical thickness of the individual oil layers was compiled (see enclosure 7).

These individual theoretical layers, superimposed on the drawing of the cavern configuration, are shown in enclosure 8.

This theoretical stratification served as a basis for determining the sampling depths.

For comparison with the cavern shape which was determined theoretically, enclosure 9 shows the actual configuration determined by a sonar survey performed subsequent to the sampling process. The actual shape is in close agreement with the theoretical shape.

3.2 Lesum site - background data

The Lesum cavern field, near Bremen, consists of six caverns. Three of these caverns are filled with fuel oil, and the others with crude oil.

MOBIL OIL AG GERMANY is the owner of these storage facilities. The caverns are intended to serve as

- a) an emergency reserve, and
- b) a seasonal storage facility for the refinery.

One fuel oil cavern is equipped with a submersible centrifugal pump for operational storage purposes.

The caverns have volumes of from 350 000 to 450 000 m³ ((2.2 - 2.8) · 10⁶ US bbl), but some are not completely filled with crude or fuel oil.

In contrast to the Etzel project, only one type of crude oil is stored at Lesum, namely Arabian Light. At Lesum, one cavern filled with crude oil and another one filled with fuel oil were selected for test purposes. These are L 103 (crude oil) and L 104 (fuel oil).

From April 1971, when solution mining commenced, until completion of filling at the end of 1975, exclusively Arabian Light crude oil was stored in cavern L 103 (in total 295 580 m³ $\approx$ 1.86 · 10⁶ US bbl). Solution mining was terminated in May 1972.

The fuel oil stored in cavern L 104, was injected during the interval December 1971 through December 1977. Solution mining lasted from December 1971 through November 1975. The amount of fuel oil stored by December 1977 was 224 163 m³ ($1.41 \cdot 10^6$ US bbl).

3.2.1 Crude and fuel oil landing

The crude oil arrived by tanker at Wilhelmshaven and was subsequently transferred to river tankers and transported to the GTL (a large tank farm) at Bremen.

At Bremen, where the MOBIL OIL refinery is also situated, the oil was either refined or pumped directly, via pipeline, to the Lesum storage facilities.

3.2.2 Crude oil storage in L 103

During the initial phase of the leaching process the cavern roof was protected by a small amount of fuel oil. When the roof diameter increased sufficiently, crude oil was pumped in for storage purposes.

At present, the total amount of crude oil in the cavern is 295 580 m³ ($1.86 \cdot 10^6$ US bbl). The dates and amounts pumped in are shown in enclosure 10.

Analytical data on the stored crude oil is nearly non-existent, because only random samples were obtained for analyses during injection. Moreover, nearly all the analytical results were lost during the process of building new offices and the subsequent move in 1975.

An analysis of Arabian Light crude oil, made in 1972, was provided by MOBIL OIL as reference material. The results of this analysis are shown in enclosure 11. Further data was given to KBB in late 1978 (see "Comparison of Historical and Present Data, " May 1979).

The sonar survey shown in enclosure 12 represents the final measurement of the cavern, and was made after the end of solution mining. The cavity volume was determined by PRAKLA SEISMOS to be $V = 346\,000\text{ m}^3$ ($2.18 \cdot 10^6$ bbl). Thus, another $50\,000\text{ m}^3$ ($0.31 \cdot 10^6$ US bbl) can be stored if necessary.

3.2.3 Fuel oil storage in L 104

As cavern L 104 had been planned to be used for the storage of fuel oil, there was no problem in selecting the blanket fluid. Fuel oil was injected into the cavern during leaching operations and the cavern was partially filled with the same fuel oil when leaching was finished. Filling was started in December 1972, when leaching began, and continued until December 1977. Leaching of the cavern was completed in November 1973.

Cavern L 104 presently contains $224\,163\text{ m}^3$ ($1.4 \cdot 10^6$ US bbl). The volume of the cavern is $353\,700\text{ m}^3$ ($2.2 \cdot 10^6$ US bbl). Thus, another $130\,000\text{ m}^3$ of fuel oil could be stored.

A chronological record of the amounts of oil pumped into the cavern is shown in enclosure 13. Enclosure 14 presents the results of the last sonar survey for this cavern.

Enclosure 15 provides the specification of the fuel oil. Further data has not been provided.

3.3 Etzel site - historical data

3.3.1 Leaching/oil fill/brine displacement operations

Leaching operations were conducted with an average freshwater injection rate of about 300 m³/h. This corresponds to an average volume increase of about 1 000 m³/d.

During the sump phase of cavern construction, 422 m³ of fuel oil EL were used as a blanket for the protection of the cavern roof. This fuel oil had a density of 0.835 kg/l at 12° C, a viscosity of from 1.3 - 2.6 cP at 20° C, and contained less than 1 % sulfur. As soon as the roof had been completed, the partial filling of crude oil began.

Partial oil filling

The upper area of the cavern attains the maximum permissible diameter during the roof leaching phase. Following completion of the roof, the cavern was measured by sonar survey, the 10 3/4" casing was set deeper and crude oil was injected into the upper area.

The cavern was then continuously developed in accordance with the specific volume diagram, as determined from the planned oil filling and withdrawal procedure.

This partial filling was accomplished in two ways:

- a) Oil was continuously injected during the leaching process through the blanket pipeline suspended in the 13 3/8" - 10 3/4" annulus space.

However, this method does not allow the injection rate to exceed $.40 \text{ m}^3/\text{h}$.

- b) Alternatively, oil was injected, at intervals, through the $13 \frac{3}{8}" - 10 \frac{3}{4}"$ annulus space.

Based on the leaching concept, the interval injection method described above was found to be more suitable. However, to effect injection by this method, leaching had to be interrupted as there were only two pipelines (for freshwater and brine) from the pump station manifold to the cavern.

The crude oil arriving from the NWO tanks in Wilhelms-haven was injected by the main pumps, through the freshwater pipeline into the cavern. During this process, the brine was displaced through the 7" brine casing and flowed through the normal processing path to the oil separator, the brine pond, and the brine disposal system.

The oil injection rates depend on

- the capacity of the pipelines,
- the depth of the cavern and
- the maximum flowrate through the oval wheel meter

and vary between 200 and $250 \text{ m}^3/\text{h}$.

Freshwater injection

During brine displacement, salt crystals grow on the inside of the 7" tubing. Heavy salt deposits form, particularly in the area of the wellhead, as this is the area of lowest temperature.

An increase in wellhead pressure and a decrease in flow rate indicate salt crystallization and injection is halted. Approx. 200 m³ of freshwater are injected into the 7" tubing to dissolve the salt crystals. After 1/2 to 1 hour this water is drained into the brine disposal system and oil injection can be continued.

Final oil filling

After the planned cavern capacity had been achieved, the leaching pipe combination was removed and 8 5/8" and 2 3/8" pipes installed.

Freshwater may be added to the brine at intervals during the final oil-filling phase as well as during the later oil storage and brine disposal phases. This water is pumped in through the 2 3/8" brine tubing at a rate of about 40 m³/h.

The freshwater injection rate depends on the number of caverns being serviced, the frictional pressure losses in the 2 3/8" tubing, and on the "on-field" pipeline system.

Temperature and gamma logs

Temperature and gamma-gamma logs were frequently run on all caverns at Etzel. The level of the oil/brine interface was measured at irregular intervals.

Three temperature logs for K 117, run in March 1977, August 1977, and August 1978, are plotted in the diagram in enclosure 16. From these temperature

profiles it may be seen that the crude oil stored in the cavern is gradually heating up. The diagram shows an average temperature increase of about $0.8^{\circ}\text{C/month}$ from March through August 1977, and of about $0.33^{\circ}\text{C/month}$ from August 1977 through August 1978. The reason for the significant decrease in the rate of reheating is thought to be due to a decrease in the differential temperature between the enclosing salt and the crude oil.

3.3.2 Preventing the brine from being contaminated with oil

During the leaching process, and also during the oil-injection phase, the blanket (fuel or crude oil) can be ejected from the cavern by

- a) solution or entrainment in the brine being displaced,
- b) overfilling the cavern and/or
- c) operating single sections of pipelines alternately with oil and sea water. However, this only happens in the freshwater pipeline in the direction of the cavern, so the cavern acts as a separator.

In order to prevent oil from flowing into the Jade Bay during the above situations the following security systems were installed in the pipeline system.

Preventing turbulence

The shoe of the $10\frac{3}{4}$ " freshwater injection tubing is maintained far enough below the brine/oil interface so that turbulence is negligible. This ensures that the amount of blanket dissolved or entrained in the brine remains below the 10 ppm level.

Oil-separating system

The brine pipelines from the caverns (as shown in enclosure 17) lead to an oil-water separator. This separator removes most of the oil from the brine and the oil is collected in an accumulation pond. The oil-free brine flows from the separator through a run-off into a brine pond, which serves as a feed basin for the brine pumps. This brine pond is fitted with a level monitor which switches the brine pumps on and off. This monitor can also switch off the main pumps if the basin is in danger of overflowing.

Oil separator

The removal of oil from waste water is primarily accomplished by a mechanical separator system, especially during continuous operations. The functioning of these gravity separators results from differences in the specific gravities of the mediums to be separated. The criteria for the effective operation of these devices are based on various factors such as flow rate, holding time, configuration of the separator, and physical characteristics of brine and oil.

Generally, DIN or API standards are used as a basis for the design and construction of separators.

The petrochemical and related industries predominantly use the API guidelines as a design basis. These guidelines include a formula for calculating the exact design of the separator.

Monitoring

The separating system is monitored by continuous sampling of the brine at the inlet of the oil separator. Moreover, the oil separator is regularly inspected for traces of oil. Brine samples are also taken from the main brine pipeline at the exit of the brine pumping station. Sampling is conducted by an automatic sampler every 15 minutes. These samplers are examined for traces of oil in a field laboratory.

Determination of the oil content in the brine

An increase in the content of oil in brine will be detected during an established sampling and analyses program. This program includes the following:

- a) 30 day composite sample of brine is prepared and analysed for mercury, cadmium and petroleum ether extractable compounds.
- b) Daily sampling, at frequent intervals is carried out to visually ascertain if there are any traces of oil.

Safety control

A safety control plan has been drawn up to cover accidents.

3.4 Lesum site - historical data

3.4.1 Determination of the oil/brine interface level and temperature logs

During leaching operations, gamma logs to monitor the level of the oil/brine interface were only run during large-scale oil injections. The leaching operations and thus the interface level monitoring consisted of daily calculations according to a differential pressure method. By this method the oil/brine interface level could be determined to an accuracy of about ± 5 m.

Two temperature logs of L 103 were provided. The temperature variations are plotted on a graph and given in enclosure 18. They are the logs from May 1972 and September 1978.

The log of May 1972 was run directly after the completion of the solution mining process. At this time, L 103 only held about 44 000 m³ ($2.7 \cdot 10^5$ bbl) of crude oil. This corresponds to an oil/brine interface depth of about 660 m. The fluctuations in the log are as expected, and are due to a number of factors.

The second log, which was run on the oil-filled cavern just before the sampling in September 1978, shows an almost uniform temperature through the crude oil. Even the transition from oil to brine, at a depth of about 752 m, can no longer be seen. The temperature of the oil in the cavern increased, between May 1972 and September 1978, by about 17.5° C.

The temperature logs of L 104 for May 1975 and September 1978 are shown in enclosure 19.

The temperature log for May 1975 was run following large oil withdrawals during the preceding 3 months, and the volume of fuel oil in the cavern fell to about 2 313 m³. The oil/brine interface level at that time was at an approximate depth of 722 m.

The temperature log of September 1978 was run directly prior to the sampling. At that time there was about 224 163 m³ of oil in the cavern and the oil/brine interface level was established as being at a depth of 881.5 m.

After emptying the cavern in early 1975, the refilling of L 104 began in July 1976 and continued until December 1977. By September 1978, the temperature of the oil in the cavern had increased to about 24°C. However, the oil had not attained a temperature equilibrium with the salt.

Because of a corresponding increase in the temperature of the brine below the oil, the depth of the interface level cannot be established from this log.

3.4.2 Separating Systems

During leaching operations separators were used to prevent solids from the caverns being carried into the river Weser with the brine. Two open separators, each having a capacity of 50 m³, were located at the cavern site.

Behind these separators there was a brine observation tank with a capacity of 1 m³. This tank was illuminated by an ultraviolet lamp to reveal oil traces. However, as visual checking for oil traces was not conducted continuously, significant amounts of oil could collect in the separators during the intervals between inspections. If this oil was noticed in time the leaching process could be interrupted and the oil layer drawn off.

Just before the water is released into the river Weser, another system eliminates any remaining oil traces. This system consists of five 20 m³ gravity-settling tanks, arranged in series. The last tank is equipped with an oil-warning alarm.

Large-scale oil spills have not occurred in the Lesum operation. The oil content of the brine was not measured at Lesum and thus, data is not available.

Sampling at the Etzel and Lesum site

After the contract had been signed, discussions with the cavern owners about information and data concerning the stored oil and peripheral processes started. Also the final development of the PRAKLA-SEISMOS sampler and its swing-out cantilever commenced.

Agreements with DOE were reached on the following items:

- 1) In order to obtain a sufficient volume for the planned chemical analyses by both DOE and KBB, a sampler capacity of 6 l was required.
- 2) The probe should be equipped with a swing-out cantilever for taking samples at a distance of 4.5 m from the perpendicular.
- 3) For Etzel K 117 between 20 and 30 samples would be necessary, and for each of the Lesum caverns between 10 and 15 would be collected.
- 4) The reference samples should be dispatched to the DOE Laboratory in Oklahoma by air freight.
- 5) Receptacles for crude and distillate fuel oil must be made of tin-plated metal; the receptacles for brine of glass.
- 6) Brine samples should also be taken at the wellhead during depressurization.

4.1 Preparatory work at Etzel

Due to the expiration of the KBB/IVG contract on August 31, 1978, it was decided to conduct sampling initially at the Etzel cavern K 117 (pictures 1 and 2).

4.1.1 Depressurization

On August 15, 1978, preparatory work began at cavern K 117 with depressurization. First, brine was drawn off into the Jade Bay at Wilhelmshaven, and then oil was pumped into other caverns.

Due to the influence of temperature and creep closure of the cavern, more oil than expected had to be pumped off. During the first four days, before the tubing string was pulled, 2 531 m³ were pumped off. In addition, 385 m³ were drawn off during the following days, while sampling was being carried out. The exact amounts removed are shown in enclosure 20.

The crude oil rises to the surface through the annular space formed by the 13 3/8" and 8 5/8" concentric pipes. When the oil reached the surface it went through a hopper mounted to the 12" ground flange and then into a flexible hose fixed to the upper extremity of the hopper (see pictures 20 and 21). The oil then flowed to a reservoir situated near the wellhead and finally, was forced into another cavern by a mobile piston pump through "on-field" blanket-oil pipes.

4.1.2 Workover operation

Workover operations commenced on August 18, with the 2 3/8" tubing being pulled. This tubing is part of completion equipment that has been in K 117 since January 1977, and is used with the 8 5/8" brine pipe for the injection of freshwater. Enclosure 21 shows the completion equipment components.

On August 21, preparatory work continued by setting a Baker bridge plug at a depth of 1 390.2 m in the 8 5/8" tubing string (8 5/8" tubing shoe is at 1 395.78 m). The exact position was determined previously by means of a Casing Collar Log (pictures 4 and 5).

After installation of the plug it was expected that the water from each pipe joint would spill at the surface as it was pulled. However, this only happened after the thirtieth pipe was pulled, because the brine column level in the 8 5/8" tubing string had dropped during previous depressurization.

Calculations showed that ground level and brine level were spaced 406.5 m apart. Since the drilling reports indicated that 30 pipes or 410.4 m did not contain any brine, it could be concluded that calculation predictions had been correct. This provides further proof that the plug was tight.

4.2 Sampling at Etzel

No temperature logs, or gamma logs for determination of the oil/brine interface level were run at the time of sampling, because relevant data was available from previous regular loggings. The depth of the oil/brine interface level was known to be 1 382.4 m.

Twenty-five samples were collected from K 117 - twenty of these came from various oil layers, 2 came from the oil/brine interface, 1 came from overspill at the cavern well head, and 2 came from the brine below the oil/brine interface.

Sampling procedure and depths

Prior to sampling K 117, the sampler was tested several times, both above ground and in another oil-filled cavern. All tests showed that the sampler was functioning efficiently (see enclosure 22).

The sampling equipment includes the following devices:

- sampling device with recording truck
- tin-plated and glass receptacles
- cleaning devices and vacuum pump
- compressed nitrogen bottle.

The sampler can be seen in pictures 8, 9, 14 and 15.

Prior to sampling, the sampler was clean and dry, since the sample container and the tubes were rinsed with a cleaning solution and emptied with the vacuum pump (see pictures 10 and 11).

The receptacles were rinsed with nitrogen and were marked appropriately.

After all preparations had been completed the probe was lowered to the individual sampling depths previously determined.

Having reached the correct depth, the cantilever swung out, the inlet valve opened for about 5 minutes, then closed, and the cantilever then swung back, and the probe was pulled back to the surface (see pictures 12 and 13).

At the surface, a hose was connected to the inlet port of the motor valve, and the hose was again connected to a nitrogen cylinder. Another hose was linked to the discharge socket of the sample container. This hose led to the sample receptacles. By opening the oil discharge tap and simultaneously opening the motor valve, thus exerting pressure by the nitrogen influx, the oil flowed into the sample receptacles and the nitrogen was displaced, passing through a small tube to the air (see enclosure 23 and pictures 16, 17, 18 and 19).

