

8.2 Oil Removal Experiment by Pretreatment with Bench Scale Equipment

8.2.1 Installation of Pretreatment Equipment

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1. Introduction

This unit consists of an oil feed control unit, and oil filtrating removal unit and an filter tower regeneration unit Mock oil polluted sea water is made by the oil feed control unit. The oil content is then controlled using the oil filtrating removal unit. The system is then evaluated with the filter tower regeneration unit under continuous operation.

1.1 Purpose of the Equipment

(1) Oil Feed Control Unit

Oil is added to the sea water at the specified concentration and dispersed in the sea water. The polluted water is then fed to the oil filtrating removal unit and the filter tower regeneration unit.

(2) Oil Filtrating Removal Unit

In this unit oil polluted sea water is processed with filter material and oil is extracted. Examination is made of the filter material, the coagulant (types & quantity) and the condition of the water filtered.

(3) Filter Drum Regeneration Unit

This unit is used to evaluate the filter material for extraction properties and back-wash when operating continuously for long periods with water from the oil filtrating removal unit.

1.2 Capacity

(1) Oil Feed Control Unit

Treated Water Flow Rate 4 m³/H

Filtrating Removal Unit (0.1 m³/H) + Filter Drum Regeneration Unit (1.0 m³/H) + Filter Unit (Approx.: 2.0 m³/H)

Oil Feed Rate 100 mL/H

(2) Oil Filtrating Removal Unit

Water Flow Rate 100L/H

Concentration of Oil Content 10 ppm

When Washing 500/H

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- (3) **Filter Drum Regeneration Unit**
Water Flow Rate 1 m³/H
Concentration of Oil Content 10 ppm
During Back-wash 5 m³/H

1.3 Specifications & Standards

- (1) **Oil Feed Control Unit**
Water Supply Sea Water
Oil Heavy Oil
Method of Oil Dispersion Pump Mixing and Ultrasonic
Extraction of Un-dispersed Oil Atmospheric Flotation
Treated Water Flow Rate Maximum 4 m³/H
Oil Feed Rate 100 mL/H

- (2) **Oil Filtrating Removal Unit**
Water Supply
Oil Contaminated Sea Water
Input Concentration of Oil Content 10 ppm
Methods Of filtration
Fixed Bed
Column Size Internal Diameter 100 mm x 1000 mm
Surface Area 78.5 cm²
Filter Packing Height 600 mm
Treated Water Flow Rate 100 L/H
LV 13 m/H
SV 21/H
Washing Unit Using Sea Water
Automatic Sampling Unit Active

- (3) **Filter Drum Regeneration Unit**
Water Supply Oil contaminated Sea Water
Input Concentration of Oil Content 10 ppm
Method of Filtration Fixed Bed
Column Size Internal Diameter 400 mm x 1500 mm
Surface Area 1256 cm²

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Filter Packing Height	1000 mm
Treated Water Flow Rate	1 m³/H
LV	8m/H
SV	8/H
Back-wash Unit	Using Sea Water+Air

1.4 Flow Description

(1) Oil Feed Control Unit

Sea water supply is fed into sea water tank (TK-1) located inside the laboratory. The water is then pumped to the testing equipment inside. To prevent the suspended substances from settling below the seawater tank is fitted with a mixer.

The oil pump (P-3) pumps a set amount of oil from the oil tank (TK-2) to the intake of the seawater pump (P-2). In the seawater pump the mixture is compressed and this compressed mixture is subjected to an ultrasonic homogenizer (UH-1) dispersing the oil in the sea water at a very high concentration. This oil concentrated seawater is pumped to the preparation tank (TK-3).

In the preparation tank the oil contaminated water is exposed to air from a blower (B-1) combining air/fluid and using a mixer (MX-2). Surplus oil and suspended substances attach to the micro air bubbles which then float in the atmospheric flotation tank (TK-4) and are scraped off.

The treated water passes into the intermediate tank (TK-8) and the via the intermediate pump (P-9) to the oil polluted seawater tank (TK-31) or to the existing filter bed.

The scraped sea water from the flotation tank passes to the scum tank (TK-5), the water layers are recycled via the scum pump (P-7) back to the separate tank increasing the concentration of the oil content.

(2) Oil Filtrating Removal Unit

The sea water in the oil polluted seawater tank (TK-31) passes via the oil polluted seawater pump (P-21) to the filter columns (D-21, 22, 23). Where the oil content is extracted by the filter packing. The oil contaminated water can be processed in a single tower or in series through a number of towers. The water supply and the treated water can be sampled automatically by the automatic sampler (AS-21).

After replacing the filter material washing is conducted by switching the intake on the oil polluted seawater pump from oil polluted seawater pump from oil polluted seawater tank (TK-31) to sea water tank (TK-1).

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The addition of coagulant is supplied in fixed amounts by the coagulant feed unit from (P-34, TK-33).

(3) Filter Drum Regeneration Unit

Sea water from the oil polluted seawater tank (TK-31) is fed by the oil polluted seawater pump (p-31) to the filter drums (D-31, 32, 33, 34), the oil being removed by the filter packing. The four filter drums are arranged in series for treating the water. Treated water passes to the treated seawater tank (TK-32) and pH level is adjusted by the pH control unit (P-35, TK-34). The pH adjusted water is pumped via the treated seawater pump (P-32) to the mini module testing unit.

When the filters become blocked with pollutants the pressure difference between the inlet and outlet of the filter drum increases. When the pressure difference has risen above set value the washing blower (B-31) and washing pump (P-33) are used to clean the filters. The back-wash waste water has a high concentration of oil, it therefore treated by filtration and discharged.

1.5 Utilities & Additional Materials

(1) Utilities

Electric Power Supply 3 phase 200V
Stepped down to 60 HZ100v by transformer

(2) Additional Materials

Filter Sand	Yamabishi, Mean Diameter 0.6 mm Distribution less than 1.7
Polymer Adsorbant	Tonen, Chemseptor - CM121
Anthracite	Yamabishi,
Activated Carbon	Kurare GW 20 - 42, Diameter of granules 0.35 0.7mm Agents
Inorganic Coagulant	Ferric chloride
Polymer Coagulant	Cation Polymer
Desalting Agents	SBS
pH Regulator	Sulfuric acid

2. Operating Conditions

Supplied Sea Water Temperature : Summer 30-35°C

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Winter 15–17°C

(1) Oil Feed Control Unit

Water Flow Rate

Sea Water Pump 4 m³/H

Specimen Pump 100L/H

Oil Flow Rate 100mL/H

Air Flow Rate 4Nm³/H (Equal to the Water Flow Rate 4 m³/H)

(2) Oil Filtrating Removal Unit

Oil Removal Operation

Treated Water Flow Rate 100L/H

During Washing Filter Material

Unusual for washing water capacity to be greater than the filter material

(3) Filter Drum Regeneration Unit

Oil Removal Operating

Treated Water Flow Rate 1 m³/H

During Back-Wash

Unusual for back-wash water capacity to be greater than the filter material.

Refer to the operating method including the back-wash method.

3. Operation of Each Unit

The following describes operating procedures, methods of adjustment and standard settings for each unit. Refer to the respective Operating Manual for detailed operating instructions.

(1) Oil Feed Control Unit

Under the operation of the sea water pump (P-2) and oil feed pump (P-3), oil is dispersed in the sea water. After dispersion is checked by the flow meter at the outlet of the sea water pump, the oil feed pump (P-3) starts to operate. When operating the pump with the operating switch set to [AUTO] the level of the water in the sea water tank falls. When the water level falls below the setting on the level switch (LS-1) the LLA lamp on the control panel lights up and the pumps (P-1, 2, 3, 33) stop. The sea water tank is replenished and when the water level rises above the level setting the pump operates automatically.

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To adjust the flow rate of each pump adjust the opening on the outlet valve and return valves while watching the respective flow gauge. To measure the flow rate of the oil feed pump open the air valve on the outlet side and measure the oil flow into the cylinder.

Before operating the pump run the mixer (MX-1) for 5 minutes to mix the water in the sea water tank. However, when sea water pollution is very low the mixer may be operated for shorter period.

Only when the sea water from the sea water pump reaches the ultrasonic homogenizer (UH-1) is the homogenizer operated. The oil contaminated sea water in the preparation tank (TK-3) over flows into the flotation tank (TK-4). Here the gas/fluid mixer (MX-2) and blower (B-1) are operated. To start the blower open the air and waste water valves of the blower outlet and release the pressure. When the blower is operating normally open the valve on the preparation tank and close the air valve.

The floating oil in the flotation tank is removed by the scrape to the scum tank (TK-5). The scum pump is operated manually while watching the water level of the scum tank. The overflow water from the flotation tank flows into the intermediate tank (TK-8). The intermediate pump (P-9) then transfers the water to the oil polluted seawater tank. The intermediate pump is controlled automatically by the intermediate tank level switch (LS-2).

The flow rate of the coagulant pumps (P-4,P-5) and the desalting agent pump (P-6) are measured in the same way as for the oil pump by opening the air valve on the outlet side and allow the respective agent go flow into the cylinder.

(2) Oil Filtrating Removal Unit

Remove the upper cover from the filter packing on the filter towers (D-21, 22, 23). The standard thickness of the filter packing is 60% of the height of the column (600mm). After packing the filter material reconnect the inlet port of the specimen pump (P-21) to the sea water tank (TK-1) so the sea water flows upward to wash the packing and remove excess air. As the oil contaminated seawater, mixed by the specimen control unit, flows into the oil polluted seawater tank (TK-31) operate the coagulant pump (P-34) to introduce coagulant, and with the mixer produce flock.

Although sea water flows sporadically from the intermediate tank to the oil polluted seawater tank coagulant must be fed continuously, corresponding to the amount of treated water in the flotation tank. As the flow of treated water from the oil filtrating removal unit is as small as 1/40 of the flow from the oil feed control unit this

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unit works intermittently.

Treated water flows downwards through the columns during operation.

The valve in the unit are opened and closed to control the flow water down ward through the columns. The coagulation flock from the oil polluted seawater tank is pumped via the specimen pump to the filter drum. Control the flow by adjusting the valve while observing the flow meter. At the beginning of operation keep the upper air valve open while the water is flowing to allow the remaining air to escape. To remove the filter packing disconnect the piping at the union joints, raise the column slightly and place a vinyl bag under the column. Remove the lower cover and allow the packing to drop into the bag.

(3) Filter Drum Regeneration Unit

To insert filter packing into the filter drums (D-31, 32, 33, 34) remove the piping and hoses and open the upper cover. The standard packing thickness is 1000 mm. The amount of packing is calculated from the distance from the top of the filter drum to the top of the packing, and the cross section area of the drum. To remove the packing remove the piping and hoses from the lower cover, suspend the lower cover by the four long bolts and remove the packing by passing it between the drum body and the lower cover. Wash the packing using the back-wash pump.

The polluted sea water is transferred from the oil polluted seawater tank to the filter drum by the oil polluted seawater pump (P-31). While this experiment can be conducted with only one drum, a number of drums in series is preferable. The operating conditions are controlled by the valves in the unit. During operating observe the pressure gauges on each drum. If the pressure differences is greater than (0.3kg/cm^2) then back-wash the filters. Alternatively, back-wash the filters after treating a preset amount of water. Back-wash the filters manually, during that time stop the water treatment system.

Back-washing is conducted in 3 stages

Stage 1 With a prescribed LV wash with water for 5 minutes with the back-wash pump (P-33) operating.

Stage 2 Set the air flow to 1m/min and wash(dry) for 10 minutes, with the back-wash blower (B-31) operating.

Stage 3 With a prescribed LV wash with water for 10 minutes with the back-wash pump (P-33) operating.

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Propagation Ratio LV (m/H)

polymer	30	10
Activated Carbon	50	30
Sand	30	50
Anthracite	30	30

For water temperature at 20°C. For water at temperature shown below the above LV values should be multiplied by the respective factor.

Water Temperature (°C) Factor

10	0.91
20	1.0
30	1.08
40	1.15

During operation the packing, if removed from the top or bottom, should be replaced. Also if the back-wash cycle is too short all the packing should be replaced.

The concentration of oil contaminated water from back-washing the filter drum is very high and cannot be released into the drainage system untreated. It should only be discharged after filter treatment in a waste water filter.

4. Precautions During Operation

- (1) Before starting pump operation check that the inlet & outlet valves are open and take care that unit does not run without water.**
- (2) After stopping operation close all the valves between the units to prevent back flow of water.**
- (3) If the equipment breaks down stop the system, investigate the cause and correct the fault before continuing operation. However, operation may continue if the cause of fault is known and the operation of the faulty unit does not effect the operation of another unit.**

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- (4) Do not run the pumps or mixers when dry. Be sure there is enough water in tanks. Also care must be taken not to run the ultrasonic homogenize dry or when there is no water in the outlet port.

5. Equipment list

TANK

TK-1	Sea Water Tank
TK-2	Oil Tank
TK-3	Preparation Tank
TK-4	Flotation Tank
TK-5	Scum Tank
TK-6	Inorganic Coagulant Tank
TK-7	Polymer Coagulant Tank
TK-8	Intermediate Tank
TK-31	Oil Polluted Seawater Tank
TK-32	Treated Water Tank
TK-33	Inorganic Coagulant Tank
TK-34	pH Regulator Tank

FILTER UNITS

D-21	No. 1 Filter Column
D-22	No. 2 Filter Column
D-23	No. 3 Filter Column
D-31	No. 1 Filter Drum
D-32	No. 2 Filter Drum
D-33	No. 3 Filter Drum
D-34	No. 4 Filter Drum

PUMPS

P-1	Sea Water Pump
P-2	Seawater Pump
P-3	Oil Feed Pump
P-4	Inorganic Coagulant Pump
P-5	Polymer Coagulant Pump
P-6	Desalting Agents Pump

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- P-7 Scum Pump
- P-8 Intermediate Pump
- P-21 Oil Polluted Seawater Pump
- P-31 Oil Polluted Seawater Pump
- P-32 Treated Water Pump
- P-33 Back-wash Pump
- P-34 pH Regulator Pump

BLOWERS

- B-1 Blower
- B-31 Back-wash Blower

MIXERS

- MX-1 Sea Water Tank Mixer
- MX-2 Gas/fluid Mixer
- MX-31 Oil Polluted Seawater Tank Mixer
- MX-32 Treated Compound Mixer
- LMX-1 Polymer Compound Mixer
- LMX-2 Desalting Agents Compound Mixer

OTHER EQUIPMENT

- UH-1 Ultrasonic Homogenizer
- AS-21 Automatic Sampling Unit

INSTRUMENT

FLOW GAUGES

- FI-1 Sea Water Pump
- FI-2 Seawater Pump
- FI-3 Blower
- FI-21 No. 1 Adsorption Tower
- FI-22 No. 2 Adsorption Tower
- FI-23 No. 3 Adsorption Tower
- FI-31 Oil Polluted Seawater Pump
- FI-32 Back-wash Pump
- FI-33 Back-wash Blower

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LEVEL SWITCHES

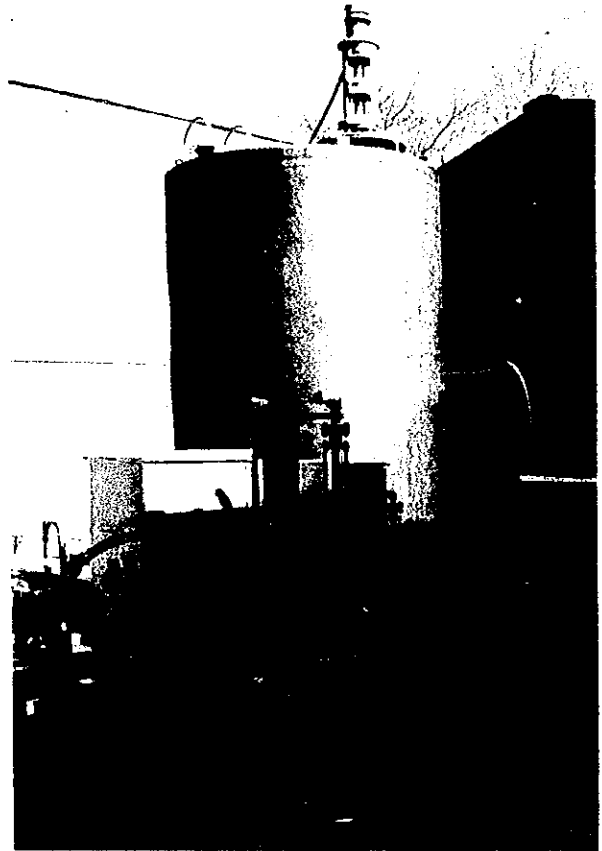
- LS-1 Sea Water Tank**
- LS-2 Intermediate Tank**
- LS-31 Oil Polluted Seawater Tank**
- LS-32 Treated Water Tank**

pH Meter

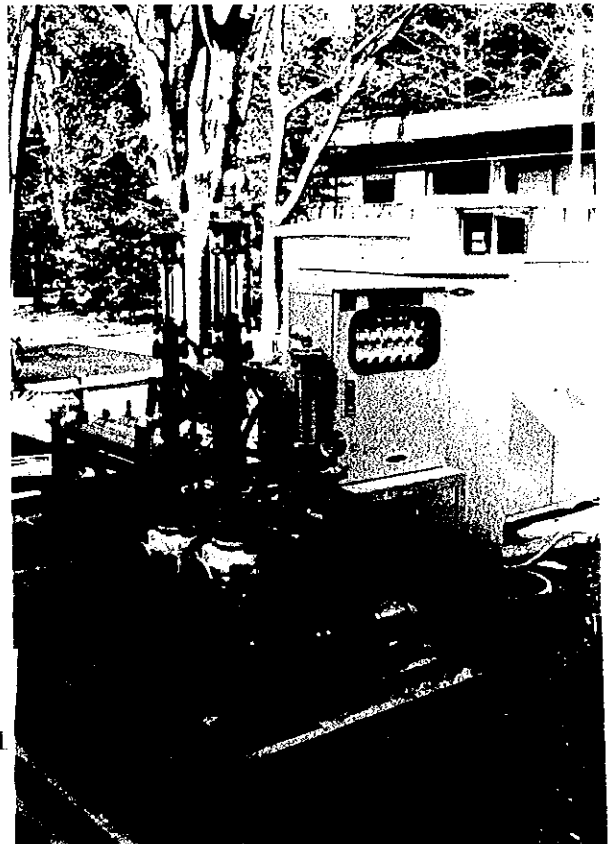
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6. Photographs of Equipment

TK-1
Sea Water Tank

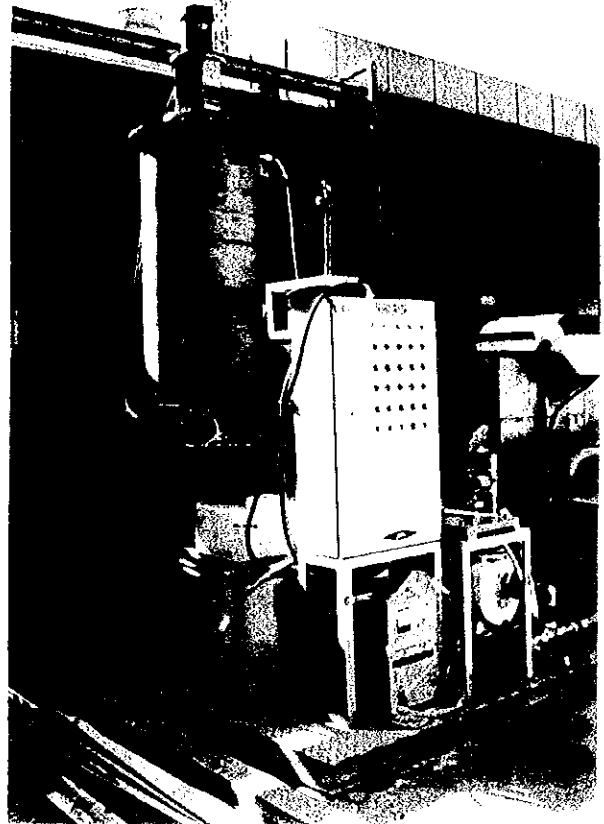


P-1,2,33
Sea Water Pump & Control Panel

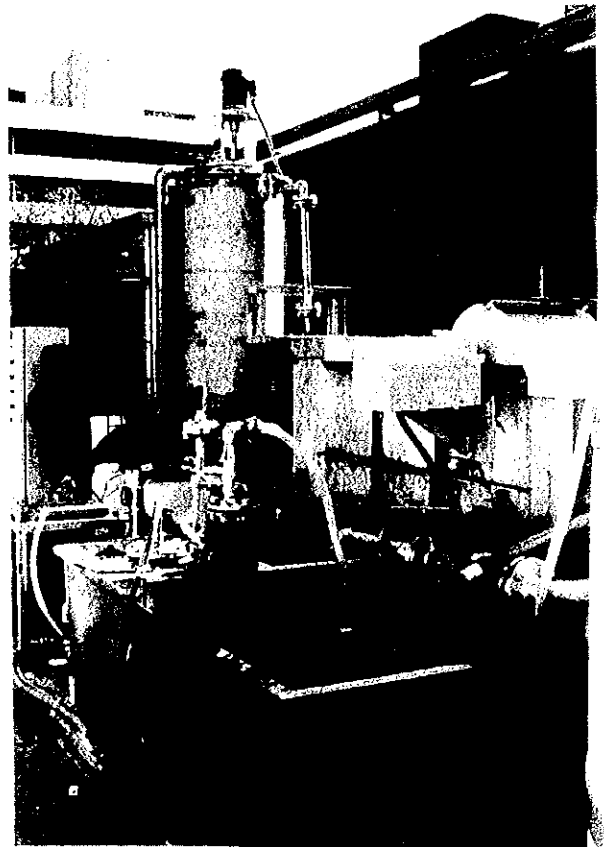


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TK-3
Preparation Tank

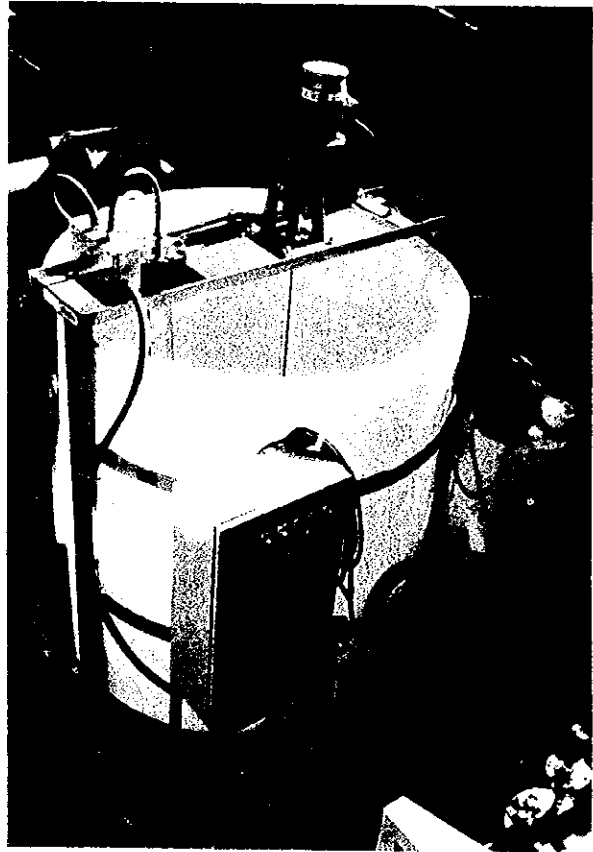


TK-4,5,8
Floatation Tank
Scum Tank
Intermediate Tank

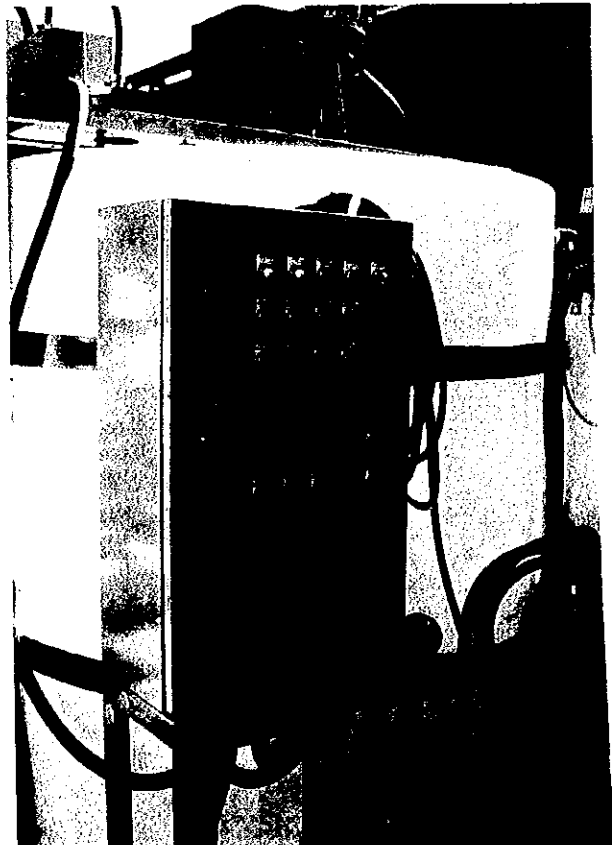


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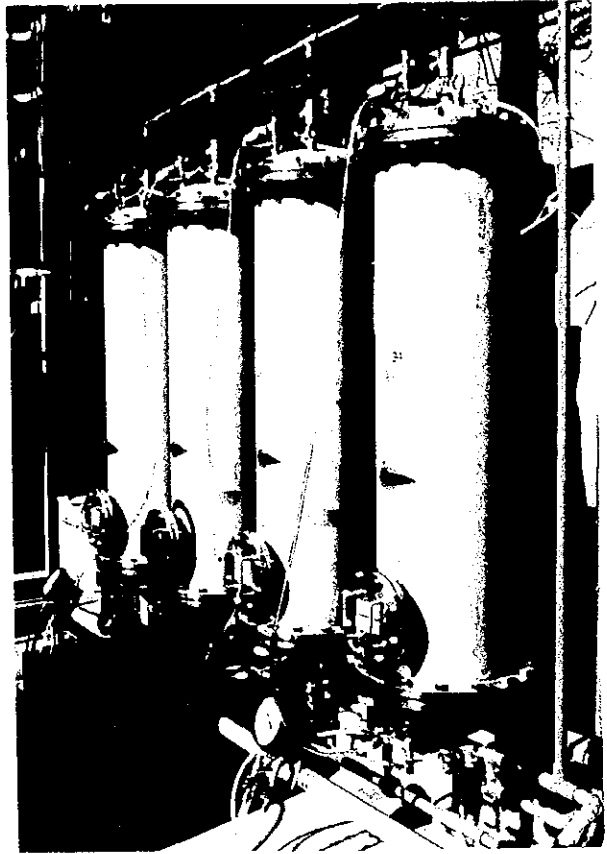
TK-33
Inorganic Coagulant Tank