After the sample container was emptied, it was cleaned again as described earlier.

The sampling of cavern K 117 lasted from August 22 - 25, 1978. Sampling was continued overnight, and a total time of 63.5 hours was needed for 24 samples; thus an average time of 2.6 h was needed for each sample. For sampling depths and the amount of time needed see enclosures 24 and 25.

4.3 Preparatory work at Lesum

At the Lesum site, considerable preparations had to be done before sampling could start.

Three months before the oil sampling contract was signed, an extensive cavern wellhead insulation program was carried out by Mobil Oil.

All of the insulation and electrical heating elements for the valves had to be removed. For the assembly of the workover rig, pipe supports had to be taken out of the concrete.

4.3.1 Depressurization

The two caverns, L 103 and L 104, were initially depressurized by withdrawing brine, which was drained through the cross-country pipelines into the river Weser.

Following brine depressurization, crude oil (L 103) and fuel oil (L 104) were pumped off (see enclosure 26).

Because of unforeseen technical difficulties, it was impossible to pump the crude oil into large holding tanks. Therefore, a pump truck was engaged to pump the oil from cavern L 103 to another cavern via 2 3/8" pipes.

Only the fuel oil from L 104 could be pumped through the cross-country pipelines to the Mobil Oil tank farm.

4.3.2 Workover rig operation and plugging

After rigging up at L 103, a Baker bridge plug was set in the 7" tubing string to prevent the brine standing in the tubing from flowing downwards into the cavern when the pipes were pulled (picture 3).

The plug in L 104 was set directly after setting the plug in L 103 (see enclosures 27 and 28).

On September 5, 1978, the 7" pipes of L 103 began to be pulled. When each joint was unscrewed brine spilling out of the tubings made work more difficult.

4.4 Sampling at Lesum

After running gamma and temperature logs to determine the brine/oil interface level, sampling of cavern L 103 started on September 7, 1978.

Because of the experience gained during sampling at Etzel, where the sampling cycle had taken 2.6 hours, and because of the shallower depths at Lesum, sampling cycles here took on the average 1.8 hours for L 103 and 1.7 hours for L 104.

The sampling procedure was the same as that used in Etzel. From cavern L 103 twelve samples were collected: 10 of these were taken from the Arabian Light crude oil and 2 from the brine below the oil/brine interface (see pictures 6 and 7).

Fourteen samples were collected from cavern L 104. Of these 10 were taken from the fuel oil and 4 from the brine.

For sampling depths and the amount of time needed for sampling in both Lesum caverns, see enclosures 29, 30, 31 and 32.

After sampling at both caverns was completed, the workover rig was removed and reinstallation of wellheads, pipes and insulation started, and the cavern sites restored to their initial state.

5 Chemical analysis program

The objective of this contract is to provide the Department of Energy (DOE) with information regarding the probable effects of long term storage in salt cavities on the properties of crude and fuel oils.

To determine the present properties of the crude and fuel oils and to identify any changes which may have happened, a comprehensive analysis program was set up for the 31 crude oil samples from Etzel cavern K 117 and Lesum cavern L 103, for the 10 fuel oil samples from Lesum cavern L 104, and for the 9 brine samples from all 3 caverns.

To evaluate these changes the following properties were evaluated.

Water is inherently present in all crudes, and is commonly found as an emulsion, and also as suspended droplets. Disadvantages of water in crudes are well known in refineries where excessive quantities can flood distillation columns, and form virtually unbreakable emulsions. Moreover, water in conjunction with salt contributes to corrosion problems.

Sediment is also inherent in every crude, and commonly consists of chlorides, sulfates, fine sand and clay, and scale from pipelines and tanks. All these sediments can lead to different problems during refining.

Crude oils normally contain various salts, which are dissolved primarily in the water and to a much lesser extent in the oil. Salt crystals may also occur.

During solution mining and subsequent storage, there is continual contact between brine and oil at the oil/brine interface and a contact at the cavern walls, which may increase the salt content of the stored oils.

In any case, salts in oil are highly undesirable in refining, and it is important to know the concentration and the types of salt in oil.

API gravity and viscosity are measures upon which a classification of different types of crude oil is frequently made.

Also sulfur, H_2S , and mercaptan contents are important considerations in the refining procedure.

For the evaluation of mixing or stratification in the caverns, distillation and light hydrocarbon analysis are very informative.

5.1 Analyses

In order to ascertain what has happened to the oils, the following analyses were carried out:

A.	Crude oil analysis	DIN Standard
1	Water content	51582
2	Sediment	51789
3	Density with conversion to API gravity	51757

A.	Crude oil analysis, continued	DIN Standard
4	Kinematic viscosity	53015
5	Pour point	51597
6	Vapor pressure	51754
7	Total sulfur	51768
8	H ₂ S and mercaptans	51796
9	Nitrogen content	Automatic Nitrogen Analyzer
10	Salt content	51576
11	Optical density color	Spectrophotometric Absorbance
12	Neutralization no.	Titration
13	Trace metals (vanadium, nickel, copper, iron)	Atomic Absorption Spectrophotometry
14	TBP distillation	DIN 51751 and ASTM D 1160
15	Light hydrocarbon analysis	Gas Chromatography

For the results of these analyses for the crude oil samples from Etzel cavern K 117 see enclosures 33 to 59, and for Lesum cavern L 103 see enclosures 60 to 70.

B.	Distillate fuel oil analysis	DIN Standard
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1	<u>Standard specifications for fuel oil</u>
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1.1	Flame point	51758
1.2	Pour point	51597
1.3	Water content	51582
1.4	Carbon residue	51551
1.5	Ash content	51575
1.6	Distillation	51751
1.7	Viscosity	53015
1.8	Density	51757
1.9	Copper strip corrosion	51759
1.10	Total sulfur	51768

B. Distillate fuel oil analysis DIN Standard

2 Salt content 51576

3 Optical density color

Optical density "OD" color was determined spectrophotometrically by measuring the absorbance of the samples at 410 nm in a 1 cm cuvette. The fuel oil samples were not diluted prior to measuring their absorbance.

For the results of these analyses for the fuel oil samples from Lesum cavern L 104 see enclosures 71 to 74.

C. Brine

1 Oil in brine content

2 pH value

3 Salinity

For the results of these analyses for the brine samples from all caverns see enclosure 75. Additional details of the sample analysis procedures are given in the report "Comparison of the Present and Historical Analyses", June 1979.

Pictures 22 to 30 are of several of the chemical analysis procedures and instrumentation.

5.2 Special analyses procedures

Although there are no DIN standards, strictly speaking, for true boiling point distillation, and for the determination of trace metals, neutralization number, and nitrogen in crude oils, widely accepted methods of analysis were used in determining these properties.

5.2.1 TBP distillation

Initially an Engler distillation, according to DIN 51751, of the crude oil samples was performed, but it was found that this atmospheric distillation would not suffice for characterizing the boiling range of the whole crude.

During the project review meeting held in Washington in December 1978, between DOE and KBB, a distillation procedure was agreed upon which corresponds to a true boiling point distillation according to ASTM D 2892 for temperatures to 343° C (650° F). The distillation was extended to 565° C (1 050° F) using ASTM D 1160.

The procedure was planned to be carried out in three steps:

- 1) Atmospheric distillation with a column of 17 theoretical plates

Fractions:

- 1) ambient to 63° C (145° F)
- 2) 63 - 130° C (145 - 266° F)

- 2) Vacuum distillation at 40 torr with a 4 - 5 theoretical plate column

Fractions:

- 3) 130 - 191° C (266 - 376° F)
- 4) 191 - 232° C (376 - 450° F)
- 5) 232 - 343° C (450 - 650° F)

3) Vacuum distillation at 10^{-2} torr

Fractions:

- 6) 343 - 419° C (650 - 787° F)
- 7) 419 - 454° C (787 - 850° F)
- 8) 454 - 496° C (850 - 925° F)
- 9) 496 - 537° C (925 - 1 000° F)
- 10) 537 - 565° C (1 000 - 1 050° F)
- 11) > 565° C (> 1 050° F)

Because only 2 l of each sample remained for TBP distillation, a series of test distillations was run with crude oil from the wellhead of Etzel cavern K 117. Three different initial amounts, 3.5 kg, 2 kg, and 1 kg, were distilled in order to find out if there would be any differences in the results.

These tests showed that the results are very similar (see enclosure 76) and it was decided to distill the samples using an initial charge of 1 000 g, except for Etzel samples 21 and 22 for which only 580 g were available.

5.2.2 Atomic absorption spectrophotometry (AAS)

The trace metals vanadium, copper, nickel, and iron were determined by flameless atomic absorption spectrophotometry (AAS).

For the determination of vanadium, the following method was employed:

The crude oil samples were diluted with a benzene-toluene mixture at a ratio of 80 : 20. By means of an automatic sampler, 20 μ l of the diluted sample was injected into the graphite tube. With

the temperature program, the sample was heated to 150° C for 20 seconds to vaporize the solvent. After that, the temperature was slowly raised to 600° C. In doing so, the higher boiling oil portions were separated and cracked. After 20 seconds, the temperature was increased to 1 300° C, effectively ashing the organic matrix. The atomization ensued at 2 700° C in connection with a simultaneous measurement of the atomic absorption.

By means of the process calculator integrated with the instrument, the absorbance determined by the detector was gauged. The concentrations are thus directly read and printed out. After that, another analysis could be performed.

For the determination of copper, nickel and iron, additional temperature programs were developed and applied.

The examination of the oils turned out to be difficult, as considerable interferences occurred due to the organic matrix.

Within a series of measurements, the values showed a heavy drift. Corrections were made using standards which were periodically measured during the series.

Particular samples were analyzed according to the method of standard addition.

By an exact observance of the working conditions, however, the relative standard deviation of the repeated measurements was lowered to 3 %.

5.2.3 Determination of the total amount of nitrogen in crude oil by use of an automatic nitrogen analyzer

Initially, determination of the total amount of nitrogen in the crude oil was attempted using the Kjeldahl method. However, unforeseen difficulties arose with this method. Unusually large amounts of foam formed on the crude oils, leading to a systematic error which could not be eliminated, even when the test conditions were changed.

For this reason the samples were reanalyzed using the automatic nitrogen analyzer EA 415 from the firm Heraeus (Hanau). The measurement results using this method were adequately reproducible. The instrument was calibrated using appropriate standards.

Method

The crude oil sample is quantitatively burned over copper oxide, in a stream of pure oxygen. The resulting gases then pass over metallic copper, and flow into the "acetimeter".

By this method the organically bound nitrogen is partly oxidized into nitrogen oxide, and is then reduced, over copper, to nitrogen. The acid gases, such as CO_2 are absorbed in potassium hydroxide. The nitrogen is measured volumetrically in the "acetimeter".

The apparatus consists of two parts:

- 1) the combustion chamber, and
- 2) the "acetimeter."

The whole process is automated, beginning at the time the weighed sample is inserted in the combustion chamber, and ending when the data is printed out.

The relative standard deviation was 4.21% for seven replicate measurements, while the average value was 0.19%.

A diagram of the apparatus is shown on the next page.

Heraeus

Elementary Analysis Apparatus for Determination of Nitrogen

6 Evaluation of results

Following completion of the sample collection program at the Etzel and Lesum sites, and after the conclusion of crude and fuel oil analyses at the Preussag Laboratory, four interim interpretative reports were prepared. The first three of these reports were published separately, whereas the fourth report, because of its briefness, is included as a section in this Final Report.

6.1 Comparison of present and historical data

The first interim report, entitled "A Comparison of the Present and Historical Analyses of Crude and Distillate Fuel Oil," May, 1979, includes the recent analytical results on all of the samples, as well as the historical and baseline analytical data made available by the site owners.

Because the owners, IVG and Mobil Oil AG, did not consider it necessary to conduct comprehensive, or even detailed inspection analyses on the oils at the time they were stored, little historical data were available. NWO and Mobil Oil AG were able to provide some average data, for the period 1976 - 1979, on certain of the crude oils stored at Etzel and Lesum, respectively. These data were included in the report and served as a partial basis of comparison.

Etzel cavern K 117

General comparisons for the crude oils stored in Etzel cavern K 117, that is Arabian Light, Iranian Light, Iranian Heavy, and Basrah, were tabulated and plotted (see enclosures 77 - 85). The most informative comparisons are for

- 1) density (enclosures 77 and 81),
- 2) viscosity (enclosures 78 and 82),
- 3) water and sediment (enclosure 85),
- 4) vapor pressure (enclosures 79 and 83),
- 5) neutralization number (enclosures 80 and 84).

These data indicate that a nearly total mixing of the various layers of crude oil within the cavern has occurred. Moreover, there appears to be an enrichment of relatively heavier components, acidic compounds, water and sediment, and salt in the oil/brine interface boundary layer. Trace metal concentrations are also significantly higher in this layer, relative to the overlying crude oil.

Lesum cavern L 103

Lesum cavern L 103 contains only Arabian Light crude oil. Although a general comparison can be made between the average data, provided by Mobil Oil AG and NWO, and the results of the present analyses, any conclusions could be invalid, because small variations in the original quality of the crude oil batches stored in the cavern could still exist and be reflected in the recent analyses.

Nevertheless, the data, especially that for density, viscosity, water and sediment, and vapor pressure, indicate that the crude oil in the cavern is moderately homogeneous in quality, and does not exhibit any gross deviations from the average data (see enclosures 11, 60, 61 and 62).

Lesum cavern L 104

Lesum cavern L 104 contains distillate fuel oil manufactured to meet the specifications shown in enclosure 15. No test results on the individual batches of fuel oil stored in the cavern are available, and therefore a detailed basis of comparison for the present analyses results does not exist.

However, the results of the current analyses indicate that the fuel oil still conforms to its original specifications (see enclosures 71, 72, 73 and 74).

Detailed assessments of the sample analysis results for caverns Etzel K 117, Lesum L 103, and Lesum L 104 were made in subsequent reports.

Mixing and stratification of crude oil and fuel oil in the caverns

The second interim report, entitled "The Degree of Mixing or Stratification of Individual Lots of Oil in Salt Caverns at Lesum and Etzel, West Germany," June 1979, assessed the analytical data to obtain a knowledge of the extent of mixing or stratification of the different oils as a function of depth and time.

Experience with the storage of a single type of crude oil in surface tanks shows that density stratification will develop with time. This also applies to the distribution of hydrocarbons in natural petroleum reservoirs. Therefore, it is conceivable that gravity could produce density stratification in crude oil stored in a salt solution cavern.

In connection with the cavern storage of different types of crude oil, one after the other, density stratification might occur. Also gravity could induce partial mixing if heavy crudes are stored above lighter crudes.

Some degree of mixing by convection and diffusion is to be expected. Mixing is dependent on temperature gradients in the cavern, and on the different chemical properties of the crude oils. Furthermore, turbulent mixing at the boundary layer of different batches of crude oil is possible.

The three caverns studied permitted three different assessments to be made:

- 1) Storage of a variety of generic crude oils (Etzel cavern K 117),
- 2) storage of one generic crude oil (Lesum cavern L 103), and
- 3) storage of distillate fuel oil (Lesum cavern L 104).

Etzel cavern K 117

When different generic crude oils are stored in a cavern sequentially, they will become mixed during subsequent quiescent storage. Where heavy crude oils are stored on top of light crude oils there is an acceleration of the mixing across the theoretical boundary level due to gravity. Light crude oils on top of heavy crude oils are mixed mainly by thermal convection. During longer periods of storage, diffusion, as well as density differences and convection, plays a role in the mixing.

The analytical data for the crude oil samples from Etzel cavern K 117 indicate a nearly complete mixing of the different crude oils. The batches of crude oil which have been stored in the cavern for the longest period exhibit the most complete mixing, as might be expected. The least degree of mixing appears to be with sample no. 1, from near the top of the cavern. This is probably due to the fact that this batch of crude oil has been in storage for the shortest period of time of any of the theoretical layers studied.

Moreover, the theoretical layer from which sample no. 1 was collected, as well as the layer immediately above it, have similar physical characteristics. As a result, mixing of the two layers is occurring only slowly by convection and diffusion, and not by gravity.

Density, viscosity, and vapor pressure exhibit only small variations between samples. Moreover, the results of the Engler analyses are quite similar, as are those of the TBP distillations. Large variations are seen only for samples nos. 21 and 22, which are from the oil/brine interface layer.

The gas chromatographic analyses of fractions 2 and 4 from the TBP distillations indicate no significant differences for samples nos. 2 through 22.

The trace metals vanadium and nickel exhibit only minor variations down to the interface level. Iron is somewhat more variable in concentration than either vanadium or nickel, with the greatest deviation being in the uppermost samples and in the interface region. Copper exhibits the greatest variations among the trace metals. This variation cannot be explained by stratification or mixing. Contamination of the crude oil from copper grease used on tubing joint threads, although extremely unlikely, cannot be excluded. Another, somewhat more likely source of contamination, are the brass components in the device used for sampling.

The nitrogen content of the samples does not exhibit considerable variation.

Small variations in the neutralization number, sulfur, mercaptan, and salt contents, as a function of depth, are difficult to interpret due to the analytical errors associated with the

techniques. Except for sample no. 1, the mercaptan content is essentially constant with depth. This is consistent with virtually total mixing of the crude oils in the cavern, with the exception of the Iranian Heavy oil (sample no. 1). Similarly, the sulfur content is essentially constant with depth.

Although variable with depth, the neutralization numbers for most of the crude oils are low, and hence subject to large uncertainties. In general, the top samples, nos. 1 and 2, and the oil/brine interface samples, nos. 21 and 22, are the only samples with significantly varying values. The high values for samples nos. 1 and 2 are consistent with incomplete mixing of the cavern, whereas the large increase in the interface samples reflects an increasing acid content in this layer. The increase in these samples, that is nos. 21 and 22, can be attributed to two processes. Precipitation of asphaltenes and maltenes from the stored crude oils is one possibility. Additionally, oxidation processes at the oil/brine interface may play a role.

Variations in the salt content, as a function of depth, are probably real. The salt content in the oil above the interface boundary layer depends upon

- 1) the droplet size distribution of water in the various stored batches, and
- 2) the convection currents operative in the cavern.

Because the solubility of salt in crude oils is low, salt content as a function of depth depends on the quantities and size distributions of the water in the various stored crude oil batches. If the droplet size is greater than 0.1μ , water will settle in time. Droplets with diameters less than approximately 1μ will not sediment, but, in fact, may be raised to higher levels, depending on convection cells in the cavern. Owing to the dynamic convection in the cavern, as evidenced by the apparent homogeneity of the crude oil with depth, the salt content should not remain constant with either time or depth.

The high salt concentrations in the interface samples suggest that a large quantity of the original water was present in sufficiently large droplets so that it would settle to the oil/brine interface level. However, the viscous interface layer has prevented transport of the water droplets into the brine below. In addition, entrainment of crystalline salt from the brine is possible in this region.

Based on all of the above data, the crude oils in cavern K 117 are essentially mixed, with the exception of the top layer of Iranian Heavy. The increase in the values for the interface samples reflects apparent sedimentation of asphaltenes and petroleum resins, and of water. Evidence for gravity stratification is not apparent.

Lesum cavern L 103

Lesum cavern L 103 contains only Arabian Light crude oil, and if thermal convection cells are operative, no stratification of the crude oil should occur. However, an increase, with increasing depth, in the concentration of certain non-hydrocarbon components, due to gravity settling, could be expected.

The analytical results for the crude oil samples from this cavern give little indication of stratification. Variations noted in the determinations may be attributable to original differences in the quality of the batches of Arabian Light crude oil pumped into storage. Only neutralization number, salt, and mercaptans vary significantly within the sample series. Their respective values rise with increasing depth down to the oil/brine interface layer. This increase in neutralization number and mercaptans suggests the settling of acid components of the crude oil, and the increase in salt indicates the settling of water.

Lesum cavern L 104

The distillate fuel oil samples from Lesum cavern L 104 exhibit similar analytical results, with only minor variations for certain properties within the sample series. These minor variations might be explained by differences in the quality of the original batches of fuel oil stored in the cavern.

The temperature of the fuel oil in the cavern is still relatively low, and it seems probable that thermal convection cells have not yet become operative. Mixing by diffusion is probably negligible because of the physical and chemical similarities of the different batches of fuel oil stored. Thus, it seems likely that mixing of the fuel oil has not occurred.

Conclusions

The analysis data for the crude oil samples from Etzel cavern K 117 indicate that a nearly total mixing of the different crude oils stored in the cavern has taken place, with the exception of the top layer of Iranian Heavy. The mixing is apparently caused primarily by thermal convection. Moreover, gravity mixing, due to the settling of relatively heavier oil into lighter oil, apparently has occurred in some instances. The magnitude of values for samples from the oil/brine interface layer reflects the sedimentation of asphaltenes and petroleum resins, and of water. Evidence for stratification is not apparent.

The Arabian Light crude oil in Lesum cavern L 103 exhibits questionable stratification. Variability noted in the results may be attributable to original differences in the quality of the crude oil. Only neutralization number, salt, and mercaptan values exhibit significant variation, and these are likely the result of normal settling processes. The quality of the crude oil has not undergone any deleterious changes.

The distillate fuel oil in Lesum cavern L 104 is not totally mixed. It still shows, after a storage period of two years, differences in properties, which are probably attributable to differences in the quality of the original batches of fuel oil pumped into storage.

6.3 Extent and stability of the emulsions
in the oil/brine interface layer

The third interim report, June 1979, is entitled "The Extent and Stability of Emulsions in Salt Caverns at Lesum and Etzel, West Germany, Following Prolonged Storage of Crude and Distillate Fuel Oils." This report, in addition to presenting an overview of the mechanisms contributing to the formation and stabilization of oil/water emulsions, assesses further the recent analytical results for certain samples with respect to emulsions.

Etzel cavern K 117

In Etzel cavern K 117, a 2 meter thick viscous layer exists at the oil/brine interface. This layer, which accounts for less than approximately 0.5 percent of the volume of crude oil stored in the cavern, contains 4.6 percent water and 0.9 percent sediment. Normal-alkane waxes are also suspected to be present in greater than usual amounts. The viscosity and relatively large concentration of water in this layer are believed to be indicative of a true water-in-oil emulsion.

This layer also has a relatively large mercaptan content, a high neutralization number, and is enriched in trace metals. These data suggest that trace metals have been precipitated concurrently with the flocculation of acidic petroleum colloids, especially the asphaltenes.

Clay particles, inherently present in crude oil, could adhere to and stabilize these floccules. The resulting aggregates, in addition to water and other sediment, would settle to the oil/brine interface under gravity. Asphaltenes and other colloidal substances are known to reduce interfacial tension, and would tend to stabilize water-in-oil emulsions at the interface.

Flocculation of colloids, and concurrent precipitation of trace metals, could be caused by the commingling of different crude oils, if the colloid systems of the different oils are incompatible. The concentration of water and sediment in the interface layer probably results from gravity settling. An enrichment of normal-alkane waxes in the boundary layer could result from incompatibility, gravity settling, or precipitation induced by contact of the warm oil with cooler brine during the early stages of cavern fill.

Lesum cavern L 103

Although a sample of the oil/brine interface layer apparently was not obtained from Lesum cavern L 103, there is evidence for the existence of a layer similar to the one in Etzel cavern K 117.

The brine tubing pulled from this cavern during the recent sampling had approximately a 1.2 meter long coating of a viscous material directly at the oil/brine interface. Moreover, a gamma log also indicated the existence of such a layer.

A noticeable increase in the concentrations of salt, and water and sediment, and an increase in the viscosity of the lowermost oil sample from this cavern, indicate that it was collected from the upper fringes of a refractory boundary layer. These trends suggest the existence of a layer similar to the one in Etzel cavern K 117.

If a layer, analogous to the one in Etzel cavern K 117, exists in Lesum cavern L 103, the supposition that this layer results, at least in part, from incompatibility of different crude oil colloid systems would be negated. Lesum cavern L 103 contains only a single crude oil, namely Arabian Light.

Lesum cavern L 104

The analyses of the samples of distillate fuel oil stored in Lesum cavern L 104 gave no indications of the formation or existence of a refractory layer or emulsion at the oil/brine interface.

Conclusions

The results of these studies indicate that a refractory layer forms, with time, at the brine/oil interface in a salt solution cavern containing crude oil. This layer is considered to be a water-

in-oil emulsion, although the water contents observed are somewhat below the levels typically thought of as being necessary for a true emulsion. On the basis of sonar and gamma log surveys it seems that this refractory layer apparently forms in all crude oil storage caverns, whether they contain a mixture or a single type of crude oil. The layer is characterized by a relatively high viscosity, a significant enrichment in trace metals and salt, and from 5 to 6 percent water and sediment. It is likely that asphaltenes and n-alkane waxes are also concentrated in the layer, contributing to its increased viscosity.

The refractory layer in Etzel cavern K 117 was sampled directly. However, it appears that the layer in Lesum cavern L 103 was not sampled, but it is known from sonar surveys, gamma logs, and the presence of a viscous coating on the brine tubing string at the level of the oil/brine interface that a refractory layer exists. In both caverns this layer comprises only about 0.5 percent of the amounts of hydrocarbons stored.

No emulsion was observed directly or indirectly in Lesum cavern L 104, which contains distillate fuel oil.

Knowledge of the extent of an emulsion layer is necessary for optimum withdrawal of crude oil from a cavern. Normally, an emulsion of small extent would not be withdrawn, but would be left in a cavern to serve as a blanket layer. If it

became necessary to completely withdraw the oil from the cavern it could probably be accomplished. The produced emulsion could possibly be processed using existing dewatering and desalting technology. Refiners who normally process heavy crudes would be able to handle the relatively larger content of n-alkanes and asphaltenes likely to be present.

The formation of oil-in-water emulsions just below the oil/brine interface in caverns is not critical, as these emulsions have a low viscosity and are relatively unstable in the presence of brine.

6.4 Chemical contamination of the brine below the oil/brine interface level

When petroleum oils are stored in salt-solution caverns, the oil is in direct contact with the brine at the oil/brine interface. During cavern fill cycles, and also during quiescent storage, hydrocarbons can become dispersed and/or dissolved in the brine. When the brine is displaced to the surface, as during leaching operations or cavern fill, the dispersed or dissolved hydrocarbons can pose a potential environmental impact. For this reason, consideration of the chemical contamination of brine is of importance in the operation of salt-solution caverns for oil storage.

In order to fully assess the potential for an environmental impact, it is necessary to know the quantity and composition of the organic substances present in the brine existing below crude oil in a storage cavern.

The solubility of hydrocarbons in water decreases with increasing salinity. In freshwater at a temperature of 20°C, the solubilities for selected hydrocarbons are

n-hexane	10	ppm
n-heptane	3	ppm
n-octane	0.6	ppm
benzene	1 780	ppm
toluene	515	ppm
ethylbenzene	150	ppm

As can be seen from these data, solubility decreases with increasing chain-length and increasing number and complexity of side-chains. The naphthenic and aromatic hydrocarbons are generally more soluble than the paraffinic hydrocarbons of the same carbon number.

Dissolved hydrocarbons, ranging from methane to pentane, are frequently found in petroleum associated brines. Higher molecular weight hydrocarbons may be dissolved or dispersed in brines under certain conditions, but the concentrations are generally low and the solutions or dispersions are relatively unstable.

Brine samples from caverns

During solution mining at Etzel, samples were collected regularly from the brine coming from the caverns, and these samples were analyzed for their oil content. As the injection of crude oil into the caverns was concurrent with solution mining, a relatively higher concentration of hydrocarbons was expected for these brine samples than for samples collected following cessation of solution-mining operations.

During oil sampling operations at Etzel, two samples, nos. 23 and 24, were collected from the brine below the oil/brine interface level in cavern K 117. For Lesum cavern L 103, samples nos. 11 and 12 were taken from the brine layer, and for Lesum cavern L 104, samples nos. 10 through 14 were obtained from the brine.

Analyses of the brine samples

The chloride content of each sample was determined by classical Mohr titration, and the results reported as grams/liter sodium chloride (NaCl). These determinations indicate that the brine is saturated with respect to NaCl.

The pH of the brine samples, as determined by standard methods, was essentially neutral, with values in the range 6.8 to 7.2.

Enclosure 75 contains the analytical results on pH-value and salinity for the brine samples from Etzel cavern K 117 and from Lesum caverns L 103 and L 104.

Petroleum acids are relatively insoluble in neutral or acidic brines, consequently contamination of the cavern brines by these compounds is unlikely.

For the determination of dissolved hydrocarbons, the brine samples were extracted with carbon tetrachloride, and the extracts analyzed by infrared spectrophotometry. Only a weak CH absorption band at $2\,930\text{ cm}^{-1}$ was observed and the paraffinic hydrocarbon content was estimated to be $< 0.1\text{ ppm}$ for all of the brine samples from both Lesum and Etzel. In contrast, brine samples taken during solution mining had hydrocarbon contents of from 0.4 to 1.5 ppm (see enclosures 86 and 87).

No organic compounds other than hydrocarbons were detected by infrared spectrophotometry.

Enclosure 88 contains a typical analysis of a brine sample taken during solution mining of Etzel cavern K 117.

Conclusions

The solubility of paraffinic hydrocarbons in saturated brine is generally below 0.1 ppm. Aromatic hydrocarbons have somewhat higher solubilities.

During solution mining, minute oil droplets may become dispersed in the brine as a result of turbulence. However, due to the absence of surfactants in the brine, permanent emulsions do not develop and the dispersed oils are rapidly "salted out" and rise to the oil/brine interface. Petroleum acids are relatively insoluble in neutral or acidic brines, as typically exist in salt-solution caverns.

Consequently, chemical contamination of the brine below the oil/brine interface will be relatively insignificant during quiescent storage.

Conclusions

Although a number of countries, notably West Germany and France, have had strategic petroleum reserves established for a number of years, apparently little consideration has been given to the long-term quality stability of the hydrocarbons stored.

It is a generally held view that long-term storage will not result in degradation of crude oil, but until now this opinion had not been rigorously tested. Distillate fuel oils are known to degrade when stored for prolonged periods in an oxygenated environment, such as a surface storage tank. However, prior to these studies virtually nothing was known about the degradation of these oils during prolonged storage in salt caverns where oxygen is absent.

In the United States, pursuant to the requirements of the Energy Policy and Conservation Act of 1975, the Department of Energy (DOE) is implementing a Strategic Petroleum Reserve (SPR). The SPR, when completed, will contain up to one billion (one million million) barrels of crude oil, which may remain in quiescent storage for as long as 25 years. Because of the sizeable investment in the SPR and the uncertainties regarding long-term hydrocarbon stability, DOE initiated the studies conducted by KBB to obtain definitive data on the behaviour and stability of crude and distillate fuel oils during long-term storage.

Three caverns at two different sites in West Germany were selected for sample collection and study. At the Etzel site, cavern K 117 contains a variety of light and heavy crude oils, which were stored sequentially. At the Lesum site, cavern L 103 contains only a single generic crude, and cavern L 104 contains distillate fuel oil.

A total of 50 crude and fuel oil and brine samples were collected from these three caverns and subsequently analyzed in accordance with a comprehensive study program. From these studies the following results were obtained, and conclusions drawn.

7.1 Etzel cavern K 117 - variety of generic
crude oils

A nearly total mixing of the different crude oils in this cavern has taken place during the two to three years of storage. Only the most recently stored crude has not yet completely mixed with the underlying crudes.

The mixing is apparently caused primarily by thermal convection. Moreover, gravity mixing, due to the settling of relatively heavier oil into lighter oil, apparently has occurred in some instances. Diffusion probably contributes to the mixing process. The analytical results for density, viscosity and vapor pressure exhibit little variation with depth, down to the oil/brine interface boundary layer. Water and sediment contents are negligible down to the boundary layer, indicating that

these components settle out under gravity with time. The values for neutralization number, trace metals, and mercaptans suggest a settling-out or flocculation of acidic petroleum colloids, including asphaltenes, with concurrent precipitation of trace metals.

No changes have occurred which would prevent the crude mixture from being processed using existing refining technology.

7.2 Lesum cavern L 103 - single generic crude oil

Only Arabian Light crude oil is stored in Lesum cavern L 103, and the bulk of this oil has been in undisturbed storage for more than six years.

The analytical results for the crude oil samples from this cavern give little indication of stratification. Density, viscosity, and vapor pressure values exhibit little variation with depth, down to the oil/brine interface boundary layer. Neutralization number, mercaptans, and salt generally increase with increasing depth, suggesting the gravity settling of acidic components and entrained water, as in Etzel cavern K 117.

Overall, the crude oil is moderately homogeneous in quality and does not exhibit any significant deviation from what is expected for Arabian Light. The quality of the crude oil has not undergone any deleterious changes.

7.3 Lesum cavern L 104 - distillate fuel oil

Lesum cavern L 104 had contained distillate fuel oil in essentially undisturbed storage for approximately 2 years, at the time it was sampled for these studies.

The fuel oil samples from this cavern exhibit similar analytical results, with only minor variations for certain properties within the sample series. These minor variations can be explained by differences in the quality of the original batches of fuel oil stored in the cavern.

The temperature of the fuel oil in the cavern is still relatively low, and it seems probable that thermal convection cells have not yet become operative. Mixing by diffusion is probably negligible. It is likely that mixing of the fuel oil batches has not occurred.

The results of the samples analyses indicate that the fuel oil still conforms to its original specifications and is a marketable product.

7.4 Emulsion development at the oil/brine interface

With time, a viscous layer, containing appreciable quantities of water, sediment, and salt, and enriched in trace metals and the acidic components of crude oil, such as asphaltenes and mercaptans, develops at the oil/brine interface in crude oil

storage caverns. It is thought that this layer also contains n-alkane waxes in greater than usual amounts. The viscosity and water content of this layer are believed to be indicative of a true emulsion. This layer comprises only about 0.5 percent of the volume of hydrocarbons stored in caverns Etzel K 117 and Lesum L 103.

Gamma logs and sonar surveys in other crude oil storage caverns have indicated the presence of a refractory layer at the oil/brine interface level. It appears that this layer forms in all crude oil storage caverns whether they contain a single generic crude or a mixture of crudes.

This is probably a natural process, involving the settling of flocculated petroleum resins and concurrently precipitated trace metals, as well as inherent sediment and water. The acidic petroleum resins, such as asphaltenes, and clays are known to stabilize emulsions.

No emulsion layer was found in Lesum cavern L 104, which contains distillate fuel oils. Fuel oils generally do not contain surfactants or water in sufficient quantity to promote the formation of water-in-oil emulsions, and oil-in-water emulsions are unstable, especially under the conditions of temperature and salinity existing in salt-solution caverns.