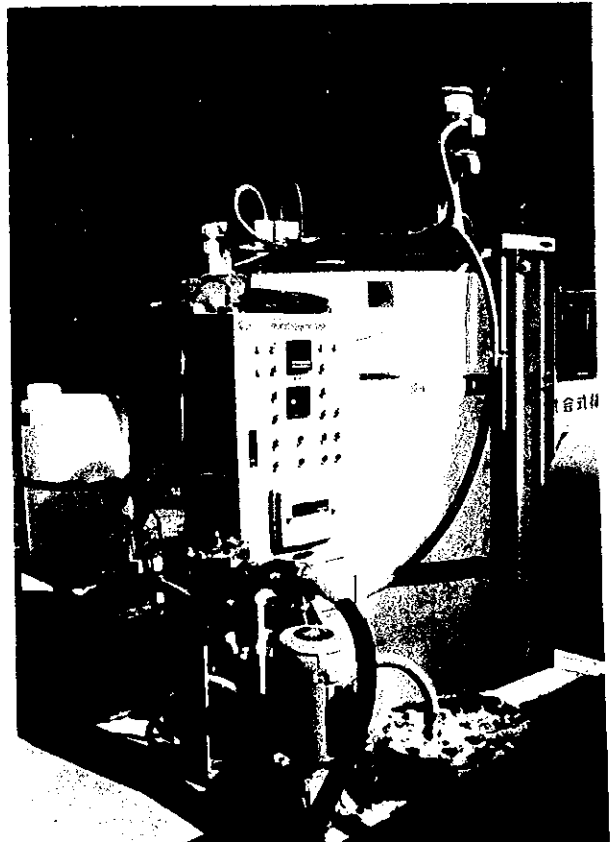
TK-31
Oil Polluted
Seawater Tank



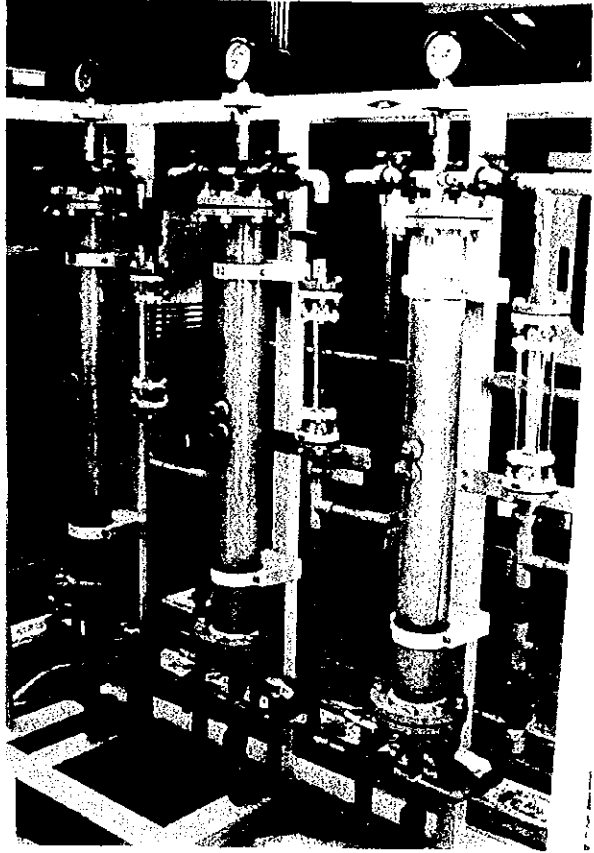
D-31 - 34
NO.1-4 Filter Drum



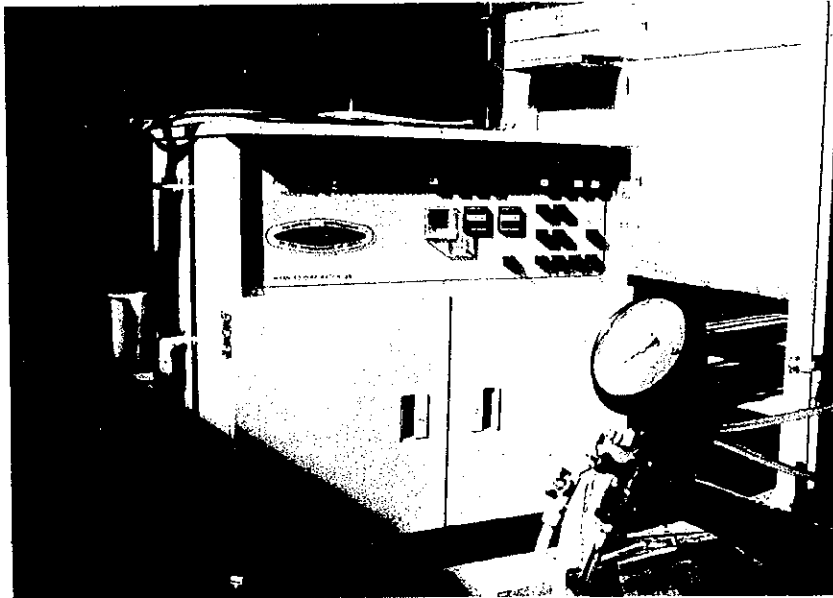
TK-32
Treated Water Tank



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D-21-23
NO.1-3 Filter Column



AS-21
Automatic Sampling Unit



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7. Progress records of work

Date	Work Items	Workers
July 31 Sun.	<ul style="list-style-type: none"> · Equipment arrive. · Unload wooden boxes. 	1 supervisor 6 general workers
August 1 Mon.	<ul style="list-style-type: none"> · Start unpacking. · Installation of the raw water tank unit. 	1 supervisor 8 general workers
August 2 Tues:	<ul style="list-style-type: none"> · Unpacking. · Check the contents of wooden boxes. · Installation of other units. (Except the seawater tank.) 	1 supervisor 8 general workers
August 3 Wed.	<ul style="list-style-type: none"> · Check the contents of wooden boxes. · Transfer the seawater tank to the installation site. · Assemble agitating blades of the seawater tank. 	1 supervisor 8 general workers
August 4 Thurs.	(Off-day)	
August 5 Fri.	(Off-day)	
August 6 Sat.	<ul style="list-style-type: none"> · Fix the sample preparation tank to the sample preparation unit. · Erect the seawater tank, mount the ladder the float switch and agitating blades. (The motor is not mounted at this stage.) · Mount agitating blades in the raw water tank and the treated water tank. 	1 supervisor 6 general workers
August 7 Sun.	<ul style="list-style-type: none"> · Fix the agitating motor to the seawater tank. · Connect pipelines to the oil adsorption and elimination unit and the oil reproduction and elimination unit, and fix the manometer and the differential manometer. · Connect a pipeline to the sample outlet of the sample preparation tank. 	1 supervisor 6 general workers

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Date	Work Items	Workers
August 8 Mon.	<ul style="list-style-type: none">• Fix water drain and supply valves and the water gauge to the seawater tank.• Fix the air and sea water inlet nozzles to the sample preparation tank.• Start boring holes in the floor to fix units with anchor bolts.• Starts installing the distribution panel.• Start connecting hoses between units.	1 supervisor 6 general workers 2 electricians 2 fixing workers
August 9 Tues.	<ul style="list-style-type: none">• Installation of the distribution panel.• Start wiring from the distribution panel to agitators' motors.• Fix units with anchor bolts.• Connect hoses between units, and take safety measures of the hoses.	1 supervisor 5 general workers 2 electricians 2 fixing workers
August 10 Wed.	<ul style="list-style-type: none">• Installation of the distribution panel.• Provide wiring from the distribution panel to each agitator's motor, and from the distribution panel to the control panels (of the pump unit and the treated water unit).• Check the insulation of rotating machinery• Connect hoses between units.• Take safety measures of hose arrangement.• Installation of drain lines of units.	1 supervisor 2 general workers 2 electricians
August 11 Thurs.	(Off-day)	
August 12 Fri.	(Off-day)	
August 13 Sat.	<ul style="list-style-type: none">• Provide wiring from the distribution panel to the control panels (of the sample preparation unit and the raw water unit).• Take safety measures of electric wiring.• Instal drain lines of units.• Lay the sea water line (from the line branching part of the laboratory to the seawater tank).	1 supervisor 2 general workers 2 electricians

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Date	Work Items	Workers
August 14 Sun.	<ul style="list-style-type: none"> • Connect the main power source. • Take safety measures of electric wiring (grounding). • Check electric wiring and connection (from the distribution panel to control panels, and check the rotation direction of rotating machinery). • Clean the equipment. • Instal the exhaust line of the seawater tank. • Stop the leaks of the seawater lines. 	1 supervisor 2 general workers 2 electricians
August 15 Mon.	<ul style="list-style-type: none"> • Check electric wiring and connection (check the rotation direction of rotating machinery). • Connect the float switch of the seawater tank. • Conduct test operation (without using seawater). (Check that rotating machinery, the supersonic homogenizer, and the float switch are actuated.) 	1 supervisor 2 electricians
August 16 Tues.	<ul style="list-style-type: none"> • Take safety measures of electric wiring (grounding). • Installation of the drain line. • Connect a pneumatic pipeline between the blower and the sample preparation tank • Stop the leak of the flotation separation tank's valve. • Conduct test operation (without using seawater). (Check that the ultrasonic homogenizer and the float switch are actuated, and measure the flow rate and the pressure of each pump.) 	1 supervisor 1 general worker 2 electricians

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8. Test Results

1) Test Results of Each Instrument

Instrument	Work	Rotatory direction	Irregular sound and vibration	Judgement
P-1	OK	OK	OK	OK
P-2	OK	OK	OK	OK
P-3	OK	—————	OK	OK
P-4	OK	—————	OK	OK
P-5	OK	—————	OK	OK
P-6	OK	—————	OK	OK
P-7	OK	OK	OK	OK
P-8	OK	OK	OK	OK
P-21	OK	OK	OK	OK
P-31	OK	OK	OK	OK
P-32	OK	OK	OK	OK
P-33	OK	OK	OK	OK
P-34	OK	—————	OK	OK
P-35	OK	—————	OK	OK
P-36	OK	—————	OK	OK
MX-1	OK	OK	OK	OK
MX-2	OK	OK	OK	OK
MX-31	OK	OK	OK	OK
MX-32	OK	OK	OK	OK
B-1	OK	OK	OK	OK
B-31	OK	OK	OK	OK
MOTOR	OK	OK	OK	OK
UH-1	OK	—————	OK	OK

2) Floating Switches

Floating switch	Without seawater			With seawater			Work of each instrument	Judgement
	H ON/OFF	M ON/OFF	L ON/OFF	H ON/OFF	M ON/OFF	L ON/OFF		
LS-1	—	—	—	OK	OK	OK	OK (P-1, 2, 33; MX-1)	OK
LS-2	OK	OK	OK	OK	OK	OK	OK (P-8)	OK
LS-31	OK	OK	OK	OK	OK	OK	OK (P-21, 31; MX-31)	OK
LS-32	OK	OK	OK	OK	OK	OK	OK (P-32; MX-32)	OK

3) Leak and Damages

- Leak at the drain valve on TK-3 → Repaired by change of Oring
- Leak at the pipe on Filter drum unit → Repaired by screwing the connector tighter
- Leak at the connector between TK-32 and P-32 → Repaired by screwing the connector tighter

4) Flow Rate Evaluation

Flow meter	Rate		Judgement
	Designed Normal/Max	Measured Normal/Max	
FI-1	1/4 m ³ /hr.	1/ >5 m ³ /hr.	OK
FI-2	100/500 L/hr.	100/ >500 L/hr.	OK
FI-3	0.5/4 Nm ³ /hr.	0.5/ >5 Nm ³ /hr.	OK
FI-21	100/500 L/hr.	100/ >500 L/hr.	OK
FI-22	100/500 L/hr.	100/ >500 L/hr.	OK
FI-23	100/500 L/hr.	100/ >500 L/hr.	OK
FI-31	1/1.5 m ³ /hr.	1/ >3 m ³ /hr.	OK
FI-32	5/6 m ³ /hr.	5/9 m ³ /hr.	OK
FI-33	5/10 Nm ³ /hr.	5/ >10 Nm ³ /hr.	OK

5) Pressure Gauge

Pressure gauge	Measured [kgf/cm ²] (Normal/Max)	Flow rate (Normal/Max)	Judgement
PG-1	3.4/3.1	1/4 m ³ /hr.	OK
PG-2	4.3/3.5	100/500L/hr.	OK
PG-3	3.5/2.0	0.5/4 Nm ³ /hr.	OK
PG-4	0.3	0.4 m ³ /hr. (Max)	OK
PG-5	1.2	12 m ³ /hr. (Max)	OK
PG-21	0.8/0.7	100/500 L/hr.	OK
PG-22	0.0/0.4	100/500 L/hr.	OK
PG-23	0.0/0.4	100/500 L/hr.	OK
PG-24	0.0/0.3	100/500 L/hr.	OK
PG-25	0.0/0.2	100/500 L/hr.	OK
PG-26	0.0/0.2	100/500 L/hr.	OK
PG-27	0.0/0.0	100/500 L/hr.	OK
PG-31	3.4/3.3	1/1.5 m ³ /hr.	OK
PG-33	4.1/4.0	5/6 m ³ /hr.	OK
PG-34	0.4/0.2	5/10 Nm ³ /hr.	OK
PDI-31	0.00/0.00	1/1.5 m ³ /hr.	OK
PDI-32	0.00/0.00	1/1.5 m ³ /hr.	OK
PDI-33	0.00/0.00	1/1.5 m ³ /hr.	OK
PDI-34	0.00/0.00	1/1.5 m ³ /hr.	OK

6) Sampling by Auto-Sampler

Equipment	Sampling test run
Auto-Sampler	OK

7) Test Result of Insulation

Instrument	Insulation	Judgement
P-1	1000MΩ <	OK
P-2	1000MΩ <	OK
P-7	1000MΩ <	OK
P-8	1000MΩ <	OK
P-21	1000MΩ <	OK
P-31	1000MΩ <	OK
P-32	1000MΩ <	OK
P-33	1000MΩ <	OK
B-1	1000MΩ <	OK
B-31	1000MΩ <	OK
MX-1	1000MΩ <	OK
MX-2	1000MΩ <	OK
MX-31	1000MΩ <	OK
MX-32	1000MΩ <	OK