7.5 Contamination of brine underlying crude oil

Contamination of the brine below the oil/brine interface level, as a consequence of solution or dispersion of hydrocarbons or organic derivatives of hydrocarbons will be negligible during long-term storage. The solubility of hydrocarbons in brines is typically very low. Due to the absence of surfactants in the brine, permanent emulsions will not develop, and any dispersed oils will rapidly "salt out" and rise to the oil/brine interface. Petroleum acids are relatively insoluble in neutral or acidic brines, as typically exist in salt-solution caverns.

Recommendations

As a result of these studies, the following recommendations can be made:

- 1) Detailed analytical data should be obtained on each batch of crude oil to be stored in a cavern. These baseline data are essential for comparative purposes in future studies on the quality of the stored oils.
- 2) Ideally, only a single generic crude oil should be stored in any one cavern. If different crude oils are to be commingled they should have similar physical and chemical properties, including hydrocarbon-type composition.
- 3) The viscous layer which appears likely to develop at the oil/brine interface in crude oil caverns should not be withdrawn, but should be left in the cavern to serve as a blanket layer.
- 4) Another sampling and analysis program in two to three years, using the results of these studies as a comprehensive data base, would provide greater knowledge of the effects of long-term storage in salt caverns on the quality of crude and distillate fuel oils.

Amounts of Blanket/Crude Oil Filled into K 117

Date	Quantity (m ³)	Quantity cumulative (m ³)	Type of oil	Remarks
7/74	1.7	1.7	Blanket	ex blanket tank
8/74	128.9	130.6	Blanket	ex blanket tank
9/74	4	134.6	Blanket	ex blanket tank
10/74	25	137.1	Blanket	ex blanket tank
12/74	1.6	138.7	Blanket	ex blanket tank
1/75	2.7	141.4	Blanket	ex blanket tank
2/75	- 25.9	115.5	Blanket	back to blanket tank
3/75	- 70.4	45.1	Blanket	back to blanket tank
4/75	8	53.1	Blanket	ex blanket tank
7/75	5.8	58.9	Blanket	ex blanket tank
8/75	363.1	422	Blanket	ex blanket tank
9/75	350	772	Arabian Light	ex K 111
9/75	520	1,292	Arabian Light	ex K 101
10/75	130	1,422	Arabian Light	ex K 111
10/75	900	2,322	Arabian Light	ex K 111
11/75	1,200	3,522	Arabian Light	ex K 102
11/75	2,784	6,306	Arabian Light	ex K 102
11/75	5,298	11,604	Arabian Light	ex K 111
12/75	3,201	14,805	Arabian Light	ex K 111
12/75	4,446	19,251	Arabian Light	ex K 111
1/76	1,997	21,248	Iran Light	from NWO
1/76	6,044	27,431	Iran Light	from NWO
1/76	6,183	33,475	Iran Light	from NWO
1/76	11,480	44,955	Basrah	from NWO
1/76	11,541	56,496	Basrah	from NWO
1/76	38,062	94,558	Basrah	from NWO
1/76	4,573	99,131	Arabian Light	from NWO

Date	Quantity (m ³)	Quantity cumulative (m ³)	Type of oil	Remarks
2/76	- 1,696	97,435	Arabian Light	into K 105
3/76	494	97,929	Blanket	ex K 125
3/76	250	98,179	Arabian Light	ex K 111
3/76	272	98,451	Basrah	ex K 110
3/76	1,026	99,477	Arabian Light	ex K 111
3/76	612	100,089	Iran Light	ex K 124
4/76	5,320	105,409	Arabian Light	ex K 132
4/76	540	105,949	Iran Light	ex K 124
4/76	331	106,280	Arabian Light	ex K 132
4/76	3,354	109,634	Arabian Light	from NWO
4/76	5,636	115,270	Arabian Light	from NWO
4/76	4,794	120,064	Arabian Light	from NWO
4/76	5,370	125,434	Arabian Light	from NWO
4/76	5,399	130,833	Arabian Light	from NWO
5/76	17,134	147,967	Arabian Light	from NWO
5/76	2,436	150,403	Arabian Light	from NWO
6/76	31,006	181,409	Iran Heavy	from NWO
7/76	7,322	188,731	Iran Heavy	from NWO
7/76	24,906	213,637	Arabian Light	from NWO
8/76	- 1,497	212,140	Arabian Light	into K 132
8/76	1,099	213,239	Arabian Light	ex K 105
8/76	830	214,069	Arabian Light	ex K 114
9/76	26,540	240,609	Iran Heavy	from NWO
9/76	6,784	247,393	Iran Heavy	from NWO
9/76	3,655	251,048	Iran Heavy	from NWO
9/76	14,555	265,603	Iran Heavy	from NWO
9/76	11,785	277,388	Iran Heavy	from NWO
9/76	30,343	307,731	Iran Heavy	from NWO
10/76	389	308,120	IVG-Pool	ex K 127
11/76	3,297	311,417	IVG-Pool	ex K 127
11/76	2,114	313,531	IVG-Pool	from NWO
11/76	6,589	320,120	IVG-Pool	ex K 127

Date	Quantity (m ³)	Quantity cumulative (m ³)	Type of oil	Remarks
12/76	12,786	332,906	Iran Heavy	from NWO
12/76	13,543	346,449	Iran Heavy	from NWO
12/76	14,497	360,946	Iran Heavy	from NWO
12/76	4,401	365,347	IVG-Pool	from NWO
12/76	11,541	376,888	IVG-Pool	from NWO
12/76	6,763	383,651	IVG-Pool	from NWO
12/76	14,821	398,472	IVG-Pool	from NWO
12/76	17,345	415,817	IVG-Pool	from NWO
1/77	2,875	418,692	Arabian Light	from NWO
1/77	5,683	424,375	Arabian Light	from NWO
1/77	12,531	436,906	Arabian Light	from NWO
1/77	18,750	455,656	Arabian Light	from NWO
1/77	4,991	460,647	Arabian Light	from NWO
3/78	- 2,581	458,066	Arabian Light	from NWO

Site Etzel

IVG-Pool

Delivery → NWO → Etzel

Date	→ NWO-Tanks Crude oil (m ³)	NWO → Etzel Crude Oil (m ³)
September 76	27,780	
September 76	28,072	
September 76	28,161	
September 76	27,843	
September 76		28,060
September 76	28,222	27,832
September 76		28,158
September 76	27,995	28,218
October 76	27,826	
October 76		27,999
October 76	27,986	27,785
October 76	28,317	
October 76		27,810
October 76	27,979	27,967
October 76	27,821	28,303
October 76		27,804
October 76	28,226	27,616
October 76	27,874	
October 76		28,155
October 76	27,593	28,018
October 76	28,001	27,605
October 76	28,003	27,993
October 76	20,243	27,994
October 76	27,631	20,250
October 76	26,041	26,051

Date	→ NWO-Tanks Crude Oil (m ³)	NWO → Etzel Crude Oil (m ³)
October 76	17,241	23,955
October 76		25,998
October 76	27,999	11,001
October 76	28,083	
October 76	26,067	
October 76	20,904	
November 76	25,299	
November 76	30,815	
November 76		32,519
November 76		28,288
November 76		28,363
November 76		27,761
November 76		26,346
December 76	26,276	
December 76	26,488	
December 76	25,958	
December 76		26,269
December 76		26,479
December 76	25,931	
December 76		25,957
December 76	26,514	25,934
December 76	25,952	26,413
December 76	25,276	25,890
December 76	16,948	25,260
December 76		20,105
December 76		16,975
January 77	26,450	
January 77	26,226	
January 77	26,100	
January 77		26,444
January 77	25,897	26,202

Date	→ NWO-Tanks Crude Oil (m ³)	NWO → Etzel Crude Oil (m ³)
January 77	26,390	
January 77	26,159	26,149
January 77		25,852
January 77	26,057	26,395
January 77	25,921	
January 77	26,422	21,314
January 77	21,332	26,029
January 77		25,939
January 77	26,015	26,446
January 77	25,874	26,144
January 77		26,021
January 77	26,429	
January 77		25,872
January 77	26,125	
January 77	26,006	
January 77	25,920	
January 77	20,011	
January 77	12,789	
February 77	36,452	
March 77		27,251
March 77		25,724
March 77		26,532
March 77		26,505
March 77		22,647
May 77		5,415
May 77		39,870

Site Etzel

Oil shipments to NWO

Date	Vessel	Type of Oil	Quantity in m ³	DIN Density at 15°C kg/l	API Gravity at 60°F
October 74	Nausicaa	Nigerian Light	61,257	0.836	37.7
November 74	Golar Togo	Khafji	93,409	0.886	28.1
December 74	Friesland	Arabian Light	137,526	0.85	35
January 75	Golar Togo	Khafji	93,992	0.8846	28.37
February 75	Friesland	Basrah	93,604	0.85	35
September 75	Esso-Kawasaki	Arabian Light	273,758	0.8589	33.16
October 75	Daring	Arabian Medium	155,119	0.8714	30.79
November 75	Westfalen	Arabian Light	263,038	0.86	33
November 75	Jedforest	Iran Heavy	168,493	0.8715	30.78
November 75	Ronitz	Iran Heavy	94,206	0.8725	30.59
November 75	Odenfeld	Iran Heavy	57,449	0.8721	30.66
November 75	Odenfeld	Iran Heavy	48,162	0.8721	30.66
December 75	Margaret-Simone	Iran Heavy	92,047	0.8706	30.95
December 75	Egmont	Arabian Light	253,241	0.863	33.9

ENCLOSURE 4

Date	Vessel	Type of Oil	Quantity m ³	DIN Density at 15°C kg/l	API Gravity at 60°F
January 76	World Horizon	Arabian Light	257,859	0.8571	33.51
January 76	Sea Swift	Basrah	284,077	0.8542	34.07
January 76	Adele	Iran Light	166,981	0.8566	33.61
May 76	Taurus	Iran Heavy	135,036	0.8704	30.98
June 76	Taurus	Iran Heavy	137,016	0.8697	31.11
August 76	British Resolution	Iran Heavy	278,889	0.8718	30.72
September 76	Shinko-Marui	Iran Heavy	259,693	0.8706	30.95
September 76	Esso-Norway	Arabian Light	23,974	0.8602	33.05
November 76	Fairfield Sunrise	Iran Heavy	254,349	0.8713	30.81
January 77	Kolbjorg	Arabian Light.	261,269	0.8575	33.43
July 77	Gresham	Arabian Heavy	240,903	0.8883	27.71

Amounts and types of crude oil pumped into cavern no. 117

No.	Storing Dates	Quantity (m ³)	Type of oil	Cumulative quantity (m ³)
1	July 74 to August 75	422	Blanket	422
2	September 75 to December 75	18,829	Arabian Light	18,829
3	January 76	14,224	Iranian Light	14,224
4	January 76	61,083	Basrah	61,083
5	January 76 to February 76	2,877	Arabian Light	21,706
6	March 76	494	Blanket	916
7	March 76	250	Arabian Light	21,956
8	March 76	272	Basrah	61,355
9	March 76	1,026	Arabian Light	22,982
10	March 76	612	Iranian Light	14,836
11	April 76	5,320	Arabian Light	28,302
12	April 76	540	Iranian Light	15,376
13	April 76 to June 76	44,454	Arabian Light	72,756
14	June 76 to July 76	32,328	Iranian Heavy	38,328
15	July 76 to August 76	25,338	Arabian Light	98,094
16	September 76	93,662	Iranian Heavy	131,990
17	October 76 to November 76	12,389	IVG-Pool	12,389
18	December 76	40,826	Iranian Heavy	172,816
19	December 76	54,871	IVG-Pool	67,260
20	January 77	42,249	Arabian Light	140,343

Cavity Caliper of K 117

No.	Depth region from...to (m)	Depth Intervals (m)	Diameter (m)
0	0 - 777	777	0.33
1	777 - 818.4	41.4	0.5
2	818.4 - 856	37.6	5 - 13
3	856 - 880.4	24.4	38.1
4	880 - 909.5	29.1	38.7
5	909.5 - 920.9	11.4	37.3
6	920.9 - 923.8	2.9	39.3
7	923.8 - 928.5	4.7	41.7
8	928.5 - 935.6	7.1	43.1
9	935.6 - 942.8	7.2	42.6
10	942.8 - 950.9	8.1	40.7
11	950.9 - 956.6	5.7	38.6
12	956.6 - 961.8	5.2	32.5
13	961.8 - 970	8.2	39.1
14	970 - 980.4	10.4	33.4
15	980.4 - 986.1	5.7	33.7
16	986.1 - 998.1	12	31.1
17	998.1 - 1,009.7	11.6	31.2
18	1,009.7 - 1,030.9	21.2	36.3
19	1,030.9 - 1,032.4	1.5	32.8
20	1,032.4 - 1,048	15.6	33.9
21	1,048 - 1,058	10	34.2
22	1,058 - 1,068.5	10.5	33.5
23	1,068.5 - 1,089.7	21.2	33.1
24	1,089.7 - 1,105.9	16.2	31.9
25	1,105.9 - 1,126.3	20.4	33.1

No.	Depth region from...to (m)	Depth Intervals (m)	Diameter (m)
26	1,126.3 - 1,147	20.7	28
27	1,147 - 1,160.7	13.7	29.4
28	1,160 - 1,183.9	23.2	31.7
29	1,183.9 - 1,209.8	25.9	30.5
30	1,209.8 - 1,221	11.2	29.6
31	1,221 - 1,254.7	33.7	29.2
32	1,254.7 - 1,282.7	28	29.6
33	1,282.7 - 1,286	3.3	27.5
34	1,286 - 1,332	46	28
35	1,332 - 1,360	28	25.9
36	1,360 - 1,377.5	17.5	27.1

Oil Layer Thickness
of Cavity No. 117

No.	Charge of oil (m ³)	Depth region from - to (m)	Layer thickness (m)	Type of oil
1	422	1,377.5 - 1,378.2	0.7	Blanket
2	18,829	1,344.6 - 1,377.5	32.9	Arabian Light
3	14,224	1,319.8 - 1,344.6	24.8	Iranian Light
4	61,083	1,226.6 - 1,319.8	93.2	Basrah
5	2,877	1,222.3 - 1,226.6	4.3	Arabian Light
6	494	1,221.6 - 1,222.3	0.7	Blanket
7	250	1,221.2 - 1,221.6	0.4	Arabian Light
8	272	1,220.8 - 1,221.2	0.4	Basrah
9	1,026	1,219.3 - 1,220.8	1.5	Arabian Light
10	612	1,218.4 - 1,219.3	0.9	Iranian Light
11	5,320	1,210.7 - 1,218.4	7.7	Arabian Light
12	540	1,209.9 - 1,210.7	0.8	Iranian Light
13	44,454	1,150.2 - 1,209.9	59.7	Arabian Light
14	38,328	1,098.5 - 1,150.2	51.7	Iranian Light
15	25,338	1,068.4 - 1,098.5	30.1	Arabian Light
16	93,662	965.3 - 1,068.4	103.1	Iranian Heavy
17	12,389	953.3 - 965.3	12.0	IVG-Pool
18	40,826	923.5 - 953.3	29.8	Iranian Heavy
19	54,871	876.1 - 923.5	47.4	IVG-Pool
20	42,249	0 - 876.1	876.1	Arabian Light

Site Lesum

Cavern L 103

Amounts of Arabian Light Crude Oil

Date	pumped in (m ³)	Amounts of Crude pumped out (m ³)	Cumulative quantity (m ³)
April 71			8,646
May 71	11,953		20,599
June 71	4,000		24,599
July 71	3,000		27,599
August 71	3,004		30,603
January 72		10,451	20,152
February 72	3,437	4,017	19,572
March 72		85	19,487
April 72			26,118
May 72	20,801		44,707
June 72	35,335	43	79,999
July 72	40,982		120,981
August 72	51,141		172,122
September 72	42,190		214,314
October 72	39,668		249,388
November 72	23,255		272,643
December 72	20,404		293,047
January 73	10,294	16,277	287,064
February 73	4,180		291,244
March 73	50	2,261	289,033
April 73	4,037	1,785	291,285
May 73	928		292,213
July 73		14,423	277,790
October 73			278,130
December 73	13,111		291,241

Date	pumped in (m ³)	Amounts of Crude pumped out (m ³)	Cumulative quantity (m ³)
January 74	15,119	37,426	268,934
February 74	2,500	14,742	281,176
May 74		153	281,023
June 74	193		281,216
August 74	153		281,369
September 74	158		281,784
February 75	22		281,806
July 75	4,848		286,654
August 75	1,596		288,250
September 75	775		289,025
October 75	5,845		294,870
November 75	998		295,868
December 75		2	295,866
January 76		324	295,542
March 76		9	295,533
May 76	47		295,580

Characteristics of Whole Crude

Properties

Bureau of Mines Classification-Intermediate

Identity

Crude: Arabian Light
Country of Origin: Saudi Arabia
Type Sample: Cargo
Tanker: General Colocotronis
Port and Date: Paulsboro - January 8, 1972

Gravity, °API	34.7
Specific gravity, 60/60°F	0.8514
Distillation	M - 1040
	°F
IBP	60
5 pct vol recovered	150
10 "	213
20 "	305
30 "	394
40 "	483
Fraction 400-500°F, pct vol	30.5 - 41.8
	11.3
Mercaptan Sulphur, ppm	171
Flash point, °F	< 0
Pour point, °F, (upper)	- 65
Viscosity:	
Kinematic, 60°F (15.6°C) cSt	10.25
100°F (37.8°C) cSt	5.90
130°F (54.4°C) cSt	4.28

Saybolt Universal, 60°F (15.6°C) s	50.6	
100°F (37.8°C) s	45.3	
130°F (54.4°C) s	40.2	
Redwood no. 1, 100°F (37.8°C), s	40.1	
Carbon residue, pct wt	3.74	
Aniline point, °F	158.5	
Sulphur, pct wt	1.70	
*Hydrogen sulfide, ppm	0.0	
Neutralization no., total acid (D664)	0.0	
*Water and sediment, pct vol	0.10	
*Salt content, pounds/1000 bbl	23.1	
*Reid vapor pressure, psia	4.3	
Nitrogen, total, ppm	770	
Nickel, ppm	3.2	
Vanadium, ppm	13	
Carbon, pct wt	85.8	
Hydrogen, pct wt	12.8	
Carbon/hydrogen ratio	6.70	
**U.O.P. characterization factor "K"	11.7	
*Light hydrocarbon analysis:	pct wt	pct vol
Ethane and lighter	0.00	0.0
Propane	0.15	0.25
Iso-butane	0.15	0.23
N-butane	1.02	1.69
C4 and lighter	1.32	2.17
Iso-pentane	0.71	0.97
N-pentane	1.57	2.12
Total C5	2.28	3.09
C5 and lighter	3.60	5.26

*may be influenced by manner of sampling and sample handling

**estimated

Site Lesum

90

Cavern L 104

Amounts of Fuel Oil

Date	pumped in (m ³)	Amounts of Fuel Oil pumped out (m ³)	Cumulative quantity (m ³)
December 71	68		68
January 72	10		78
February 72	46.6		124.6
April 72	125		149.6
May 72	150		399.6
June 72	250.4		650
August 72	300		950
September 72	400		1,350
November 72	206		1,556
December 72	194		1,750
February 73	20		1,770
March 73		50	1,720
May 73	4,724	100	6,344
June 73	36,760		43,104
July 73	11,323		54,427
September 73	8,934	200	63,161
October 73	37,634	100	100,695
November 73	36,214	1,532	135,377
December 73	41,072		176,449
January 74	5,909	100	182,258
February 74	5,867		188,125
March 74	11,892		200,017
May 74	3,370		203,387
June 74		60	203,327
August 74		79	203,248
September 74		1,142	202,106

Date	pumped in (m ³)	Amounts of Fuel Oil pumped out (m ³)	Cumulative quantity (m ³)
October 74	8,377	130	210,353
November 74		450	209,903
December 74	24,042	1,300	232,645
January 75		15,649	216,996
February 75	13,037	59,405	170,628
March 75		87,866	82,762
April 75		52,249	30,513
May 75	435	28,635	2,313
September 75	450		2,763
October 75		48	2,715
November 75		469	2,246
April 76	32		2,278
July 76	26,081		28,359
August 76	6,882		35,241
September 76	18,039		53,280
October 76	55,669	1,729	107,220
November 76	22,794	15	129,999
December 76	24,387		154,386
January 77	34,258		188,644
February 77	8,872	174	197,342
March 77		13	197,329
April 77		392	196,937
June 77		390	196,547
October 77	12,434	45	208,936
November 77		17	208,919
December 77	15,244		224,163

Site Lesum

Fuel Oil

Black and White Product Specification

Additives: Conductivity additive RT 748 C (as required to reach abt. 100 c.u. at 0 °C (October - March)
or at 12 °C (April - September)
Fluidity improver (optional)

Tests	Limits		Methods
Density at 15° C	g/ml	min. 0.810 max. 0.855	DIN 51 757
<u>Combustion</u>			
Smoke point	mm	min. 18	ASTM D 1322
Gross calorific value	kcal/kg	min. 10 100	DIN 51 900
Conradson carbon res.	wt. %	max. 0.05	DIN 51 551
Ash (Oxide)	ppm	max. 50	DIN 51 575
<u>Volatility</u>			
Distillation			DIN 51 751
at 230 °C	Vol. % rec.	min. 10	
at 250 °C	Vol. % rec.	max. 64	
at 350 °C	Vol. % rec.	min. 75	

Tests		Limits	Methods
<u>Fluidity</u>			
Viscosity at 20 °C	cSt	min. 2.5	max. 5.0
		<u>North and West</u>	
		March 1 - October 31	November 1 - February 28
Cold filter plugging point (CFPP)	°C	max. - 7	max. - 10
Cloudpoint	°C	max. + 3	max. ± 0
		<u>South and Southwest</u>	
		March - November 20	November 21 - February
CFPP	°C	max. - 7	max. - 11
Cloudpoint	°C	max. + 3	max. - 1
Pour point		to be reported	
<u>Cleanliness and Purity</u>			
Appearance		clear and bright *	
Color (ASTM)		max. 2.0	
Odor		not objectionable	
Water (non-settling)	wt. %	max. 0.1	
Sediment (extraction)	wt. %	max. 0.01	
Neutralization No.	mg KOH/g	max. 0.2	

* Corresponds to a maximum water content of 100 ppm
DIN 51 603 requires max. 0.1 wt. % (1,000 ppm)

Tests		Limits	Methods
<u>Corrosiveness</u>			
Sulfur	wt. %	max. 0.50	DIN 51 768
Mercaptan Sulfur	ppm	max. 100	ASTM D 1323
Mercaptan factor		max. 130	MM 39/524 *
Copper corrosion	(3 h at 50 °C)	max. 1	DIN 51 759
<u>Storage</u>			
Accelerated stability	mg/100 ml	to be reported	ASTM D 2274
<u>Safety</u>			
Flash point, AP/PM	°C	min. 56	DIN 51 755

* MM = Mobil Method

TEMPERATURE LOGS

SITE ETZEL
Cavern No. 11713 1/2 m. Csg. at: 777 m
Cavity Volume: 458 650 m³Temp. Logs from: 11 March 1977
21 August 1977
31 August 1978

Log from March, 8, 77

Log from August, 18, 77

Log from August, 24, 78

FLOW SHEET - ETZEL

TEMPERATURE LOGS

18 7/8" csm. Csg. at 537.1m
13 7/8" Csg. at 717.1m
Cavity Volume on
May 1975 : 353700m³

SITE LESUM
L 104

Temperature Logs from
a) May 1975
b) Sept. 1978

Temperature
15°C 20°C 25°C 30°C 35°C

Log from
May 14, 1975

Log from
Sept. 12, 1978

700m
710m
720m
730m
740m
750m
760m
830m
840m
850m
860m
870m
880m
920m

Etzel Site

Depressurization of the K 117 cavern
for oil sampling

Date	Amount withdrawn (m ³)	Fluid
15.08.78	161.0	brine
15.08.78	140.0	crude oil
16.08.78	913.0	crude oil
17.08.78	788.0	crude oil
18.08.78	690.0	crude oil
20.08.78	154.0	crude oil
21.08.78	67.6	crude oil
22.08.78	55.0	crude oil
23.08.78	47.0	crude oil
24.08.78	43.0	crude oil
25.08.78	18.4	crude oil

2 916.0 m³ of crude oil were pumped off during depressurization so that the oil volume of the cavern then amounted to 455 149.014 m³.

ETZEL SITE
Cavern K 117 Completion

SAMPLING DEVICE FOR OIL SAMPLING

Encl. 22

102

JULY 78 Nie/M

FILLING PROCEDURE

103

for sample receptacles

ENCL. 23

No	Depth m	Time of sampling h	Oil Temperature °C	Remarks
1	978	3.8	28	gas sample *
2	1 064	2.3	27.5	
3	1 074	2.0	26.5	
4	1 094	2.3	24.6	gas sample
5	1 104	2.5		gas sample
6	1 145	2.0	**	
7	1 155	2.3		
8	1 200	2.5		gas sample
9	1 240	2.7		gas sample
10	1 315	2.7	23.5	
11	1 325	2.5	22.5	gas sample
12	1 340	2.0	24.6	
13	1 350	2.0	23.5	gas sample
14	1 370	2.3	22.6	
15	1 084	2.0	19.6	gas sample
16	1 125	2.3	19.5	
17	1 180	2.3	18.3	gas sample
18	1 270	2.3	21.6	
19	1 376	3.5	21.5	gas sample
20	1 378	4.3	22.5	
21	1 380	3.5	24.5	*** brine
22	1 382	3.3		*** brine
23	1 398	3.0		*** brine
24	1 403	3.0		*** brine

Remarks

* Some gas samples were taken in order to find out if depressurisation caused gas escape during decanting.

** Temperature of the oil was measured after the sample receptacles were filled, if sufficient oil remained in the probe.

*** Sampling conducted with central probe without cantilever.

EXACT SAMPLING DEPTHS

SITE ETZEL

CAVERN No. 117

CAVITY CONFIGURATION DETERMINED BY
AMOUNTS OF OIL PUMPED IN

Lesum Site

Depressurization of the L 103 and L 104 caverns
for oil sampling

As no metering device is installed, the amounts of brine withdrawn from both caverns during depressurization could not be determined.

An amount of

$$V_{\text{oil}} = 465 \text{ m}^3 \text{ (crude oil)}$$

was pumped from the L 103 cavern into the L 201 cavern during depressurization of the L 103.

In addition, another 399 m^3 were pumped off by suction trucks during the sampling procedure.

185 m^3 of fuel oil were pumped off from the L 104 cavern for depressurization before sampling took place. During sampling no oil emerged.

LESUM SITE
CAVERN L 103
COMPLETION

107

LESUM SITE

108

CAVERN L 104

COMPLETION

Sampling at Lesum - L 103

No	Depth (m)	Time of sampling (h)	Density (kg/m ³)	Temperature (°C)	Remarks
1	630	2.5	849	19.5 **	
2	645	1.5			
3	660	1.3	847	17.2	
4	675	1.3	846	15.0	gas sample *
5	690	1.3	852	17.3	
6	705	1.6	849	15.5	
7	720	1.5	850	17.0	
8	735	1.9	850	20.0	gas sample
9	749	1.6	848	21.0	
10	751	3.7			
11	758	2.0	1 204	22.5	brine
12	762	1.0	1 201	21.5	brine

Remarks

If density and temperature are not given, the relevant samples did not have sufficient residual oil.

* Some gas samples were taken in order to find out if depressurisation caused gas escape during decanting.

** Temperature of the oil was measured after the sample receptacles were filled, if sufficient oil remained in the probe.

Sampling at Lesum - L 104

No	Depth (m)	Time of sampling (h)	Density (kg/m ³)	Temperature (°C)	Remarks
1	730	1.5	836	15.8 *	
2	745	1.3			
3	760	1.2			
4	775	1.3			
5	790	1.8		16.8	**
6	805	1.1		16.5	
7	820	1.5		17.0	
8	835	1.3			
9	850	1.6			
10	880.5	1.3			
11	882.5	3.5	1 202		brine
12	883.5	1.5	1 208	21.0	brine
13	885.5	1.6	1 207	21.0	brine
14	893.5	3.2	1 204	20.0	brine

* Temperature of the oil was measured after the sample receptacles were filled, if sufficient oil remained in the probe.

** Gas samples were not taken, as no degassing of the samples was evident.

EXACT SAMPLING DEPTHS

111

SITE LESUM

CAVERN L103

CRUDE OIL

EXACT SAMPLING DEPTHS

SITE LESUM

CAVERN L 104

FUEL OIL

S 45° W

N 45° E

25m

0

25m

50m

18 7/8" CEMENTED CASING SHOE - 537.1m

13 7/8" CSG AT 717.13m

1 730m

2 745m

3 760m

4 775m

5 790m

6 805m

7 820m

8 835m

9 850m

10 860.5m

11 862.5m

12 864.5m

13 866.5m

14 868.5m

SONAR SURVEY NO. 6

FROM MAY 1975

CAVITY VOLUME 353,700m³

OIL CONTENT 22% 163 m³

OIL - BRINE INTERFACE - 881.5m

FROM SEPT 1978

Etzel Site

Cavern No. 117

Table: Water and Sediment in Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Water ²⁾ content (% wt)	Sediment (% wt)
1	978	Iranian Heavy	traces	< 0.01
2	1 064		traces	< 0.01
3	1 074	Arabian Light	traces	< 0.01
15	1 084		traces	< 0.01
4	1 094		traces	< 0.01
5	1 104	Iranian Heavy	traces	< 0.01
16	1 125		traces	< 0.01
6	1 145		traces	< 0.01
7	1 155	Arabian Light	traces	< 0.01
17	1 180		traces	< 0.01
8	1 200		traces	< 0.01
9	1 240	Basrah	traces	< 0.01
18	1 270		traces	< 0.01
10	1 315		traces	< 0.01
11	1 325	Iranian Light	traces	< 0.01
12	1 340		traces	< 0.01
13	1 350	Arabian Light	traces	< 0.01
14	1 370		traces	< 0.01
19	1 376		traces	< 0.01
20	1 378	Oil/Brine Interface Region	traces	< 0.01
21	1 380		3.9	0.83
22	1 382		4.6	0.91
Oil from overflow during sampling			traces	< 0.01

1) Stratification see also enclosure 25

2) Traces means less than 0.1 %

Crude Oil Analysis

114

Etzel Site

Cavern No. 117

Table : Density of Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Density at 20 ^o C (kg/m ³)	API-Gravity ²⁾ at 60 ^o F
1	978	Iranian Heavy	859	32.56
2	1 064		860	32.36
3	1 074	Arabian Light	860	32.36
15	1 084		860	32.36
4	1 094		860	32.36
5	1 104	Iranian Heavy	860	32.36
16	1 125		859	32.59
6	1 145		860	32.36
7	1 155	Arabian Light	860	32.36
17	1 180		859	32.59
8	1 200		860	32.36
9	1 240	Basrah	860	32.36
18	1 270		859	32.59
10	1 315		860	32.36
11	1 325	Iranian Light	860	32.36
12	1 340		860	32.36
13	1 350	Arabian Light	860	32.36
14	1 370		860	32.36
19	1 376		860	32.36
20	1 378	Oil/Brine Interface	860	32.36
21	1 380		868	30.86
22	1 382		873	29.96
Oil from overflow during sampling			860	32.36

1) Stratification see also enclosure 25

2) Density converted to API-Gravity

ENCLOSURE 34

Crude Oil Analysis

Etzel Site

Cavern No. 117

2)

Table: Mercaptan, H_2S and Total Sulfur

Sample No.	Depth (m)	Theoretical ¹⁾ Stratification of Crude Oil	Mercaptan (mg/kg)	Total Sulfur (wt%)
1	978	Iranian Heavy	58	1.88
2	1 064		91	1.88
3	1 074	Arabian Light	100	2.05
15	1 084		92	1.83
4	1 094		98	1.97
5	1 104	Iranian Heavy	91	1.87
16	1 125		95	1.84
6	1 145		103	1.81
7	1 155	Arabian Light	106	2.01
17	1 180		97	1.91
8	1 200		95	1.94
9	1 240	Basrah	89	2.12
18	1 270		94	1.88
10	1 315		97	1.90
11	1 325	Iranian Light	81	1.86
12	1 340		81	1.86
13	1 350	Arabian Light	80	1.85
14	1 370		94	1.85
19	1 376		96	1.89
20	1 378	Oil/Brine Interface Region	102	1.90
21	1 380		92	1.83
22	1 382		109	1.80
Oil from overflow during sampling			151	1.83

1) Stratification see also enclosure 25

2) H_2S was analyzed qualitatively, all results were negative

Etzel Site

Cavern No. 117

Table: Pourpoint of Samples

Sample No.	Depth (m)	Theoretical Stratification of Crude Oil ¹⁾	Pour point (upper)	
			°C	°F
1	978	Iranian Heavy	- 19	- 2.2
2	1 064		- 20	- 4.0
3	1 074	Arabian Light	- 18	- 0.4
15	1 084		- 15	5.0
4	1 094		- 18	- 0.4
5	1 104	Iranian Heavy	- 21	- 5.8
16	1 125		- 13	8.6
6	1 145		- 14	6.8
7	1 155	Arabian Light	- 20	- 4.0
17	1 180		- 12	10.4
8	1 200		- 15	5.0
9	1 240	Basrah	- 14	6.8
18	1 270		- 11	12.2
10	1 315		- 17	1.4
11	1 325	Iranian Light	- 19	- 2.2
12	1 340		- 20	- 0.4
13	1 350	Arabian Light	- 16	3.2
14	1 370		- 16	3.2
19	1 376		- 10	14.0
20	1 378	Oil/Brine Interface Region	- 9	15.8
21	1 380			
22	1 382			
Oil from overflow during sampling			- 21	- 5.8

1) Stratification see also enclosure 25

Crude Oil Analysis

Etzel Site

Cavern No. 117

Salt Content

Sample No.	Depth (m)	Theoretical ¹⁾ Stratification of Crude Oil	Salt Content (mg/kg)
1	978	Iranian Heavy	85
2	1 064		159
3	1 074	Arabian Light	105
15	1 084		85
4	1 094		86
5	1 104	Iranian Heavy	102
16	1 125		110
6	1 145		97
7	1 155	Arabian Light	122
17	1 180		86
8	1 200		123
9	1 240	Basrah	111
18	1 270		110
10	1 315		183
11	1 325	Iranian Light	90
12	1 340		95
13	1 350	Arabian Light	100
14	1 370		138
19	1 376		178
20	1 378	Oil/Brine Interface Region	186
21	1 380		14 586
22	1 382		24 983
Oil from overflow during sampling			55