*Instrument for measurement : YOKOGAWA DIGITAL INSULATION TESTER 2407

*Voltage for measurement : 500V

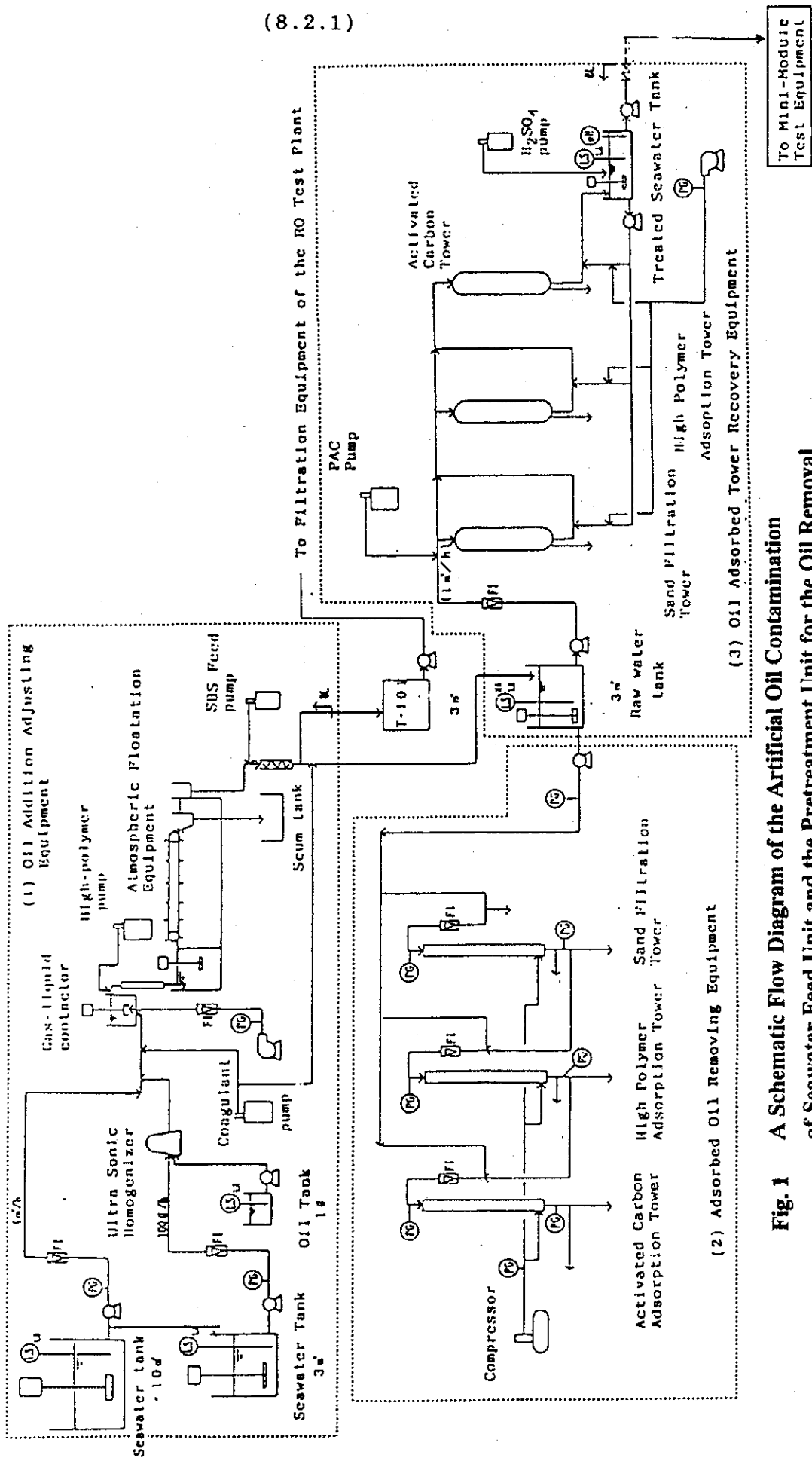


Fig. 1 A Schematic Flow Diagram of the Artificial Oil Contamination of Seawater Feed Unit and the Pretreatment Unit for the Oil Removal

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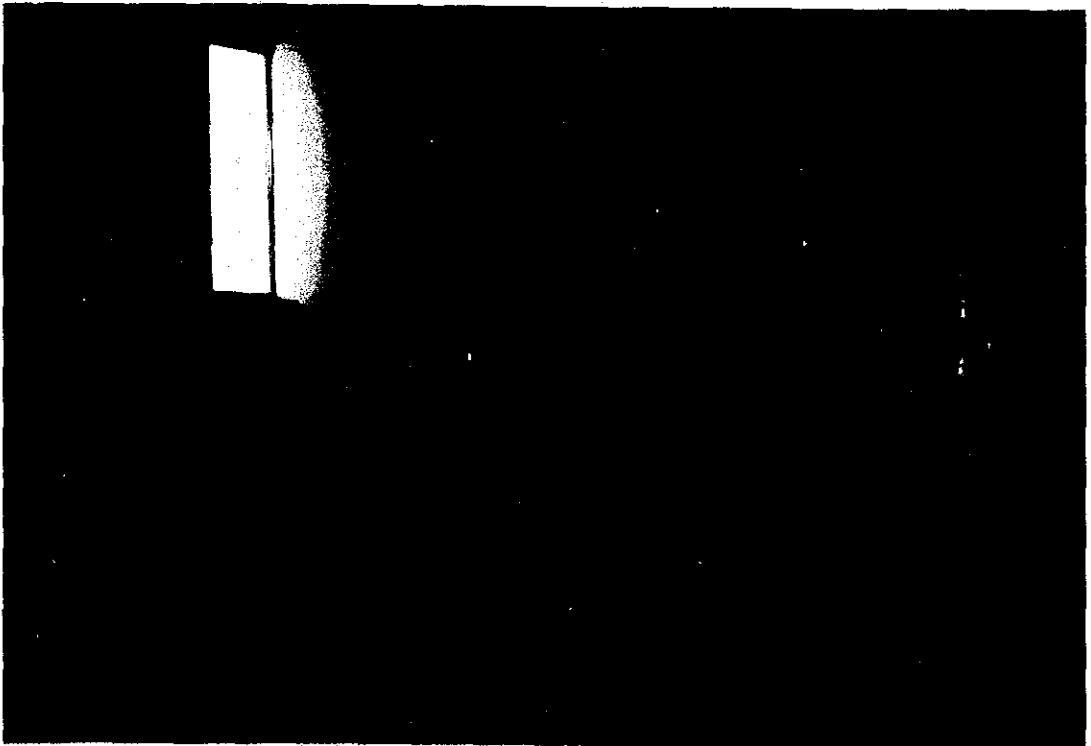


Fig. 2 Oil Addition Adjusting Equipment

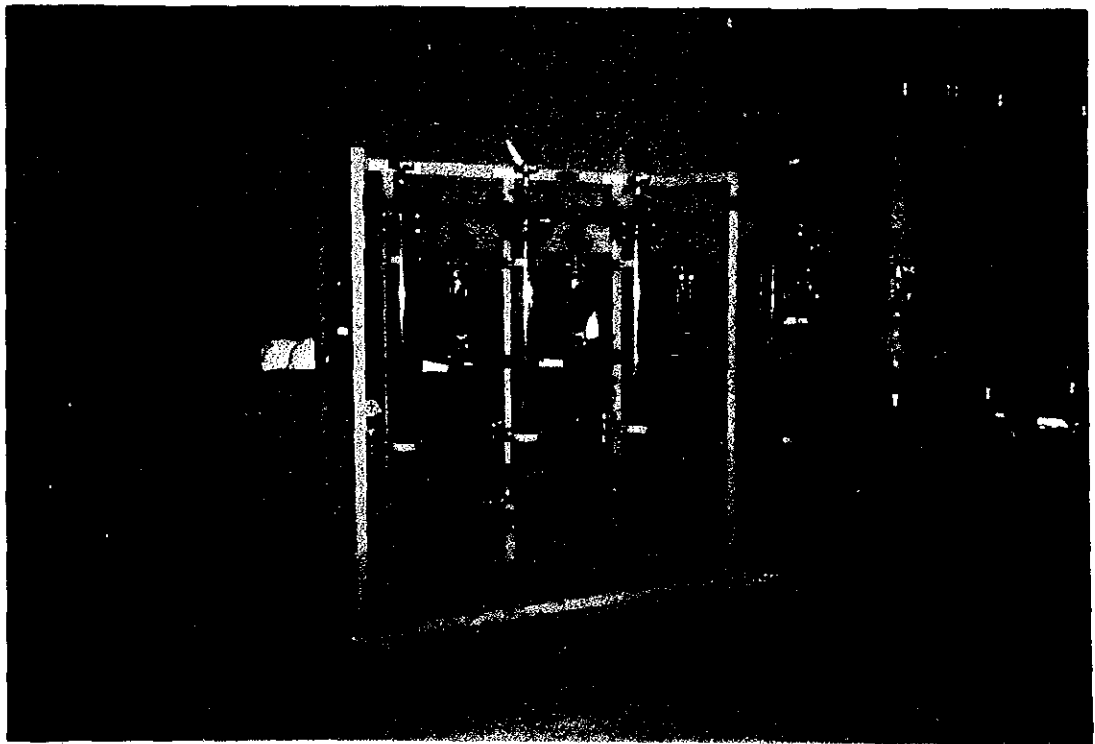


Fig. 3 Oil Adsorbed Tower Recovery Equipment

(8.2.1)



Fig. 4 Oil Adsorbed Tower Recovery Equipment

8.2.2 Oil Dosing Experiment

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We established a method for supplying oil dispersed in sea water (artificial oil-contaminated sea water) for pre-treatment experiments. This necessitated the specification of the concentration of oil dispersed in sea water, the selection of a type of oil and the establishment of a method of stable dispersion at a scale of 4 ton/hr (to suit the specifications of the pre-treatment installation).

1. Specification of oil content in artificial oil-contaminated sea water

(1) Objective

To determine the concentration of oil dispersed in sea water (artificially oil-contaminated sea water) for pre-treatment experiments. We made a literature search of oil concentrations involved in the discharge of crude oil into the sea and used this as a basis for the oil content of our oil-sea water dispersion.

(2) Results

Table 1 shows the thickness of the oil film in various cases of discharge of crude oil into the sea. As shown here, when oil is discharged into the ocean, the film is of the order of 0.01 to 0.2 mm thick. Furthermore, the concentration in the water phase under conditions of oil solution and dispersion is limited by the mean film thickness. For instance, if the oil film thickness is 0.1 mm, the oil content due to complete dispersion and mixing down to a depth of one metre from the surface does not exceed 100 ppm and, in the case of complete mixing down to a depth of 10 m, the oil content does not exceed 10 ppm.^{2.1}

Actually, even in cases of severe surface winds, it is difficult to imagine complete mixing below a depth of several meters and at the depths involved in the positioning of the intake ports of desalination equipment (4 to 12 m in the case of the SWCC reverse osmosis desalination plants^{2.2}), the oil content must be much less.

(8.2.2)

Table 1 Mean thickness of oil films in maritime crude oil spills.^{2,1}

Crude oil discharge	Amount of discharge [m ³]	Mean film thickness [mm]
API Experiment	1.6 or 3.2	0.1 to 0.2
North Sea Experiment	750	0.013 to 0.06
Mexico Bay, Main Pass Block 41C Platform Blowout	240 to 950/day	0.02 to 0.09
North Sea, Ekofisk Bravo Platform Blowout	2800	0.01
Mexico Bay, Ixtoc I	4800	0.07

The dissolved oil content is not greatly affected by the mass balance of the discharged oil and is thought to be of 1%. Reported values are less than 0.1 ppm. Also, these oils in solution are readily volatilized and their time in solution is said to be comparatively brief.¹

The action of continuous strong winds and waves breaks the oil into small droplets of 0.01 to 0.1 mm which are said to remain in the water phase.¹ With La Rosa, Murban and Prudhoe Bay crudes, the concentration at a depth of 1 m was one to two ppm.^{1,3,4} Concentration decreases with depth. When 100 t of Light Statfjord crude was discharged in the North Sea, it was about 90 ppb and 10 to 15 ppb at a depth of ten meters.⁵ The trend is the same, even in cases of large scale oil spills, the reported levels being 0.4 to 88 ppb⁶, 100 to 300 ppb¹ and several ppm.⁷

The results of an oil spill experiment by the American Petroleum Institute using oil containing volatile and soluble components C₁ to C₁₀ (these levels include the hydrocarbons which are mainly in solution when the surplus oil reaches equilibrium with the sea water) show that the soluble oil concentration is extremely low. In an experiment using La Rosa and Murban crudes, oil contents at depths of 1.5 and 3 meters were measured over a period of more than seven hours. Oil was detected in only five out of 68 samples. These were sampled twenty minutes after discharge and the concentrations were 60, 16, 6, 3 and 2 ppb.^{1,3}

In a recent study, the content of general soluble oil components (C₁ to C₁₀) in La Rosa,

(8.2.2)

Murban and Prudhoe Bay crudes sampled at a depth of 3 m 15 to 30 minutes after discharge was 1 to 3.5 ppb and, after 24 hours, it was no more than 0.002 ppm-h. In another study using dispersant 10 to 30 minutes after expelling Prudhoe Bay crude into the sea, the total oil content was reported to be 10 to 40 ppm and the soluble component (C_1 to C_{10}) content was 46 ppb. After 30 to 60 minutes it was 16 ppb, after 3 to 4 hours it was 2 ppb and after 6 to 7 hours it had fallen by half.¹⁴

Thus, there is said to be a great difference between the solubility of the separate components contained in petroleum and the measurements taken after the crude oil or petroleum products are mixed with sea water. In general, the low molecular aromatics are most soluble, the solubility of benzene being about 1000 mg/l. (It is also known that there is a direct relationship between molecular weight and solubility. The solubilities of the naphthenes are said to be several tens of milligrams per liter and even the low molecular paraffins have a solubility of 10 mg/l. However, light oil and crude oil, which are hydrocarbon compounds, have extremely small measurements and their solubility is thought to be due to the formation of a uniform phase which is rather different from that of general soluble substances.⁸

As the above study shows, we understand that when crude oil is discharged into the sea, although the oil content in sea water (in the aqueous phase) differs according to various circumstances such as the conditions of the discharge, time after discharge, water depth and so on, it is generally low.

On this occasion, in the case of the oil content of sea water entering the desalination equipment, which is the object of oil removal, the intake ports are located at a depth of several meters (4 to 12 m in the case of the SWCC reverse osmosis desalination plants) and, even if oil is discharged, since it is difficult to imagine its reaching the intake port immediately, the concentration levels are assumed to be low (ppb levels).

Therefore, the target value for the oil content in the oil-sea water dispersion (artificial oil-contaminated sea water) which is to be prepared in this study has been set conservatively at 10 ppm. If we can confirm that an oil content of this level can be removed adequately from the oil-sea water dispersion, even if an actual oil spill should occur and the contaminated sea water enters the intake port of the desalination plant it will be able to be removed adequately.

2. Determination of the type of oil to be used for artificial oil-contaminated sea water

2.1 Argument for Using Fuel Oil Type A

(1) Objective

To study oil types for the artificial adjustment of oil-contaminated sea water in the case of maritime crude oil spills.

According to literature searches, when crude oil is discharged into the sea, in the initial stage, most of the low molecular compounds in the oil evaporate before the dissolve. Most of the oil components with boiling points up to 270°C are said to have evaporated within 24 hours⁹ and, in the case of paraffin, they relate to C₁₅ or less. Consequently, when crude oil is discharged into the sea, the main components are considered to be those with boiling points higher than 270°C (paraffins higher than C₁₅) and these correspond to the carbon number distribution of Fuel Oil Type A. Accordingly, in simulating a maritime crude oil spill, we shall analyse the carbon number distribution of test materials at this time and compare it with the carbon number distribution of Fuel Oil Type A in order to study the suitability of using Fuel Oil Type A for adjusting the test water.

(2) Experimental Method

We carried out a simulation of a maritime crude oil spill and compared this test material with the component distribution of Fuel Oil Type A.

Apparatus and Reagents

Infrared spectrophotometer (Nihon Bunko IR-810)

Gas chromatography analysis equipment

Separating funnel (2 liter)

Sea water

Crude oil (Arabian light) See Table 2 for properties

Fuel Oil Type A

Carbon tetrachloride (Wako special grade)

(8.2.2)

Table 2 Properties of crude oil used in these tests

No.	Test Item	Test Reading	No.	Test Item	Test Reading
1	Gravity Sp.GR., 15/4°C	0.8567	7	Metal Content on Crude V Wt.ppm	10.1
	API 60°F	33.6		Ni	3.2
2	RVP, Kg/cm ² @37.8°C	0.250		Na	2.5
3	VIS. cst @37.8°C	—	8	Yield,Liq.,Vol.% (Modified Hempel) IBP-182°C	23.5
	@ 50 °C	4.580		182-243°C	12.0
	@ °C	—		243-316°C	14.5
4	Sulfur, wt %	1.78		316-371°C	11.0
5	Water & Sed.Vol % ,BS&W	0.02		371°C(+)	37.0
	Water Vol %	0.00		Loss, vol %	2.0
6	Dist.Atm.Pres., °C IBP	26	9	Reduced Crude (371°C)	—
	5%	94		SP.GR 15/4°C	0.9642
	10%	119		Vis @50°C Cst	496.5
	20%	176		Vis @98.9°C Cst	—
	30%	214		Sulfur wt %	3.39
	40%	265		N wt %	0.19
	50%	316		CCR wt %	11.5
	60%	370		P.Pt °C	+12.5
	70%				
	80%				
	90%				
	371°C Total Dist,vol %	61.0			

(8.2.2)

Procedure

- 1) Preparation of test materials for analysis and simulation of maritime oil spill.
 - We added 550 mg of crude oil to 1,100 ml of sea water in a separating funnel and stirred for 30 minutes at 300 rpm at room temperature.
 - After leaving for 24 hours, one liter of sea water was extracted with carbon tetrachloride, the oil content was measured by the IR method and a component analysis of the oil content was made by gas chromatography.

GC analysis conditions

Equipment	:	Shimadzu GC-9A
Column	:	OV-101
Detector	:	FID
Carrier gas	:	N ₂
Heating conditions	:	50 to 340°C 10°C/min

(3) Results and discussion

1) Results of maritime oil spill simulation test material analysis

Oil content : 8.2 ppm

Components : n-paraffin correspondence C₁₀ to C₂₅ (Fig. 1)

2) Fuel Oil Type A analysis

Components : n-paraffin correspondence C₁₀ to C₂₅ (Fig. 2)

However, the component distribution ratio differed from the crude oil.

The results of this experiment showed clearly that the oil components dispersed in the sea water correspond generally to n-paraffins ranging from C₁₀ to C₂₅. According to the results of the literature search, when crude oil is discharged into the sea, most of the hydrocarbons with boiling points below 270°C (corresponding to C₁₅ n-paraffin) are said to have evaporated²⁹ within 24 hours. Our results confirmed the existence of some fractions which were lighter than this.

A possible reason for the observation of this difference is that the sea is subject to the influence of wind and waves and other factors and, by comparison with this experiment, the conditions for evaporation are much more favourable. In this experiment the material was allowed to rest after shaking and mixing and a range of

(8.2.2)

hydrocarbon components which would have evaporated in the open sea were detected in the course of this experiment. Also, the range of heavy components is not influenced by either system and there is little difference.

In the case of an actual maritime crude oil spill, we can expect that the component distribution will differ according to the various circumstances of the incident and some may approximate the conditions of this experiment.

From these results, the component distribution in the case of a maritime crude oil spill and the Fuel Oil Type A component distribution will be similar, although the chances of each existing are different. This fact suggested that the use of Fuel Oil Type A in preparing artificial oil-sea water dispersions would enable the component distribution to be reproduced satisfactorily.

(8.2.2)

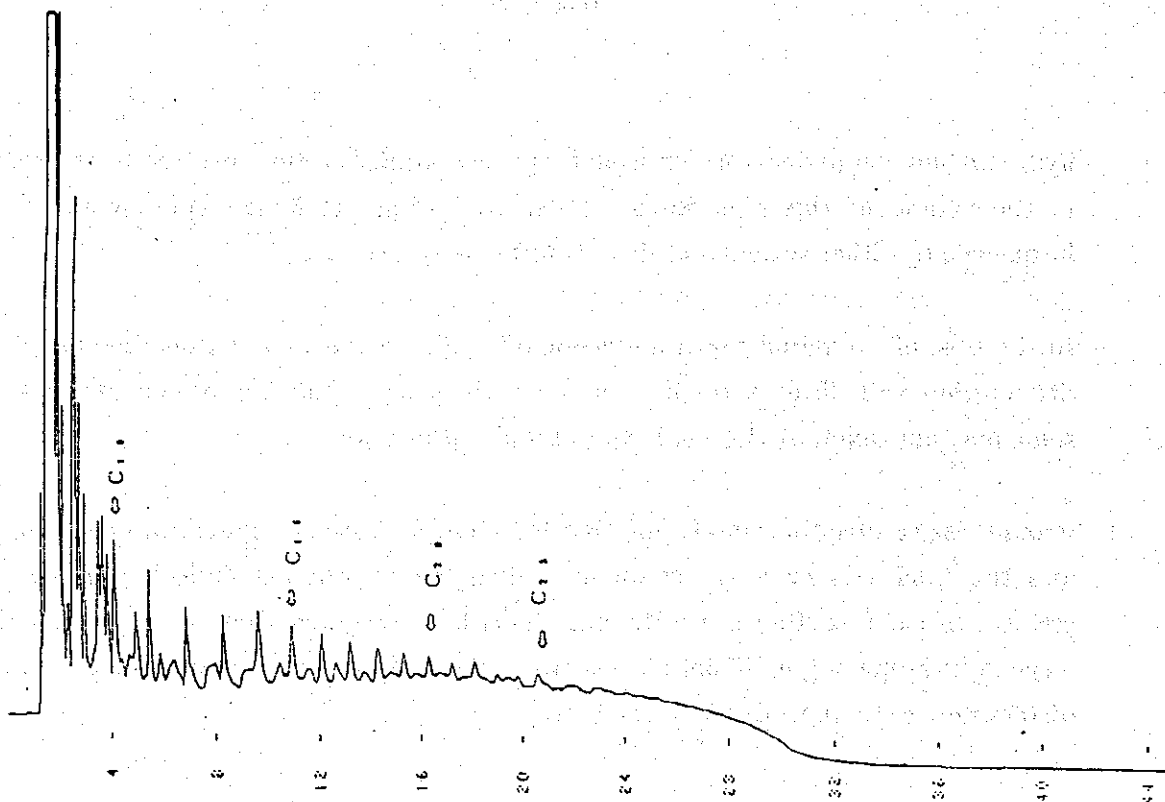
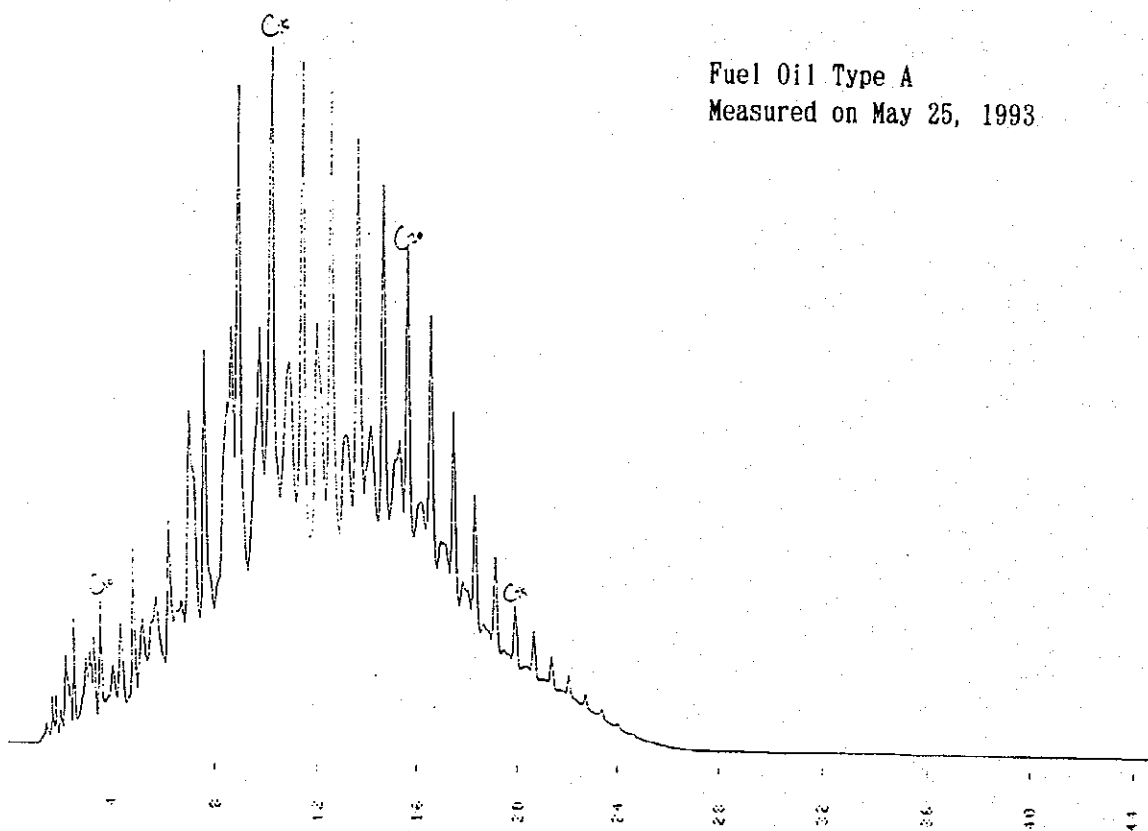


Fig. 1 Chromatgram for crude oil/sea water (1 day after mixing)



Fuel Oil Type A
Measured on May 25, 1993

Fig. 2 Chromatgram for Fuel Oil Type A

2.2 Argument for using Diesel Oil

(1) Background and Objective

Although it had been intended to use fuel oil A for the oil removal experiments with the on-site pre-treatment plant, fuel oil A and similar materials were difficult to obtain locally. However, diesel oil (No. 2) which was used as fuel for the evaporative desalination plant was abundantly available. So it was that diesel oil had to be used in preparing the test materials.

Therefore, in the same way that justification for the use of fuel oil A to prepare the artificial oil-contaminated test material was confirmed before conducting the experiments, here we have confirmed the use of diesel oil in the same way.