1) Stratification see also enclosure 25

Etzel Site

Cavern No. 117

Table: Viscosity of Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Kinematic at 25°C (77°F) (cSt)	Viscosity at 50°C (122°F) (cSt)
1	978	Iranian Heavy	11.57	5.96
2	1 064		10.85	5.60
3	1 074	Arabian Light	10.85	5.48
15	1 084		10.74	5.60
4	1 094		10.85	5.60
5	1 104	Iranian Light	10.62	5.60
16	1 125		10.63	5.71
6	1 145		10.74	5.60
7	1 155	Arabian Light	10.74	5.60
17	1 180		10.75	5.60
8	1 200		10.62	5.60
9	1 240	Basrah	10.74	5.48
18	1 270		10.86	5.60
10	1 315		10.85	5.60
11	1 325	Iranian Light	10.85	5.60
12	1 340		10.85	5.60
13	1 350	Arabian Light	10.85	5.60
14	1 370		11.20	5.60
19	1 376		11.32	5.71
20	1 378	Oil/Brine Interface Region	11.32	5.71
21	1 380		-	9.08
22	1 382		-	9.50
Oil from overflow during sampling			11.55	5.60

1) Stratification see also enclosure 25

Etzel Site

Cavern No. 117

Table: Neutralization Number

Sample No.	Depth (m)	Theoretical ¹⁾ Stratification of Crude Oil	Neutralization Number (mgKOH/g)
1	978	Iranian Heavy	0.44
2	1 064		0.49
3	1 074	Arabian Light	0.28
15	1 084		0.11
4	1 094		0.49
5	1 104	Iranian Heavy	0.22
16	1 125		0.17
6	1 145		0.22
7	1 155	Arabian Light	0.16
17	1 180		0.05
8	1 200		0.17
9	1 240	Basrah	0.20
18	1 270		0.00
10	1 315		0.25
11	1 325	Iranian Light	0.11
12	1 340		0.10
13	1 350	Arabian Light	0.11
14	1 370		0.11
19	1 376		0.00
20	1 378	Oil/Brine Interface Region	0.05
21	1 380		1.50
22	1 382		2.43
Oil from overflow during sampling			0.86

1) Stratification see also enclosure 25

Crude Oil Analysis

120

Etzel Site

Cavern No. 117

Table: Vapor Pressure of Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Vapor pressure ² (bar)	(psia)
1	978	Iranian Heavy	0.29	4.2
2	1 064		0.35	5.1
3	1 074	Arabian Light	0.36	5.2
15	1 084		0.36	5.2
4	1 094		0.35	5.1
5	1 104	Iranian Heavy	0.36	5.2
16	1 125		0.35	5.1
6	1 145		0.37	5.3
7	1 155	Arabian Light	0.36	5.2
17	1 180		0.35	5.1
8	1 200		0.37	5.3
9	1 240	Basrah	0.36	5.2
18	1 270		0.35	5.1
10	1 315		0.36	5.2
11	1 325	Iranian Light	0.37	5.3
12	1 340		0.36	5.2
13	1 350	Arabian Light	0.35	5.1
14	1 370		0.36	5.2
19	1 376		0.36	5.2
20	1 378	Oil/Brine Interface Region	0.36	5.2
21	1 380		0.29	4.2
22	1 382		0.29	4.2
Oil from overflow during sampling			0.35	5.1

1) Stratification see also enclosure 25

2) Pressure in bar converted to psia

ENCLOSURE 40

Crude Oil Analysis

Etzel Site

Cavern No. 117

Table: Optical Density Color

Sample No.	Depth (m)	Theoretical Stratification of Crude Oil ¹⁾	Optical Density Color Extinction ²⁾
1	978	Iranian Heavy	1.003
2	1 064		0.972
3	1 074	Arabian Light	0.971
15	1 084		0.982
4	1 094		0.968
5	1 104	Iranian Heavy	0.965
16	1 125		0.973
6	1 145		0.983
7	1 155	Arabian Light	0.966
17	1 180		0.975
8	1 200		0.966
9	1 240	Basrah	0.986
18	1 270		0.981
10	1 315		0.966
11	1 325	Iranian Light	0.981
12	1 340		0.978
13	1 350	Arabian Light	0.961
14	1 370		0.979
19	1 376		0.960
20	1 378	Oil/Brine Interface Region	0.972
21	1 380		1.207
22	1 382		1.269
Oil from overflow during sampling			0.939

1) Stratification see also enclosure 25

2) Absorbance measured at 410 nm

Crude Oil Analysis

Etzel Site

Cavern No. 117

Table: Nitrogen Content

Sample No.	Depth (m)	Theoretical ¹⁾ Stratification of Crude Oil	Nitrogen Content (wt%)
1	978	Iranian Heavy	0.195
2	1 064		0.166
3	1 074	Arabian Light	0.165
15	1 084		0.156
4	1 094		0.158
5	1 104	Iranian Heavy	0.153
16	1 125		0.157
6	1 145		0.159
7	1 155	Arabian Light	0.157
17	1 180		0.153
8	1 200		0.157
9	1 240	Basrah	0.154
18	1 270		0.152
10	1 315		0.155
11	1 325	Iranian Light	0.154
12	1 340		0.155
13	1 350	Arabian Light	0.156
14	1 370		0.154
19	1 376		0.152
20	1 378		0.158
21	1 380	Oil/Brine Interface Region	0.230
22	1 382		0.164

1) Stratification see also enclosure 25

Crude Oil Analysis

Etzel Site

Cavern No. 117

Table: Trace Metals - Copper

Sample No.	Depth m	Theoretical Stratification of Crude Oil *	Copper Concentration	
			.mg/l	ppm
1	978	Iranian Heavy	1.10	1.29
2	1 064		1.91	2.22
3	1 074	Arabian Light	0.28	0.33
15	1 084		0.09	0.10
4	1 094		0.11	0.13
5	1 104	Iranian Heavy	0.12	0.14
16	1 125		0.07	0.09
6	1 145		0.05	0.06
7	1 155	Arabian Light	0.08	0.09
17	1 180		0.05	0.06
8	1 200		0.18	0.21
9	1 240	Basrah	0.05	0.06
18	1 270		0.05	0.06
10	1 315		0.31	0.36
11	1 325	Iranian Light	0.84	0.98
12	1 340		0.12	0.13
13	1 350	Arabian Light	0.04	0.04
14	1 370		0.12	0.13
19	1 376		0.09	0.10
20	1 378	Oil/Brine Interface Region	0.15	0.17
21	1 380			5.31
22	1 382			9.12

* Stratification see also enclosure 25

Crude Oil Analysis

Etzel Site

Cavern No. 117

Table: Trace Metals - Iron

Sample No.	Depth m	Theoretical * Stratification of Crude Oil	Iron Concentration ppm
1	978	Iranian Heavy	14.50
2	1 064		7.85
3	1 074	Arabian Light	1.34
15	1 084		1.49
4	1 094		1.88
5	1 104	Iranian Heavy	1.37
16	1 125		1.62
6	1 145		1.50
7	1 155	Arabian Light	1.35
17	1 180		1.34
8	1 200		1.34
9	1 240	Basrah	1.32
18	1 270		1.34
10	1 315		1.44
11	1 325	Iranian Light	1.80
12	1 340		1.53
13	1 350	Arabian Light	1.42
14	1 370		3.75
19	1 376		1.85
20	1 378	Oil/Brine Interface Region	1.83
21	1 380		330.00
22	1 382		893.00

* Stratification see also enclosure 25

Crude Oil Analysis

Etzel Site

Cavern No. 117

Table: Trace Metals - Vanadium

Sample No.	Depth m	Theoretical Stratification of Crude Oil *	Vanadium Concentration	
			mg/l	ppm
1	978	Iranian Heavy	27.73	32.25
2	1 064		26.40	30.71
3	1 074	Arabian Light	23.87	27.77
15	1 084		24.30	28.26
4	1 094		23.79	27.67
5	1 104	Iranian Heavy	24.23	28.18
16	1 125		25.20	29.31
6	1 145		24.44	28.43
7	1 155	Arabian Light	24.30	28.26
17	1 180		22.98	26.73
8	1 200		23.70	27.57
9	1 240	Basrah	23.61	27.43
18	1 270		23.27	27.07
10	1 315		23.72	27.59
11	1 325	Iranian Light	23.98	27.89
12	1 340		23.77	27.65
13	1 350	Arabian Light	22.97	26.7
14	1 370		23.01	26.76
19	1 376		24.42	28.40
20	1 378	Oil/Brine Interface Region	23.96	27.86
21	1 380			36.88
22	1 382			63.82

* Stratification see also enclosure 25

ENCLOSURE 45

Crude Oil Analysis

126

Etzel Site
Cavern No. 117

Table: Trace Metals - Nickel

Sample No.	Depth m	Theoretical * Stratification of Crude Oil	Nickel Concentration	
			mg/l	ppm
1	978	Iranian Heavy	15.70	18.26
2	1 064		16.38	19.05
3	1 074	Arabian Light	15.70	18.26
15	1 084		15.12	17.58
4	1 094		16.62	19.33
5	1 104	Iranian Heavy	15.22	17.70
16	1 125		17.00	19.78
6	1 145		15.93	18.53
7	1 155	Arabian Light	15.07	17.53
17	1 180		14.46	16.82
8	1 200		14.41	16.77
9	1 240	Basrah	13.76	16.01
18	1 270		15.47	17.99
10	1 315		15.09	17.55
11	1 325	Iranian Light	16.01	18.62
12	1 340		15.54	18.08
13	1 350	Arabian Light	15.39	17.91
14	1 370		14.59	17.09
19	1 376		14.87	17.29
20	1 378		15.62	18.17
21	1 380	Oil/Brine Interface Region		18.24
22	1 382			24.97

* Stratification see also enclosure 25

Crude Oil Distillation
 Site Etzel, Cavern No. 117

Sample No. 2, Depth 1 064 m, initial quantity of 900 g

Fraction	Column Temperature from...to °C	Temperature from...to °F	Pressure torr	Amount of Crude Oil fraction g	cumulative g	Remarks	wt%
1	< 35 - 63	< 95 - 145	760	29.6	29.6	Fraction 1:	3.29
2	63 - 130	145 - 266	760	75.3	104.9	Fraction 2:	8.37
3	130 - 191	166 - 376	40	83.2	188.1	Fraction 3:	9.24
4	191 - 232	376 - 450	40	57.0	245.1	Fraction 4:	6.33
5	232 - 343	450 - 650	40	173.4	418.5	Fraction 5:	19.27
6/7	343 - 454	650 - 850	10 ⁻²	42.7	461.2	Fraction 6/7:	4.74
8	454 - 496	850 - 925	10 ⁻²	63.2	524.4	Fraction 8:	7.02
9	496 - 537	925 - 1 000	10 ⁻²	69.7	594.1	Fraction 9:	7.74
10	537 - 565	1 000 - 1 050	10 ⁻²	36.9	631.0	Fraction 10:	4.10
11	565 - 572	1 050 - 1 062	10 ⁻²	85.0	761.0	Fraction 11:	9.44

Distillate amount: 716.0 g = 79.56 wt%

Residue: 170.0 g = 18.89 wt%

Loss: 14.0 g = 1.55 wt%

Crude Oil Distillation

Site Etzel, Cavern No. 117

Sample No. 3, Depth 1 074 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C	Column Temperature from...to °F	Pressure torr	Amount of Crude Oil fraction g	Amount of Crude Oil cumulative g	Remarks	wt%
1	< 35 - 63	< 95 - 145	760	36.8	36.8	Fraction 1:	3.68
2	63 - 130	145 - 266	760	84.4	121.2	Fraction 2:	8.44
3	130 - 191	266 - 376	40	90.6	211.8	Fraction 3:	9.06
4	191 - 232	376 - 450	40	66.4	278.2	Fraction 4:	6.64
5	232 - 343	450 - 650	40	189.9	468.1	Fraction 5:	18.99
6/7	343 - 454	650 - 850	10^{-2}	46.8	514.9	Fraction 6/7:	4.68
8	454 - 496	850 - 925	10^{-2}	94.6	609.5	Fraction 8:	9.46
9	496 - 537	925 - 1 000	10^{-2}	53.7	663.2	Fraction 9:	5.37
10	537 - 565	1 000 - 1 050	10^{-2}	43.2	706.4	Fraction 10:	4.32
11	565 - 572	1 050 - 1 062	10^{-2}	87.6	794.0	Fraction 11:	8.76

Distillate amount: 794.0 g = 79.40 wt%

Residue: 182.0 g = 18.20 wt%

Loss: 24.0 g = 2.4 wt%

Crude Oil Distillation
Site Etzel, Cavern No. 117

Sample No. 8, Depth 1 200 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C	Column Temperature from...to °F	Pressure torr	Amount of Crude Oil fraction g	Amount of Crude Oil cumulative g	Remarks	wt%
1	< 35 - 63	< 95 - 145	760	37.8	37.8	Fraction 1:	3.78
2	63 - 130	145 - 286	760	88.5	126.3	Fraction 2:	8.85
3	130 - 191	266 - 376	40	85.2	211.5	Fraction 3:	8.52
4	191 - 232	376 - 450	40	63.6	275.1	Fraction 4:	6.36
5	232 - 343	450 - 650	40	196.3	471.4	Fraction 5:	19.63
6/7	343 - 454	650 - 850	10^{-2}	72.9	544.3	Fraction 6/7:	7.29
8	454 - 496	850 - 925	10^{-2}	82.2	626.5	Fraction 8:	8.22
9	496 - 537	925 - 1 000	10^{-2}	41.6	668.1	Fraction 9:	4.16
10	537 - 565	1 000 - 1 050	10^{-2}	34.2	702.3	Fraction 10:	3.42
11	565 - 572	1 050 - 1 062	10^{-2}	91.8	794.1	Fraction 11:	9.18

Distillate amount: 794.1 g = 79.41 wt%

Residue: 181.0 g = 18.10 wt%

Loss: 24.9 g = 2.49 wt%

Crude Oil Distillation

Site Etzel, Cavern No. 117

Sample No. 12, Depth 1 340 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C		from...to °F		Pressure torr	Amount of Crude Oil fraction g	cumulative g	Remarks	wt%
1	< 35	63	< 95	145	760	30.1	30.1	Fraction 1:	3.01
2	63	130	145	266	760	87.3	117.4	Fraction 2:	8.73
3	130	191	266	376	40	93.4	210.8	Fraction 3:	9.34
4	191	232	376	450	40	62.1	272.9	Fraction 4:	6.12
5	232	343	450	650	40	196.9	469.8	Fraction 5:	19.69
6/7	343	454	650	850	10 ⁻²	15.4	485.2	Fraction 6/7:	1.54
8	454	496	850	925	10 ⁻²	111.8	597.0	Fraction 8:	11.18
9	496	537	925	1 000	10 ⁻²	68.0	665.0	Fraction 9:	6.80
10	537	565	1 000	1 050	10 ⁻²	34.6	699.6	Fraction 10:	3.46
11	565	572	1 050	1 062	10 ⁻²	96.9	796.5	Fraction 11:	9.69

Distillate amount: 796.5 g = 79.65 wt%

Residue: 183.0 g = 18.3 wt%

Loss: 20.5 g = 2.05 wt%

ENCLOSURE 50

Crude Oil Distillation
Site Etzel, Cavern No. 117

Sample No. 14, Depth 1 370 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C		from...to °F		Pressure torr	Amount of Crude Oil fraction cumulative g g		Remarks	wt%
1	< 35	- 63	< 95	- 145	760	36.6	36.6	Fraction 1:	3.66
2	63	- 130	145	- 266	760	83.0	119.6	Fraction 2:	8.30
3	130	- 191	266	- 376	40	90.8	210.4	Fraction 3:	9.08
4	191	- 232	376	- 450	40	66.1	276.5	Fraction 4:	6.61
5	232	- 343	450	- 650	40	195.4	471.0	Fraction 5:	19.54
6/7	343	- 454	650	- 850	$2 \cdot 10^{-2}$	87.1	559.0	Fraction 6/7:	8.71
8	454	- 496	850	- 925	$2 \cdot 10^{-2}$	61.9	620.9	Fraction 8:	6.19
9	496	- 537	925	- 1 000	$2 \cdot 10^{-2}$	73.7	694.6	Fraction 9:	7.37
10	537	- 565	1 000	- 1 050	$2 \cdot 10^{-2}$	38.5	733.1	Fraction 10:	3.83
11	565	- 572	1 050	- 1 062	10^{-1}	56.5	789.6	Fraction 11:	5.65

Distillate amount: 789.6 g = 78.96 wt%

Residue: 193.0 g = 19.3 wt%

Loss: 17.4 g = 1.74 wt%

ENCLOSURE 51

Crude Oil Distillation
Site Etzel, Cavern No. 117

Sample No. 15, Depth 1 084 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C	from...to °F	Pressure torr	Amount of Crude Oil fraction g	cumulative g	Remarks	wt%
1	< 35 - 63	< 95 - 145	760	38.2	38.2	Fraction 1:	3.82
2	63 - 130	134 - 266	760	88.4	126.6	Fraction 2:	8.84
3	130 - 191	266 - 376	40	91.7	218.3	Fraction 3:	9.17
4	191 - 232	376 - 450	40	67.6	285.9	Fraction 4:	6.76
5	232 - 343	450 - 650	40	188.4	474.3	Fraction 5:	18.84
6/7	343 - 454	650 - 850	10^{-2}	68.4	542.7	Fraction 6/7:	6.84
8	454 - 496	850 - 925	10^{-2}	83.7	626.4	Fraction 8:	8.37
9	496 - 537	925 - 1 000	10^{-2}	78.0	704.4	Fraction 9:	7.80
10	537 - 565	1 000 - 1 050	$2 \cdot 10^{-2}$	42.7	747.1	Fraction 10:	4.27
11	565 - 572	1 050 - 1 062	10^{-1}	53.4	800.5	Fraction 11:	5.34