(2) Experimental method

The component distributions of fuel oil A and diesel oil were compared using GC-MS

Apparatus and reagents

GC-MS analysis equipment

Sea water

Fuel oil A

Diesel oil (No. 2)

GC-MS analysis conditions

Analyser	:	HP 5880-GC-MSD HP 5970B
Recorder	:	HP Plotter 7475A
Column	:	HP 1, 25 m capillary
Quantity injected	:	3 μ l
Temperature of column oven	:	50°C
Temperature of interface	:	280°C
Injection temperature	:	250°C
Gradient of column temperature	:	1) Initial : 50°C, 3 min
		2) Gradient : 10°C/min
		3) Finishing : 280°C, 10 min

(8.2.2)

(3) **Results and discussion**

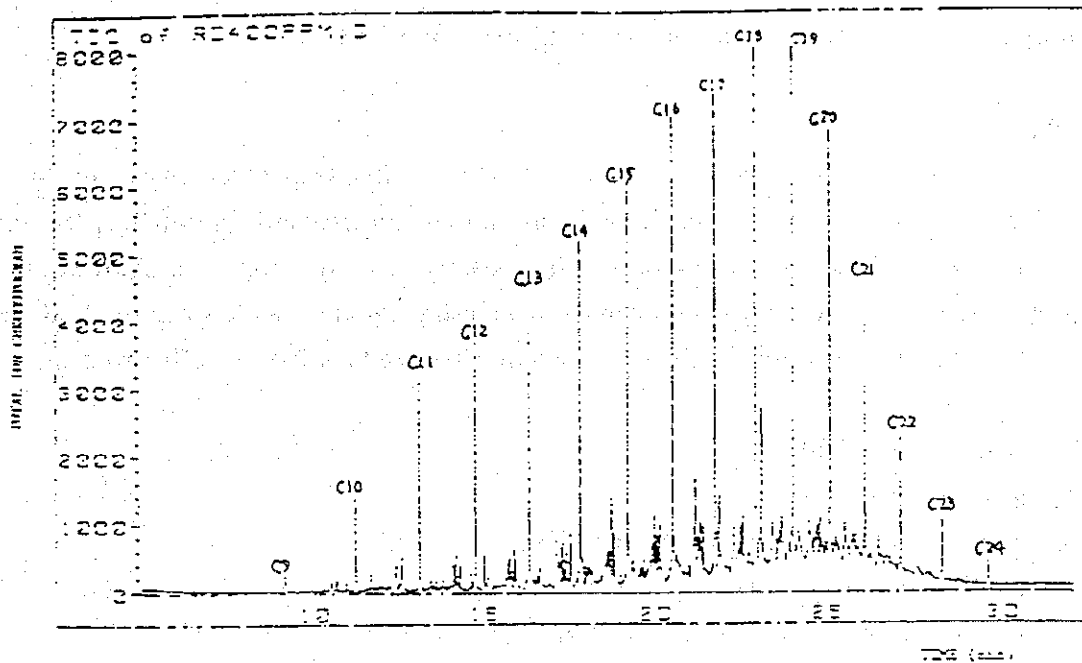
Figure 3 shows the component distribution of fuel oil A and diesel oil. As shown here, compared with fuel oil A, the diesel oil components are slightly lighter hydrocarbons (fuel oil A : C₁₀ to C₁₅, Diesel oil : C₇ to C₂₄) but the difference is small and there is little difference in component distribution between this and crude oil added to sea water and left for a day.

The above results suggest that diesel oil is just as effective as fuel oil A as a type of oil for preparing oil-sea water dispersions in artificial oil-contaminated sea water which simulates a maritime crude oil spill.

Diesel oil

FIGURE 3: A- GC MASS SPECTRUM OF LIGHT DIESEL #2
(Oil Concentration in Sample 100 mg/L)

12/7/1994



B- GC MASS SPECTRUM OF STANDARD JAPANESE FUEL
OIL A (Oil Concentration in Sample 100 mg/L)

12/7/1994

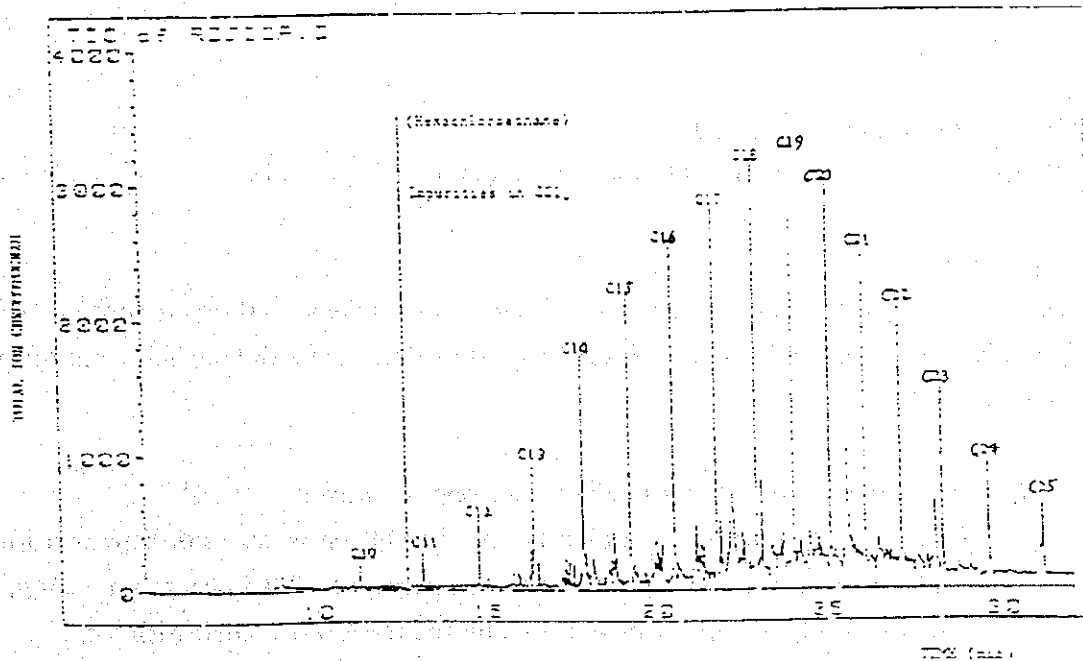


Fig. 3 component distribution between diesel oil and fuel oil Type A

3. Beaker Scale Studies

3.1 Comparison of mixer method and ultrasonic wave method

(1) Objective

Since unusually large quantities up to 4 m³/hr are involved when preparing oil-sea water dispersions with the bench scale oil content adjustment equipment, the mixer method which was used previously will not be practical. Here we shall study the preparation of test materials at a beaker scale using the ultrasonic wave method which seems suitable for producing such larger quantities and confirm its effectiveness.

(2) Experimental method

Apparatus and reagents

Oil content analyser (Horiba OCMA-220)

Mixer (National MX-X1)

Ultrasonic cleaner (Blackstone HT-1.9, 5A, 0.6 kVA)

Microscope (Olympus VANOX-S, AH-2)

Erlenmeyer flasks (2 l, 1 l)

Magnetic stirrer

Sea water

Fuel oil A

Oil content measuring method

Simple oil meter (Carbon tetrachloride extraction, IR method)

Procedure

Oil-sea water dispersions were prepared using the various methods described below. The mixer method used was that implemented in fiscal 1991 during joint research by SWCC-JICA.

1) Preparation of oil-sea water dispersions using the mixer method^{2,10}

- 5000 ppm of fuel oil A was added to one liter of sea water and dispersed for 20 minutes in a mixer. To prevent the temperature of the sea water rising, the mixer was stopped from time to time and the flask was cooled with ice.
- After standing for five minutes, a siphon was used to transfer 700 ml from the bottom of the flask to a separate vessel which was left for a further 60 minutes.

(8.2.2)

- 500 ml was then siphoned from the bottom of the container into a separate vessel and dispersed with the mixer for a further five minutes. 100 ml of the resulting test material was added to 700 ml of sea water and stirred at 100 rpm for another 5 minutes.

2) Preparation of oil-sea water dispersions using the ultrasonic wave method

- 5000 ppm of fuel oil A was added to one liter of sea water and, after stirring overnight with the magnetic stirrer, it was treated in the ultrasonic cleaner for 20 minutes.
- After standing for ten minutes after the ultrasonic treatment, a siphon was used to transfer 650 ml from the bottom of the flask to a separate vessel.
- This material was added to a beaker of sea water and treated with ultrasonic waves for another ten minutes.

(3) Results and Discussion

The methods are compared in Table 3. The greatest difference is that despite the fact that the time required differed by a factor of three, the oil droplets were smaller in the case of the ultrasonic wave method. This difference in size of oil droplets is thought to be due to the greater dispersion energy exerted in the case of the ultrasonic wave method compared with the mixer method. The smaller oil droplets are slower to float upwards which gives the oil content greater stability.

Table 3 Comparison of oil-sea water dispersion preparation methods

	Ultrasonic method	Mixer method
Main oil droplet diameter	<1 μm	1 to 2 μm
Temperature change during preparation	25 to 30°C	21 to 35°C
Time required	About 1hr.	About 1hr.

(8.2.2)

Instead of an ultrasonic wave homogenizer, a comparatively powerless ultrasonic cleaner was used for this experiment. Nevertheless, the oil droplets were very small and the oil content very stable. After this, the ultrasonic method was used for bench scale experiments.

3.2 Studies with the Ultrasonic Wave Method

(1) Objective

To prepare oil-sea water dispersions for bench tests using the ultrasonic method and investigate the component distribution and stability of the test materials obtained.

(2) Experimental Method

Apparatus and reagents

Ultrasonic cleaner (Branson B 5200; 47 kHz, 120 W)

Erlenmeyer flask (1 L)

Gas chromatography analysis equipment

Sea water

Fuel oil type A

TOC Analyser (Shimadzu TOC-500)

Microscope (Olympus BHS-PC-B-SW)

Beaker

Pipette

Oil content measurement method

TOC method

Procedure

- Oil-sea water dispersion was prepared in accordance with the flow chart shown below.
- The prepared oil-sea water dispersion was extracted with carbon tetrachloride and the oil content and component analysis determined by gas chromatography.

(8.2.2)

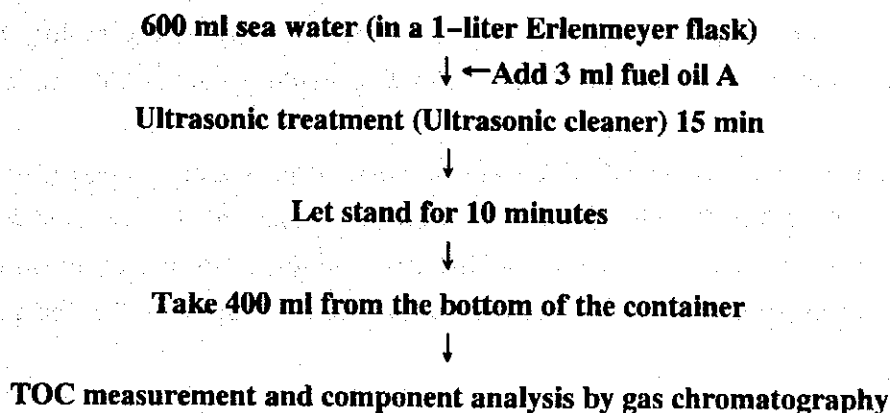


Fig. 4 Preparation of oil-sea water dispersion

Gas chromatography measurement conditions

Equipment	:	Shimadzu GC-9A
Column	:	Packed column (OV-101)
Detector	:	FID
Carrier gas	:	N ₂
Temperature gradient	:	50 to 340°C 10°C/min.

(3) Results and Discussion

The oil-sea water dispersion was prepared and the results of oil content component analysis confirmed the presence of hydrocarbons as n-paraffins with carbon counts of around 8 to 25 (Fig. 5).

Oil droplet diameters were mainly 1 micron or less and most droplets were very fine at 3 microns or less (Fig. 6). The oil-sea water dispersion with an initial concentration of about 40 ppm was stable for about one hour. Although the oil content of the oil-sea water dispersion with an initial concentration of about 10 ppm fell with time, there was no great drop after standing for 20 minutes (Figs. 7, 8).

As shown in Fig. 1, the carbon count distribution from a component analysis of crude oil added to sea water and shaken, then left to stand for one day, revealed n-paraffins with carbon counts of 10 to 25. Although the component ratios differ, in the test material component analysis this time the carbon count distribution is similar and it is considered that this test material can be used as a model of sea water for maritime crude oil spills.

(8.2.2)

Since the oil droplets prepared by the ultrasonic method are mainly an extremely small one micron in size, they float up slowly providing a stable oil content.

The above facts have clarified the effectiveness of preparing oil-sea water dispersions using fuel oil A as the oil type and ultrasonic treatment as the means of dispersion and a scaled up version of this method has been adopted for the preparation of oil-sea water dispersions by the oil additive adjustment equipment for the pre-treatment plant.