Distillate amount: 800.5 g = 80.05 wt%

Residue: 185.0 g = 18.50 wt%

Loss: 14.5 g = 1.45 wt%

Crude Oil Distillation
 Site Etzel, Cavern No. 117

Sample No. 20, Depth 1 378 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C	Column Temperature from...to °F	Pressure torr	Amount of Crude Oil fraction g	Amount of Crude Oil cumulative g	Remarks	wt%
1	< 35 - 63	< 95 - 145	760	31.6	31.6	Fraction 1:	3.16
2	63 - 130	145 - 266	760	84.2	115.8	Fraction 2:	8.42
3	130 - 191	266 - 376	40	102.7	218.5	Fraction 3:	10.27
4	191 - 232	376 - 450	40	57.3	275.8	Fraction 4:	5.73
5	232 - 343	450 - 650	40	194.9	470.7	Fraction 5:	19.49
6/7	343 - 454	650 - 850	10 ⁻²	67.0	537.7	Fraction 6/7:	6.70
8	454 - 496	850 - 925	10 ⁻²	81.9	616.6	Fraction 8:	8.19
9	496 - 537	925 - 1 000	10 ⁻²	47.8	667.4	Fraction 9:	4.78
10	537 - 565	1 000 - 1 050	10 ⁻²	57.0	724.4	Fraction 10:	5.70

Distillate amount: 724.4 g = 72.44 wt%

Residue: 258.0 g = 25.80 wt%

Loss: 17.6 g = 1.76 wt%

Crude Oil Distillation
Site Etzel, Cavern No. 117

Sample No. 21, Depth 1 380 m, initial quantity of 580 g

Fraction	Column Temperature from...to °C	from...to °F	Pressure torr	Amount of Crude Oil fraction g	cumulative g	Remarks	wt%
1	< 35 - 63	< 95 - 145	760	17.1	17.1	Fraction 1:	2.95
2	63 - 130	134 - 266	760	59.5	76.6	Fraction 2:	10.26
3	130 - 191	266 - 376	40	17.1	93.7	Fraction 3:	2.95
4	191 - 232	376 - 450	40	32.9	126.6	Fraction 4:	5.67
5	232 - 343	450 - 650	40	98.2	224.8	Fraction 5:	16.93
6/7	343 - 454	650 - 850	10^{-2}	44.1	268.9	Fraction 6/7:	7.60
8	454 - 496	850 - 925	10^{-2}	31.1	300.0	Fraction 8:	5.36
9	496 - 537	925 - 1 000	10^{-2}	34.0	334.0	Fraction 9:	5.86
10	537 - 545	1 000 - 1 050	$2 \cdot 10^{-2}$	24.8	358.8	Fraction 10:	4.28
11	565 - 572	1 050 - 1 062	$4 \cdot 10^{-2}$	49.3	408.1	Fraction 11:	8.50

Distillate amount: 408.1 g = 70.36 wt%

Residue: 147.0 g = 25.34 wt%

Loss: 24.9 g = 4.29 wt%

ENCLOSURE 54

Crude Oil Distillate
 Site Etzel, Cavern No. 117

Sample No. 22, Depth 1 382 m, initial quantity of 570 g

Fracton	Column Temperature		from...to		Pressure	Amount of Crude Oil		Remarks	wt%
	from...to		from...to		torr	fraction	cumulative		
	°C		°F			g	g		
1	< 35	- 63	< 95	- 145	760	17.2	17.2	Fraction 1:	3.01
2	63	- 130	145	- 266	760	54.5	71.7	Fraction 2:	9.56
3	130	- 191	266	- 376	40	20.2	91.9	Fraction 3:	3.54
4	191	- 232	376	- 450	40	30.9	122.8	Fraction 4:	5.42
5	232	- 343	450	- 650	40	98.9	221.7	Fraction 5:	17.35
6/7	343	- 454	650	- 850	10^{-2}	48.3	270.0	Fraction 6/7:	8.47
8	454	- 496	850	- 925	10^{-2}	37.0	307.0	Fraction 8:	6.49
9	496	- 537	925	- 1 000	$2 \cdot 10^{-2}$	33.1	340.1	Fraction 9:	5.81
10	537	- 565	1 000	- 1 050	$2 \cdot 10^{-2}$	13.6	353.7	Fraction 10:	2.39
11	565	- 572	1 050	- 1 062	$4 \cdot 10^{-2}$	35.9	389.6	Fraction 11:	6.30

Distillate amount: 389.6 g = 68.35 wt%

Residue: 153.0 g = 26.84 wt%

Loss: 27.4 g = 4.81 wt%

ENCLOSURE 55

TBP -Distillation ETZEL SITE

Etzel Site
Cavern No. 117

Table: Gas Chromatography - Fraction 2

Sample No.	Depth m	Theoretical* Stratification of Crude Oil	Hexane vol %	Heptane vol %	Octane vol %
1	978	Iranian Heavy	26.19	48.98	24.82
2	1 064		26.13	46.78	27.08
3	1 074	Arabian Light	27.63	47.94	24.42
15	1 084		28.25	47.32	24.43
4	1 094				
5	1 104	Iranian Heavy			
16	1 125				
6	1 145				
7	1 155	Arabian Light			
17	1 180				
8	1 200		28.05	47.76	24.18
9	1 240	Basrah			
18	1 270				
10	1 315				
11	1 325	Iranian Light			
12	1 340		28.27	47.80	23.92
13	1 350	Arabian Light			
14	1 370		28.45	48.35	23.20
19	1 376				
20	1 378	Oil/Brine Interface Region	25.91	48.26	25.82
21	1 380		15.31	48.46	36.22
22	1 382		13.48	50.47	36.04

* Stratification also see enclosure 25

Crude Oil Analysis

139

Etzel Site

Cavern No. 117

Table: Gas Chromatography - Fraction 4

Sample No.	Depth m	Theoretical * Stratification of Crude Oil	C 10 vol%	C 11 vol%	C 12 vol%	C 13 vol%
1	978	Iranian Heavy	11.27	37.10	36.21	15.42
2	1 064		5.37	37.35	41.36	15.92
3	1 074	Arabian Light	10.28	36.49	36.49	16.74
15	1 084		11.20	36.51	34.80	17.48
4	1 094					
5	1 104	Iranian Heavy				
16	1 125					
6	1 145					
7	1 155	Arabian Light				
17	1 180					
8	1 200		10.07	34.79	36.30	18.84
9	1 240	Basrah				
18	1 270					
10	1 315					
11	1 325	Iranian Light				
12	1 340		8.38	35.34	38.24	18.04
13	1 350	Arabian Light				
14	1 370		10.60	35.06	38.16	16.18
19	1 376					
20	1 378		9.23	37.75	38.86	14.16
21	1 380	Oil/Brine Interface Region	8.60	36.13	36.98	18.29
22	1 382		9.37	37.40	36.66	16.21

* Stratification also see enclosure 25

ENCLOSURE 58

Etzel Site
Cavern K 117
Crude Oil

140

Fraction 2-Gaschromatography

Fraction 4

Crude Oil Analyses
Site Lesum L 103

Sample No.	Depth m	Water Content wt %	Sediment wt %	Sulfur wt %	Pour Point (upper)		Salt Content mg/kg
					°C	°F	
1	630	< 0.1	< 0.01	1.58	- 27	- 16.6	24
2	645	< 0.1	< 0.01	2.33	- 25	- 13.0	29
3	660	< 0.1	< 0.01	1.67	- 24	- 11.2	29
4	675	< 0.1	< 0.01	1.63	- 24	- 11.2	30
5	690	< 0.1	< 0.01	1.63	- 24	- 11.2	33
6	705	< 0.1	< 0.01	1.68	- 24	- 11.2	38
7	720	< 0.1	< 0.01	1.67	- 23	- 9.4	36
8	735	< 0.1	< 0.01	1.68	- 22	- 7.6	40
9	749	< 0.1	< 0.01	1.65	- 20	- 4.0	54
10	751	0.2	0.03	1.71	- 17	1.4	445
9 *	749	< 0.1	< 0.01		- 20	- 4.0	56
10 *	751	0.1	0.01		- 18	- 0.4	525

* repeated analyses

Crude Oil Analyses

Site Lesum L 103

Sample No.	Depth m	Kinematic Viscosity at 77 °F cSt	Viscosity at 122 ° F cSt	Density at 20 °C kg/m ³	API-Gravity at 60 °F **	Optical Density Color Absorbance ***
1	630	7.44	4.34	850	34.25	0.719
2	645	7.27	4.07	855	34.3	0.704
3	660	7.44	4.21	850	34.25	0.726
4	675	7.56	4.21	850	34.25	0.711
5	690	7.56	4.34	850	34.25	0.709
6	705	7.56	4.34	850	34.25	0.733
7	720	7.56	4.34	850	34.25	0.705
8	735	7.56	4.34	850	34.25	0.711
9	749	8.03	4.34	850	34.25	0.720
10	751	7.90	4.33	851	34.05	0.713
9 *	749	8.05	4.22	850	34.25	0.720
10 *	751	8.84	4.45	851	34.05	0.714

* repeated analyses

**API gravity converted from density

***Absorbance measured at 410 nm

Crude Oil Analyses

Site Lesum L 103

Sample No.	Depth m	Neutralization No. mg KOH/g	Mercaptan H ₂ S* mg/kg	Nitrogen Content wt %	Vapor Pressure bar	psia***
1	630	0.055	49	0.076	0.25	3.62
2	645	0.054	59	0.078	0.26	3.77
3	660	0.055	66	0.076	0.25	3.62
4	675	0.056	68	0.075	0.25	3.62
5	690	0.056	67	0.077	0.25	3.62
6	705	0.110	70	0.080	0.26	3.77
7	720	0.170	68	0.078	0.26	3.77
8	735	0.330	72	0.074	0.26	3.77
9	749	0.270	84	0.075	0.25	3.62
10	751	0.055	195	0.072	0.26	3.77
9 **	749		60		0.26	3.77
10 **	751		141		0.26	3.77

* H₂S was analysed qualitatively, all results were negative

** repeated analyses

*** converted from bar

ENCLOSURE 62

Crude Oil Analyses
Site Lesum L 103

Trace Metals

Sample	Depth m	Nickel		Vanadium		Copper		Iron concentration ppm
		concentration mg/l	ppm	concentration mg/l	ppm	concentration mg/l	ppm	
1	630	3.89	4.53	8.56	9.96	0.38	0.44	52.9
2	645	3.84	4.47	9.53	11.09	0.19	0.22	30.5
3	660	3.65	4.25	10.48	12.19	0.12	0.14	15.6
4	675	3.72	4.33	10.55	12.27	0.13	0.15	39.3
5	690	3.78	4.40	10.02	11.66	0.12	0.14	22.0
6	705	3.65	4.26	9.46	11.01	0.13	0.15	45.2
7	720	3.74	4.35	9.08	10.57	0.12	0.14	18.8
8	735	3.70	4.30	9.11	10.60	0.10	0.12	18.4
9	749	3.72	4.33	9.21	10.72	0.13	0.15	18.4
10	751	3.74	4.35	8.78	10.22	0.22	0.25	32.8

Crude Oil Distillation

Site Lesum, Cavern No. L 103

Sample No. 1, Depth 630 m, initial quantity of 1 000 g

Fraction	Column Temperature		Pressure torr	Amount of Crude Oil		Remarks	wt%
	from...to °C	from...to °F		fraction g	cumulative g		
1	40 - 63	104 - 145	760	28.0	28.0	Fraction 1:	2.80
2	63 - 130	134 - 266	760	78.3	106.3	Fraction 2:	7.83
3	130 - 191	266 - 376	40	95.5	201.8	Fraction 3:	9.55
4	191 - 232	376 - 450	40	73.2	275.0	Fraction 4:	7.32
5	232 - 343	450 - 650	40	230.0	505.0	Fraction 5:	23.00
6/7	343 - 454	650 - 850	10^{-2}	43.3	548.4	Fraction 6/7:	4.34
8	454 - 496	850 - 925	10^{-2}	113.2	661.6	Fraction 8:	11.32
9	496 - 537	925 - 1 000	10^{-2}	69.1	730.7	Fraction 9:	6.91
10	537 - 565	1 000 - 1 050	$2 \cdot 10^{-2}$	38.2	768.9	Fraction 10:	3.82
11	565 - 572	1 050 - 1 062	$4 \cdot 10^{-2}$	45.3	814.2	Fraction 11:	4.53

Distillate amount: 814.2 g = 81.42 wt%

Residue: 163.0 g = 16.30 wt%

Loss: 22.8 g = 2.28 wt%

ENCLOSURE 64

Crude Oil Distillation

Site Lesum, Cavern No. L 103

Sample No. 2, Depth 645 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C	from...to °F	Pressure torr	Amount of Crude Oil fraction g	cumulative g	Remarks	wt%
1	38 - 63	100 - 145	760	43.4	43.4	Fraction 1:	4.34
2	63 - 130	134 - 266	760	82.8	126.2	Fraction 2:	8.28
3	130 - 191	266 - 376	40	98.6	224.8	Fraction 3:	9.86
4	191 - 232	376 - 450	40	71.7	296.5	Fraction 4:	7.17
5	232 - 343	450 - 650	40	220.4	516.9	Fraction 5:	22.04
6/7	343 - 454	650 - 850	10^{-2}	75.7	592.6	Fraction 6/7:	7.57
8	454 - 496	850 - 925	10^{-2}	91.3	683.9	Fraction 8:	9.13
9	496 - 537	925 - 1 000	10^{-2}	57.9	741.8	Fraction 9:	5.79
10	537 - 565	1 000 - 1 050	$2 \cdot 10^{-2}$	43.0	784.8	Fraction 10:	4.30
11	565 - 572	1 050 - 1 062	$4 \cdot 10^{-2}$	48.7	833.5	Fraction 11:	4.87

Distillate amount: 833.5 g = 83.35 wt%

Residue: 149.2 g = 14.92 wt%

Loss: 17.3 g = 1.73 wt%

ENCLOSURE 65

Crude Oil Distillation
Site Lesum, Cavern No. L 103

Sample No. 4, Depth 675 m, initial quantity of 1 000 g

Fraction	Column Temperature		Pressure torr	Amount of Crude Oil		Remarks	wt%
	from...to °C	from...to °F		fraction g	cumulative g		
1	39 - 63	102 - 145	760	35.8	35.8	Fraction 1:	3.58
2	63 - 130	134 - 266	760	85.8	121.6	Fraction 2:	8.58
3	130 - 191	266 - 376	40	91.6	213.2	Fraction 3:	9.16
4	191 - 232	376 - 450	40	67.7	280.9	Fraction 4:	6.77
5	232 - 343	450 - 650	40	232.0	512.9	Fraction 5:	23.20
6/7	343 - 454	650 - 850	10^{-2}	77.9	590.8	Fraction 6/7:	7.79
8	454 - 496	850 - 925	10^{-2}	84.7	675.5	Fraction 8:	8.47
9	496 - 537	925 - 1 000	10^{-2}	64.9	740.4	Fraction 9:	6.49
10	537 - 565	1 000 - 1 050	$2 \cdot 10^{-2}$	39.6	780.0	Fraction 10:	3.96
11	565 - 572	1 050 - 1 062	$6 \cdot 10^{-2}$	52.1	832.1	Fraction 11:	5.21

Distillate amount: 832.1 g = 83.21 wt%

Residue: 143.1 g = 14.31 wt%

Loss: 24.8 g = 2.48 wt%

Crude Oil Distillation

Site Lesum, Cavern No. L 103

Sample No. 6, Depth 705 m, initial quantity of 1 000 g

Fraction	Column Temperature		Pressure torr	Amount of Crude Oil		Remarks	wt%
	from...to °C	from...to °F		fraction g	cumulative g		
1	39 - 63	102 - 145	760	31.9	31.9	Fraction 1:	3.19
2	63 - 130	134 - 266	760	83.0	114.9	Fraction 2:	8.30
3	130 - 191	266 - 376	40	105.0	219.9	Fraction 3:	10.50
4	191 - 232	376 - 450	40	64.7	284.6	Fraction 4:	6.47
5	232 - 343	450 - 650	40	226.6	511.2	Fraction 5:	22.66
6/7	343 - 454	650 - 850	10^{-2}	96.6	607.8	Fraction 6/7:	9.66
8	454 - 496	850 - 925	10^{-2}	72.2	680.0	Fraction 8:	7.22
9	496 - 537	925 - 1 000	10^{-2}	73.9	753.9	Fraction 9:	7.39
10	537 - 565	1 000 - 1 050	$2 \cdot 10^{-2}$	38.0	791.9	Fraction 10:	3.80
11	565 - 572	1 050 - 1 062	$4 \cdot 10^{-2}$	35.9	827.8	Fraction 11:	3.59

Distillate amount: 827.8 g = 82.78 wt%

Residue: 156.0 g = 15.60 wt%

Loss: 16.2 g = 1.62 wt%

ENCLOSURE 67

Crude Oil Distillation
 Site Lesum, Cavern No. L 103

Sample No. 10, Depth 751 m, initial quantity of 1 000 g

Fraction	Column Temperature from...to °C	from...to °F	Pressure torr	Amount of Crude Oil fraction g	cumulative g	Remarks	wt%
1	39 - 63	102 - 145	760	32.1	32.1	Fraction 1:	3.21
2	63 - 130	134 - 266	760	83.9	116.0	Fraction 2:	8.39
3	130 - 191	266 - 376	40	98.4	214.4	Fraction 3:	9.84
4	191 - 232	376 - 450	40	70.0	284.4	Fraction 4:	7.00
5	232 - 343	450 - 650	40	220.0	504.4	Fraction 5:	22.00
6/7	343 - 454	650 - 850	10^{-2}	108.6	613.0	Fraction 6/7:	10.86
8	454 - 496	850 - 925	10^{-2}	73.7	686.7	Fraction 8:	7.37
9	496 - 537	925 - 1 000	10^{-2}	62.9	749.6	Fraction 9:	6.29
10	537 - 565	1 000 - 1 050	$2 \cdot 10^{-2}$	35.5	785.1	Fraction 10:	3.55
11	565 - 572	1 050 - 1 062	$4 \cdot 10^{-2}$	48.3	833.4	Fraction 11:	4.83