(8.2.2)

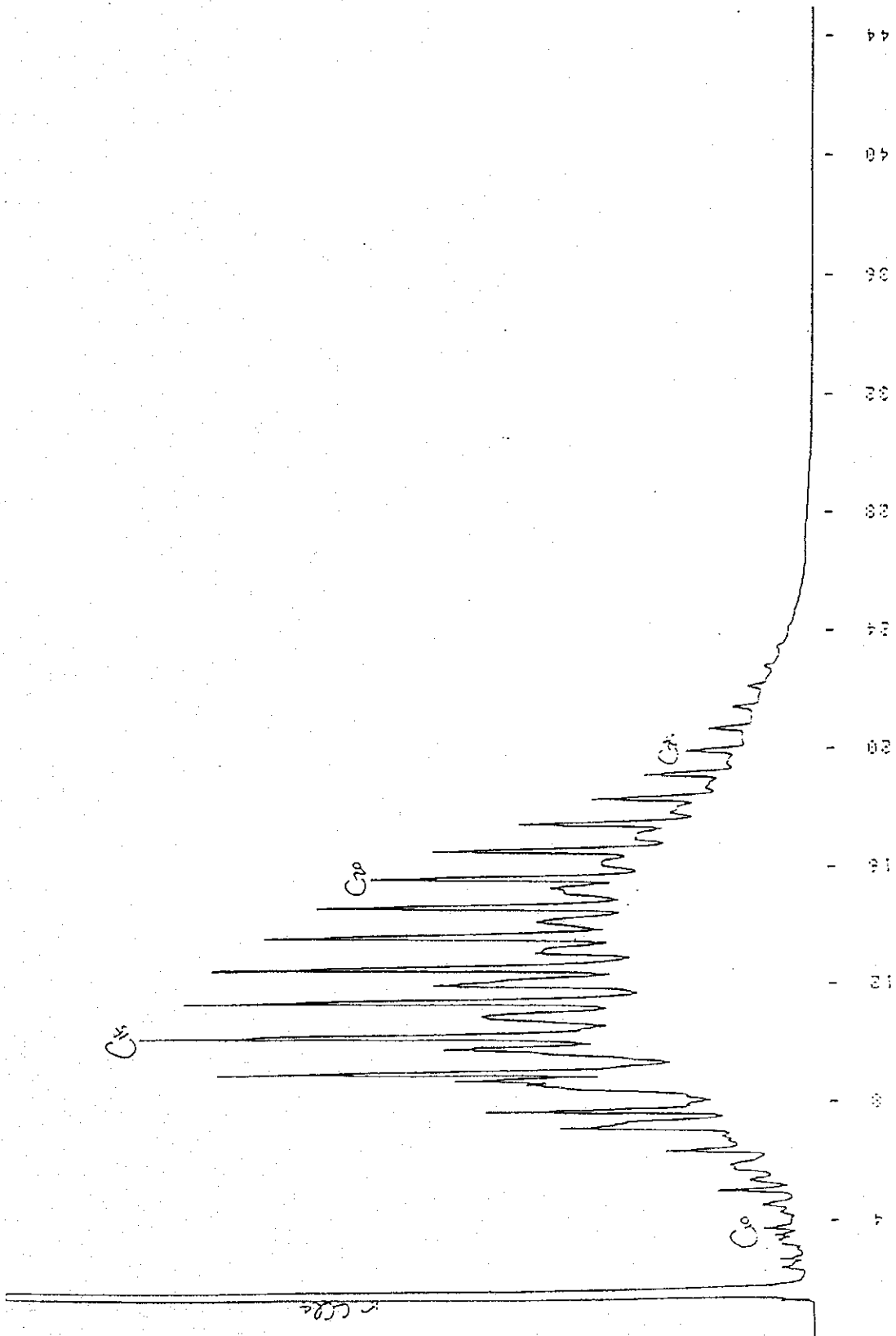


Fig. 5 Chromatogram for fuel oil A dispersed sea water

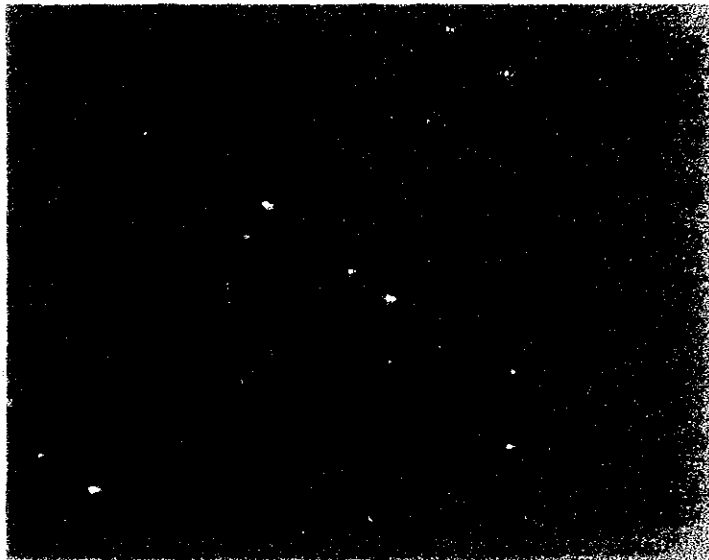


Fig. 6 Oil-sea water dispersions prepared by the ultrasonic method.
(x 400)

(8.2.2)

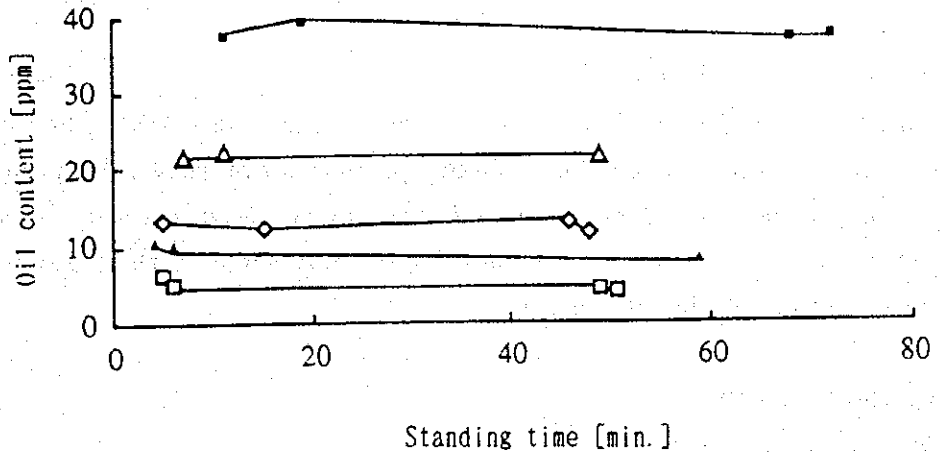


Fig. 7 Temporal changes in oil-sea water dispersions prepared by the ultrasonic method (1).

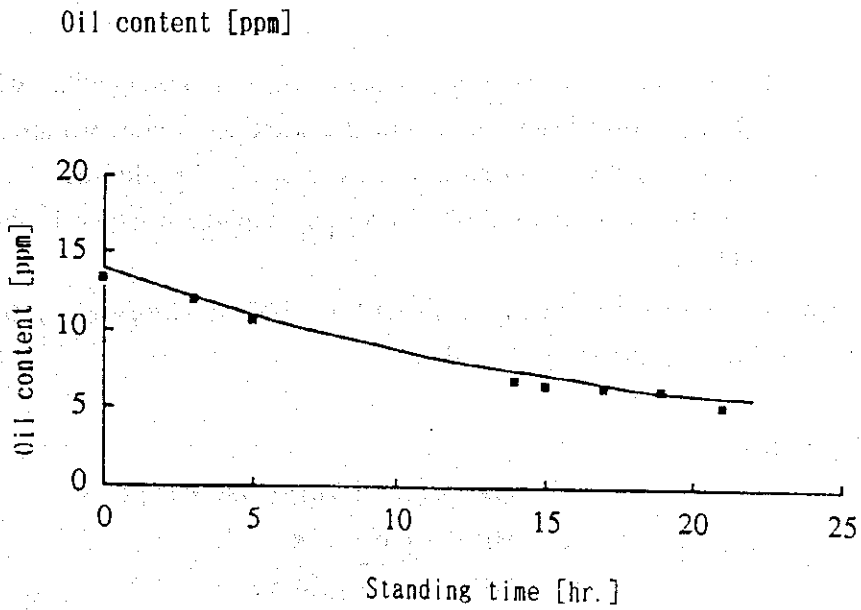


Fig. 8 Temporal changes in oil-sea water dispersions prepared by the ultrasonic method (2).

(8.2.2)

(Reference)

Composition analysis in the case of an oil-sea water dispersion made with diesel oil

(1) **Background and Objective**

As discussed under section 2.2 "Argument for using Diesel Oil", when we were unable to use the intended fuel oil A or a similar product for oil removal experiments with the local pre-treatment plant, diesel oil was hastily used instead. Accordingly, we prepared an oil-sea water dispersion by the ultrasonic method using diesel oil and confirmed the component analysis.

(2) **Experimental method**

Apparatus and reagents

GC-MS analysis equipment

Ultrasonic homogenizer (Ultrasonic Engineering USH-300Z20S)

Peristaltic pump (Tokyo Rikakiki RP-1000)

Silicone tube

Sea water

Diesel oil No. 2

Methylene chloride (Fisher, Optima grade)

Procedure

- While circulating one liter of sea water through the ultrasonic homogenizer with the peristaltic pump, inject 2.5 ml of fuel oil A into the silicone tube in which the sea water is circulating and continue the ultrasonic treatment for 10 minutes.
- Add sea water to dilute the concentrated oil-sea water dispersion which is obtained and adjust to about 30 ppm.
- Extract the adjusted oil-sea water dispersion with methylene chloride and analyse the composition of the oil content by GC-MS.

GC-MS analysis conditions

Analyser	:	HP 5880-GC-MSD HP 5970B
Recorder	:	HP Plotter 7475A
Column	:	HP 1, 25 m capillary
Injected volume	:	3 μ l
Temperature of column oven	:	50°C

(8.2.2)

Temperature of interface	:	280°C
Injection temperature	:	250°C
Gradient of column temperature	:	1) Initial : 50°C, 3 min
		2) Gradient : 10°C/min
		3) Finishing : 280°C, 10 min

(3) Results and discussion

The results of preparing the oil-sea water dispersion and conducting the component analysis on the oil content confirm the presence of hydrocarbons with n-paraffins carbon numbers of about 9 to 24 (Fig. 9). Compared with the case of fuel oil A, this represents a slight shift and the slight differences or no difference from the component distribution after crude oil is added to sea water and left to stand for one day.

The droplets were mainly less than 1 micron and most were less than 3 micron. This is similar to the behaviour with fuel oil A. Time limitations prevented us from investigating the stability of the oil-sea water dispersion on this occasion but, since the oil droplet size is similar to that of the A type fuel oil, we believe that this oil-sea water dispersion which was prepared using diesel oil will have a stability which is similar to that prepared with A type fuel oil.

This leads up to believe that even if diesel oil is used, the ultrasonic method is an effective method for preparing oil-sea water dispersions.

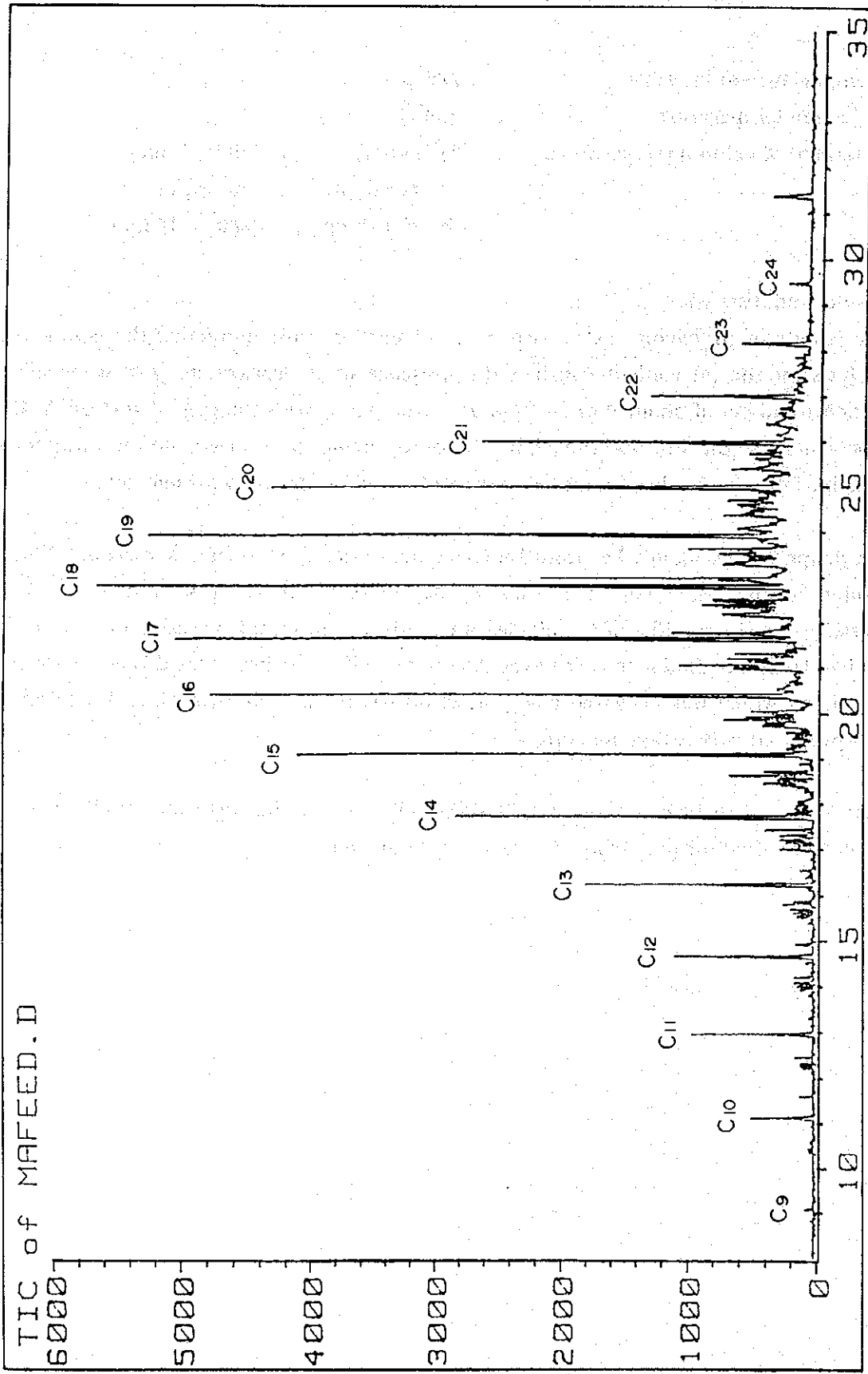


Fig. 9 Chromatogram for diesel oil dispersed sea water

4. Bench Scale Studies

(1) Objective

To confirm that oil-sea water dispersions prepared with the oil addition adjustment equipment on the pre-treatment plant using the ultrasonic method will satisfy requirements of oil content (about 10 ppm) and volume.

(2) Experimental method

Apparatus and reagents

Oil addition adjustment equipment

Fluorophotometer (Nihon Bunko FP-770)

Microscope (Olympus BHS-PC-B-SW)

Separating funnel (100 ml)

Glass funnel

Separating funnel shaker (Iwashiro KM-Shaker, V-SN)

Filter paper (Avantec, 5A 11 cm diameter)

Measuring flask (25 ml)

Sea water

Fuel oil A

Isooctane (Kanto Chemicals, For fluorometry)

Anhydrous sodium sulfate (Wako special grade)

Oil content measurement method

Fluorophotometry

Procedure

From the results of various preliminary studies, the equipment was operated as shown in Fig. 10.