Distillate amount: 833.4 g = 83.34 wt%

Residue: 148.1 g = 14.81 wt%

Loss: 18.5 g = 1.85 wt%

ENCLOSURE 68

Crude Oil Analyses
 Site Lesum L 103

Gaschromatography - Fraction 2 and 4

Sample No.	Depth m	C 6 vol%	Fraction 2		C 8 vol%	C 10 vol%	Fraction 4		C 13 vol%
			C 7 vol%				C 11 vol%	C 12 vol%	
1	630	26.18	49.66		24.15	13.46	37.80	35.41	13.31
2	645	27.83	48.17		23.98	10.16	36.25	36.49	17.09
3	660								
4	675	27.67	46.77		25.55	13.60	40.46	33.07	12.86
5	690								
6	705	26.68	47.55		24.27	7.04	35.77	40.31	16.86
7	720								
8	735								
9	749								
10	751	27.61	47.28		25.10	9.69	37.78	37.91	14.90

ENCLOSURE 69

Lesum Site
Cavern L 103
Crude Oil

151

Gaschromatography
Fractions 2 + 4

n-C6 C7 C8 C10 C11 C12 C13

Depth in
600

10%

50%

700

800

HEWLETT PACKARD

PART NO 5270 1027

ENCLOSURE 70

Fuel Oil Analyses
Site Lesum L 104

Sample No.	Depth m	Water Content wt%	Sediment wt %	Flame Point °C	Pour Point °C	Salt Content mg/kg
1	730	< 0.1	< 0.01	83	- 15	15
2	745	< 0.1	< 0.01	83	- 11	0
3	760	< 0.1	< 0.01	80	- 8	0
4	775	traces	0.01	80	- 8	0
5	790	0.1	< 0.01	82	- 8	11
6	805	traces	< 0.01	81	- 8	0
7	820	traces	< 0.01	80	- 8	0
8	835	traces	0.01	81	- 7	11
9	850	traces	< 0.01	80	- 7	11

ENCLOSURE 71

Fuel Oil Analyses
Site Lesum L 104

Sample No.	Depth m	Kinematic Viscosity		Density at 20 °C kg/m ³	API Gravity* at 60 °F	Optical Density Color
		at 77 °F cSt	at 122 °F cSt			
1	730	4.42	2.56	840	36.2	0.140
2	745	4.31	2.69	839	36.4	0.089
3	760	5.02	2.93	839	36.4	0.099
4	775	5.02	2.93	839	36.4	0.092
5	790	4.90	2.93	839	36.4	0.078
6	805	4.91	2.93	838	36.6	0.077
7	820	4.90	2.93	839	36.4	0.081
8	835	4.91	2.93	838	36.6	0.084
9	850	5.02	2.93	839	36.4	0.084

* density converted to API gravity

ENCLOSURE 72

Fuel Oil Analyses

Site Lesum L 104

Sample No.	Depth m	Ash (oxyd) wt %	Carbon Residue wt %	Total Sulphur wt %	Copper-strip Corrosion
1	730	traces*	0.08	0.30	1 b **
2	745	traces	0.03	0.31	1 b
3	760	traces	0.06	0.33	1 b
4	775	traces	0.03	0.34	1 b
5	790	traces	0.07	0.33	1 b
6	805	traces	0.03	0.33	1 b
7	820	traces	0.04	0.32	1 b
8	835	traces	0.02	0.33	1 b
9	850	traces	0.05	0.32	1 b

* traces means ash content < 50 ppm

** copper strip corrosion standard D 130 IP 154

Fuel Oil Analyses

Lesum Site

Cavern No. 104

Distillation (DIN 51751)

No.	Start	5 %	10 %	20 %	30 %	40 %	50 %	60 %	70 %	80 %	90 %	95 %	(vol %)
1.	212	217	222	232	243	255	268	285	300	317	340	357	
2.	215	220	222	227	237	248	263	279	296	316	338	355	
3.	200	210	218	233	251	267	281	298	315	332	351	365	
4.	206	214	221	235	250	265	279	298	315	331	351	365	
5.	200	208	216	233	248	264	279	298	311	328	348	362	
6.	200	211	220	235	250	268	285	300	317	332	350	367	
7.	199	210	218	232	248	264	281	297	313	330	349	364	
8.	201	211	217	234	253	267	283	297	312	329	350	365	
9.	198	209	218	232	252	266	283	298	314	331	351	365	

Temperature in °C →

BRINE ANALYSES

	pH-Value	NaCl-Content (g/l)
1. Etzel Site, K 117		
Sample No. 23		
Depth = 1 398 m	7.15	310.2
Sample No. 24		
Depth = 1 403 m	7.10	314.3
2. Lesum Site, L 103		
Sample No. 11		
Depth = 758 m	6.6	316.6
Sample No. 12		
Depth = 762 m	7.3	314.9
3. Lesum Site, L 104		
Sample No. 10		
Depth = 880.5 m	9.7 *)	315.5
Sample No. 11		
Depth = 882.5 m	6.9	312.5
Sample No. 12		
Depth = 883.5 m	6.8	313.7
Sample No. 13		
Depth = 885.5 m	7.05	314.3
Sample No. 14		
Depth = 893.5 m	6.9	312.5
4. Oil in brine content less than 0.1 ppm in all brine samples from Etzel and Lesum.		

*) Sample from tin-plated receptacle

Temp. in
degr. C

Test Distillation

157

ETZEL SITE
Cavern K 117Density
at 20 degr.C

ETZEL SITE
Cavern K 117
Crude Oil

Crude Oil Analysis

162

Etzel Site

Cavern No. 117

Table : Density of Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Density at 20 ^o C (kg/m ³)	API-Gravity ²⁾ at 60 ^o F	General: ³⁾ Density at 20 ^o C (kg/m ³)
1	978	Iranian Heavy	859	32.56	867.3
2	1 064		860	32.36	
3	1 074	Arabian Light	860	32.36	851.6
15	1 084		860	32.36	
4	1 094		860	32.36	
5	1 104	Iranian Heavy	860	32.36	867.3
16	1 125		859	32.59	
6	1 145		860	32.36	
7	1 155	Arabian Light	860	32.36	851,6
17	1 180		859	32.59	
8	1 200		860	32.36	
9	1 240	Basrah	860	32.36	851.6
18	1 270		859	32.59	
10	1 315		860	32.36	
11	1 325	Iranian Light	860	32.36	854.1
12	1 340		860	32.36	
13	1 350	Arabian Light	860	32.36	851.6
14	1 370		860	32.36	
19	1 376		860	32.36	
20	1 378	Oil/Brine Interface	860	32.36	
21	1 380		868	30.86	
22	1 382		873	29.96	
Oil from overflow during sampling			860	32.36	

1) Stratification see also enclosure 25

2) Density converted to API-Gravity

3) General Data obtained by Mobil Oil in 1978

ENCLOSURE 81

Crude Oil Analysis

163

Etzel Site

Cavern No. 117

Table: Viscosity of Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Kinematic ²⁾ at 25°C (77°F) (cSt)	Viscosity ²⁾ at 50°C (122°F) (cSt)	Kinematic ³⁾ General ³⁾ at 122°F (cSt)	Viscosity ⁴⁾ Specific ⁴⁾ at 77°F (cSt)
1	978	Iranian Heavy	11.57	5.96	7.12	13.2
2	1 064		10.85	5.60		
3	1 074	Arabian Light	10.85	5.48		
15	1 084		10.74	5.60	4.83	8.2
4	1 094		10.85	5.60		
5	1 104	Iranian Light	10.62	5.60		
16	1 125		10.63	5.71	7.12	13.2
6	1 145		10.74	5.60		
7	1 155	Arabian Light	10.74	5.60		
17	1 180		10.75	5.60	4.83	8.2
8	1 200		10.62	5.60		
9	1 240	Basrah	10.74	5.48		
18	1 270		10.86	5.60	5.19	8.8
10	1 315		10.85	5.60		
11	1 325	Iranian Light	10.85	5.60	4.80	-
12	1 340		10.85	5.60		
13	1 350	Arabian Light	10.85	5.60		
14	1 370		11.20	5.60	4.83	8.2
19	1 376		11.32	5.71		
20	1 378		11.32	5.71		
21	1 380	Oil/Brine Interface Region	-	9.08	-	-
22	1 382		-	9.50		
Oil from overflow during sampling			11.55	5.60	-	-

1) Stratification see also enclosure 25

2) Results from Analyses of Samples

3) General Data analysed by Mobil Oil in 1978

4) Specific Data from Offloading Analysis Report

ENCLOSURE 82

Crude Oil Analysis

164

Etzel Site
Cavern No. 117

Table: Vapor Pressure of Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Vapor pressure ²⁾		Vapor pressure ³⁾ General ³⁾ Specific ⁴⁾	
			(bar)	(psia)	(psia)	(psia)
1	978	Iranian Heavy	0.29	4.2	6.0	5.3
2	1 064		0.35	5.1		
3	1 074	Arabian Light	0.36	5.2	4.0	4.4
15	1 084		0.36	5.2		
4	1 094		0.35	5.1		
5	1 104	Iranian Heavy	0.36	5.2	6.0	5.3
16	1 125		0.35	5.1		
6	1 145		0.37	5.3		
7	1 155	Arabian Light	0.36	5.2	4.0	4.4
17	1 180		0.35	5.1		
8	1 200		0.37	5.3		
9	1 240	Basrah	0.36	5.2	4.0	6.0
18	1 270		0.35	5.1		
10	1 315		0.36	5.2		
11	1 325	Iranian Light	0.37	5.3	7.0	
12	1 340		0.36	5.2		
13	1 350	Arabian Light	0.35	5.1	4.0	4.4
14	1 370		0.36	5.2		
19	1 376		0.36	5.2		
20	1 378	Oil/Brine Interface Region	0.36	5.2	-	-
21	1 380		0.29	4.2		
22	1 382		0.29	4.2		
Oil from overflow during sampling			0.35	5.1		

1) Stratification see also enclosure 25

2) Pressure in bar converted to psia

3) General Data analysed by Mobil Oil in 1978

4) Specific Data from OFFLOADING Analysis Report

ENCLOSURE 83

Etzel Site

Cavern No. 117

Table: Neutralization Number

Sample No.	Depth (m)	Theoretical ¹⁾ Stratification of Crude Oil	Neutralization number	
			mg KOH/g 2)	3)
1	978	Iranian Heavy	0.44	0.08
2	1 064		0.49	
3	1 074	Arabian Light	0.28	0.05
15	1 084		0.11	
4	1 094		0.49	
5	1 104	Iranian Heavy	0.22	0.08
16	1 125		0.17	
6	1 145		0.22	
7	1 155	Arabian Light	0.16	0.05
17	1 180		0.05	
8	1 200		0.17	
9	1 240	Basrah	0.20	0.08
18	1 270		0.00	
10	1 315		0.25	
11	1 325	Iranian Light	0.11	-
12	1 340		0.10	
13	1 350	Arabian Light	0.11	0.05
14	1 370		0.11	
19	1 376		0.00	
20	1 378	Oil/Brine Interface Region	0.05	-
21	1 380		1.50	
22	1 382		2.43	

1) Stratification see also enclosure 25

2) Sample analysis

3) Data from NWO and Mobil Oil

Etzel Site

Cavern No. 117

Table: Water and Sediment of Samples

Sample No.	Depth (m)	Theoretical ¹⁾ stratification of crude oil	Water ²⁾ content (% wt)	Sediment (% wt)	General: ³⁾ Water and sediment (% vol.)	⁴⁾ Water and sediment (% vol.)
1	978	Iranian Heavy	traces	< 0.01	0.50	0.05
2	1 064		traces	< 0.01		
3	1 074	Arabian Light	traces	< 0.01	0.05	0.1
15	1 084		traces	< 0.01		
4	1 094		traces	< 0.01		
5	1 104	Iranian Heavy	traces	< 0.01	0.50	0.05
16	1 125		traces	< 0.01		
6	1 145		traces	< 0.01		
7	1 155	Arabian Light	traces	< 0.01	0.05	0.1
17	1 180		traces	< 0.01		
8	1 200		traces	< 0.01		
9	1 240	Basrah	traces	< 0.01	0.10	0.1
18	1 270		traces	< 0.01		
10	1 315		traces	< 0.01		
11	1 325	Iranian Light	traces	< 0.01	1.20	0.05
12	1 340		traces	< 0.01		
13	1 350	Arabian Light	traces	< 0.01	0.05	0.1
14	1 370		traces	< 0.01		
19	1 376		traces	< 0.01		
20	1 378	Oil/Brine Interface Region	traces	< 0.01	-	-
21	1 380		3.9	0.83		
22	1 382		4.6	0.91		
Oil from overflow during sampling			traces	< 0.01		

1) Stratification see also enclosure 25

2) Traces means less than 0.1 %

3) General Data analysed by Mobil Oil in 1978

4) Specific Data from Offloading Analysis Report

Site Etzel

Oil in Brine Content

Date	K 101 (ppm)	K 111 (ppm)	Composit of all caverns (ppm)
<u>February 77</u>			
8	1.5	1.0	1.0
9	1.3	1.2	1.6
10	1.2	0.9	1.6
11	1.3	0.8	1.9
12	0.7	0.7	1.5
13	0.6	0.8	1.7
14	1.0	1.0	1.3
15	1.4	1.5	1.2
16	1.1	1.5	1.1
17	1.0	1.0	1.0
18	0.7	1.1	0.9
19	0.7	0.4	1.3
20	0.6	0.7	0.9
21	0.5	0.8	1.3
22	0.9	1.1	0.8
23	1.1	1.0	1.1
24	0.8	0.9	1.2
25	1.0	1.2	1.4
26	0.9	0.7	1.0
27	0.5	0.6	0.7
28	0.5	1.0	1.2
29	0.7	1.0	0.8

Date	K 101 (ppm)	K 111 (ppm)	Composit of all caverns (ppm)
<u>March 77</u>			
1	1.1	1.5	0.8
2	0.9	0.8	0.8
3	0.7	0.8	0.9
4	0.8	0.6	0.8
5	0.7		0.4
6			
7

Brine Analysis from August 1977
Site Etzel
Cavern K 117

Sodium	117.62	g/l
Potassium	638.00	mg/l
Calcium	1 034.00	mg/l
Magnesium	1 269.00	mg/l
Chloride	183.27	g/l
Sulfate	4 893.00	mg/l
Suspended matter	6.00	mg/l
Density	1.20	g/ml
Temperature	20.00	° C
pH-value	7.00	
Oil in brine	0.60	mg/l
Mercury	-	ppm
Cadmium	-	ppm

The following photographs show the main steps of the sampling and analyzing procedure:

1. - 3. Sites Etzel (K 117) and Lesum (L 103)
4. - 5. The bridge plug which had to be set in the brine tubings to be pulled (Etzel)
6. - 7. The pulled tubings covered with oil grease (Lesum)
8. - 9. The sampler, a special construction for sampling in crude oil caverns
10. - 13. Details of the sampler before and after lowering (Etzel)
14. - 15. Instrumentation of the recording truck, lowering the sampler (Lesum)
16. - 17. Filling of the receptacles with oil from the hoisted sampler (Etzel)

- 18. - 19. Filling of the receptacles with viscous oil from the oil brine interface
- 20. - 21. Well-head underneath the work-over rig, with funnel and basin for oil overflow caused by extension and/or lowering of the brine tubings
- 22. - 30. Some of the 20 chemical analyses of each oil sample taken at various depths:
 - 22. Determination of ash content
 - 23. Determination of carbon residue
 - 24. Determination of vapor pressure
 - 25. Sediment determination
 - 26. Viscosity determination
 - 27. Distillation column with temperature and pressure control systems
 - 28. Determination of mercaptans by potentiometric titration
- 29. - 30. Atomic absorption spectrometer

1.
Entrance of
Site Etzel
(K 117)

2.
Site Etzel (K 117)
Workover rig and
recording trucks

3.
Site Lesum
Workover rig
(L 103/104)

4./5.

Bridge plug for 8 5/8"-pipe (Etzel)

6./7.

Crude oil grease on pulled tubings (Lesum)

8./9.

Sampler on ground (Etzel) and upright with
cantilever in 40° position (Lesum)

10./11.

Sampler before lowering:
lower part without sample
container,
mounting of sample container

12./13.

Sampler before lowering:
upper part of cantilever
with valve

Sampler after lowering:
oil covered tilt drive

14./15.

Instrumentation of the recording truck

16./17.

Filling of the receptacles

18./19.

Filling of receptacles with viscous crude oil
from oil brine interface

20./21.

Wellhead underneath
workover rig with funnel
and basin for oil overflow

22.

Determination of ash content (oxyd)

23./24.

Determination of carbon residue and vapor pressure

25./26.

Sediment determination, viscosity determination (Hüppler)

27./28.

Distillation column with temperature and pressure control systems; determination of mercaptans by potentiometric titration

29./30.

Atomic absorption spectrometer