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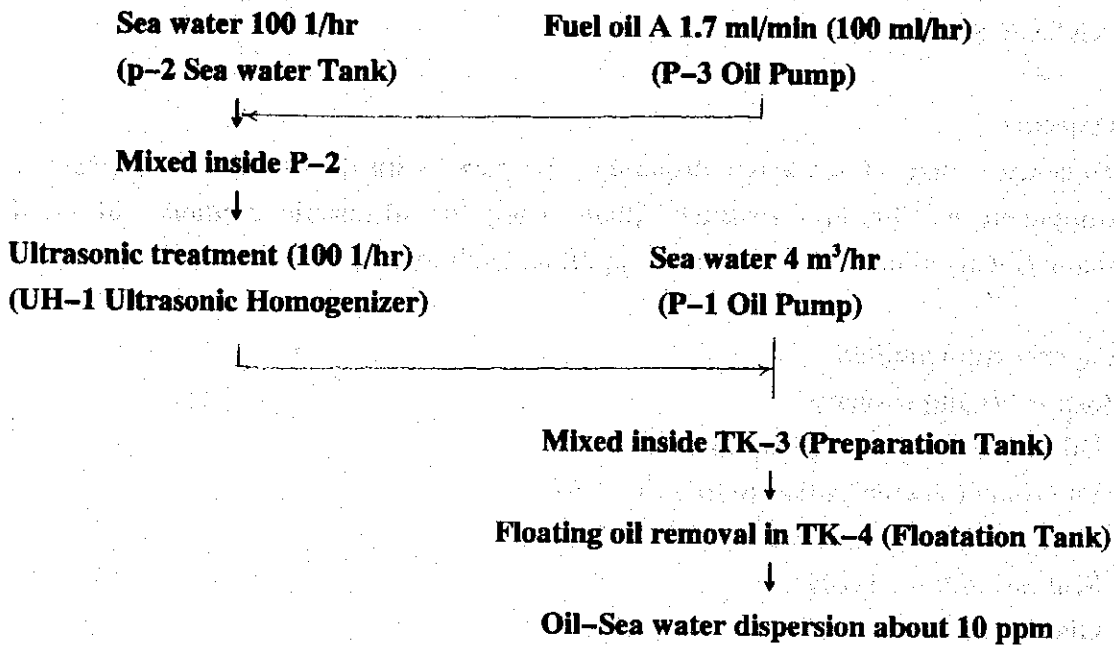


Fig. 10 Preparation of oil-sea water dispersion using oil addition adjustment equipment

Measurement conditions

Slit width : Excitation 10 nm, Fluorescence 10 nm
Excitation wavelength : 310 nm
Fluorescence wavelength : 343 nm

(3) Results and discussion

An oil-sea water dispersion of the target concentration (about 10 ppm) was prepared using the procedure shown in Fig. 10. The oil droplets in this oil-sea water dispersion were extremely fine and uniform at 1 to 3 microns (Fig. 11).

We have confirmed that bulk oil-sea water dispersions can be prepared using this method.

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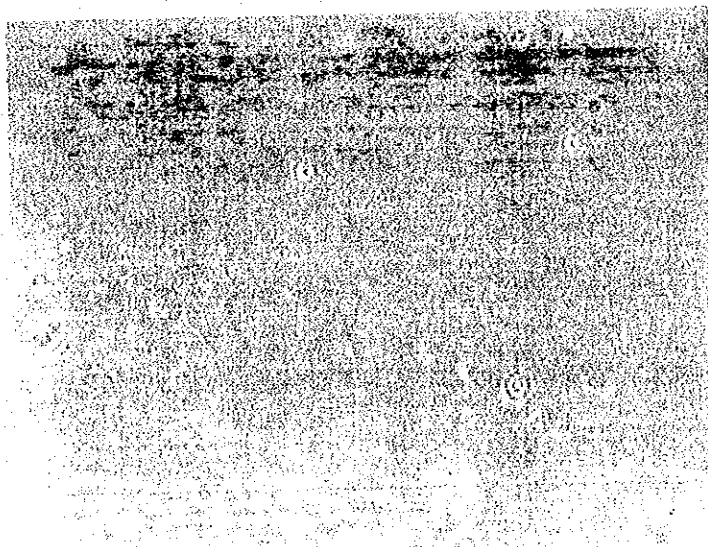


Fig. 11 Oil-sea water dispersion prepared with oil addition adjustment equipment (x 100)

5. Conclusions

- 1) When crude oil is discharge into the sea, the light components evaporate and the heavy components sink. Consequently, this needs to be considered in the typed of oil which is used for the preparation of oil-contaminated sea water.
- 2) Fuel oil A and diesel oil have a component distribution which is similar to that of fuel oil discharged into the sea and left for one day and they are therefore suitable oils to be used experimentally.
- 3) The mixer method and the ultrasonic method are methods of preparing test materials (oil dispersion methods) but the ultrasonic method has the advantages of requiring less preparation time, (which has a bearing on stability of the oil content).
- 4) We have confirmed that the ultrasonic method of preparing test materials satisfies the volumes needed for bench scale experiments (4 ton/hr) and the concentration (10 ppm).

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8.2.3 Oil Adsorption Experiment

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1. Comparison of filter media

1.1 Objectives

This investigation aims to ascertain the performance of three types of filter media, sand, anthracite (chosen as a typical example of an ordinary filter medium) and polymer, regarding the removal of oil content from oil-dispersed sea water by means of direct filtration, using the coagulation conditions established so far. The backwash regeneration performance of these media will also be compared. In addition, the characteristics of oil components that cannot be removed via the direct filtration method will also be investigated.

1.2 Experimental method

Apparatus and reagents

Oil-added sea water preparation apparatus

Oil adsorption removal apparatus

GC-MS analyzer

Fluorescence spectrophotometer (Shimadzu RF-1501)

Microscope (Olympus VANOX-S, AH-2)

Ultrasonic homogenizer (Ultrasonic Engineering USH-300Z20S)

Shaker (Yamato Scientific SA-31)

Separating funnel (100 ml)

Glass funnel

Filter paper (Advantec 5A, ϕ 11 cm)

Volumetric flask (25 ml)

Beaker

Pipet

Membrane filter (Milipore ϕ 47 mm, material polycarbonate,
pore diameter 0.4, 0.2 μ m)

Filter holder (Milipore ϕ 47 mm, material polypropylene)

Syringe (50 ml, material polypropylene)

Kuderna-Danish concentrator

Sea water

Diesel oil No. 2

Anhydrous sodium sulfate (Kanto Chemical, special grade)

Isooctane (Fisher Science HPLC grade)

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- Carbon tetrachloride (BDH analysis grade)
- Methylene chloride (Fisher Optima grade)
- Sand (0.6 mm)
- Anthracite (1.0 mm)
- Polymer filter medium (Chemceptor CM 121, 1.0 mm)
- Ferric chloride solution (chemicals required)

Procedure

The experiment was conducted in accordance with the following procedure:

1) Preparation of oil-dispersed sea water

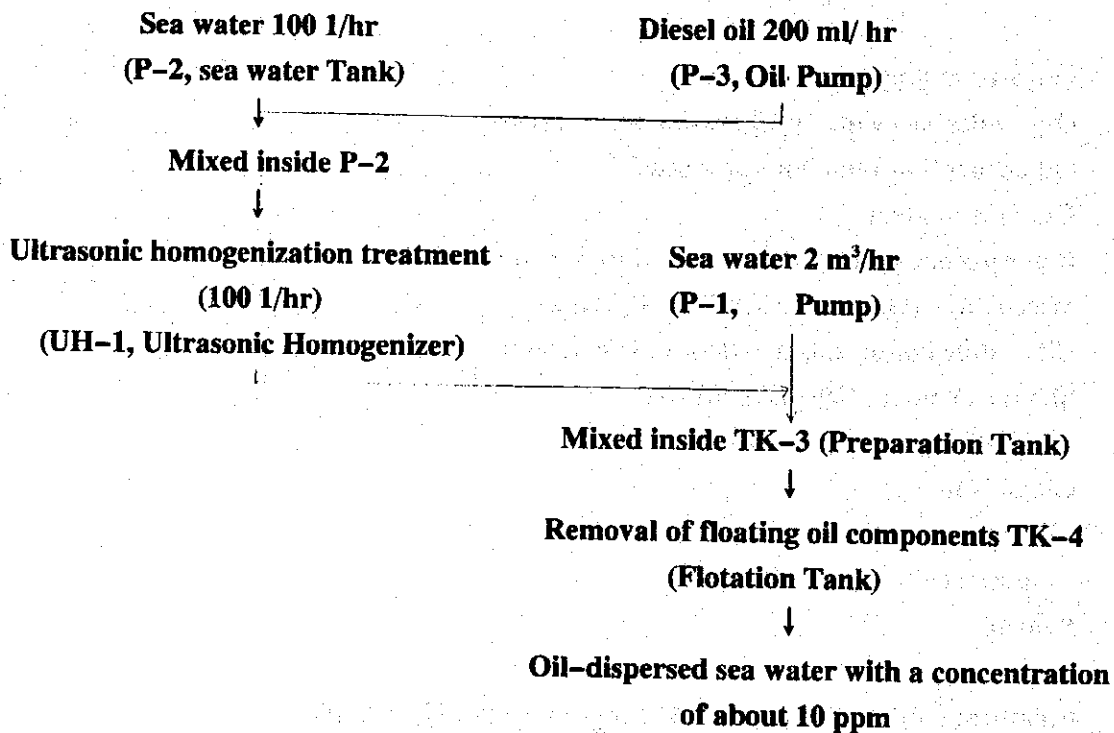


Fig. 1 Oil-dispersed Sea Water Preparation Method based on Oil-added Sea Water Preparation Apparatus

2) Investigation of oil content removal performance of various filter media

Based on the SWCC-JICA joint research results obtained so far, a direct filtration method using ferric chloride as the flocculant was investigated in this experiment, with detailed experiment conditions determined in accordance with the results of a

(8.2.3)

preliminary experiment conducted in Japan.

Oil-dispersed sea water with a concentration of about 5 – 10 ppm provided with an ultrasonic homogenization treatment was passed through a filter column filled with, an anthracite or polymer or sand medium after adding a flocculant, with the oil concentration and pressure differential measured over time. A GC-MS analysis was also carried out on some samples.

The experiment conducted in Japan showed that little of the residual oil content of the filtrate after a direct filtration treatment of oil-dispersed sea water could be removed through further sand filtration. With an actual system, however, it is desirable that a sand filter column be introduced before the RO membrane to protect it from a possible overflow of flocks from the upstream filter medium as a result of equipment problems, an error made in the choice of backwash timing, etc. by capturing them before they reach it. Assuming this to be the actual media configuration, therefore, the direct filtration experiment was conducted incorporating a sand filter column as a second stage filter unit. The flocculant was added just before the transfer pump, and the oil-dispersed sea water and flocculant, after being mixed together in the pump, were introduced into the filter columns past the flocculation column. (Fig. 2)

When breakthrough occurred, the filter media were backwashed with water, with backwash regeneration performance examined. Also, to help investigate the characteristics of oil-dispersed sea water before and after a direct filtration operation, part of its filtrate was further passed through a membrane filter, with the oil content measured before and after the operation.

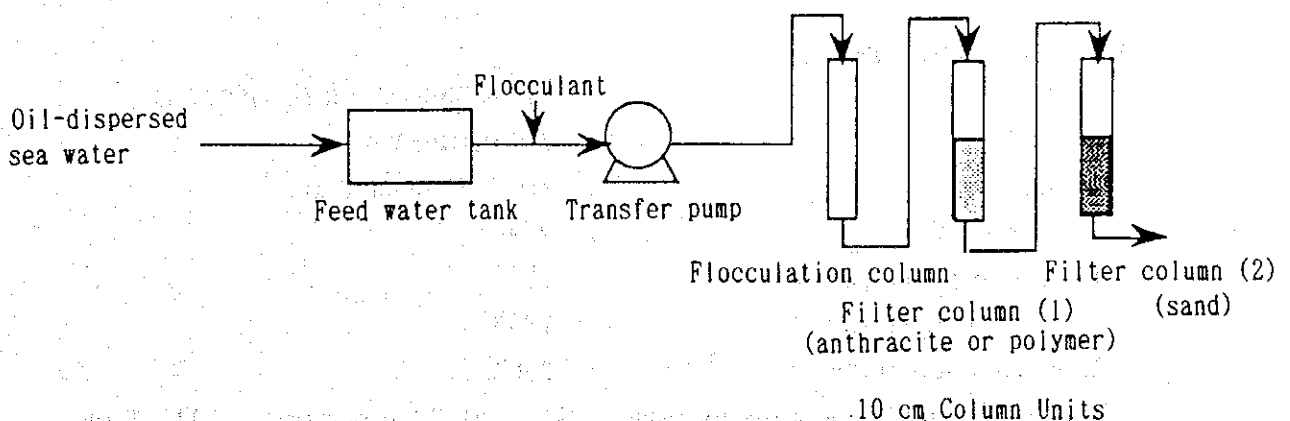


Fig. 2 Flowchart of an Oil Content Removal Experiment based on Direct Filtration

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Experimental conditions

- Column size** : ϕ 10 cm, L 100 cm
- Filter media** : Anthracite (1.0 mm), polymer (Chemceptor CM121), sand (0.6 mm)
- Filter height** : 50 cm
- Feed rate** : 80 l/hr (LV 10 m/hr, SV 20/hr)
- Flocculant** : Ferric chloride, 10 ppm (as FeCl₃)
- Flocculation column** : ϕ 10 cm, L 100 cm; one unit
- Flocculation reaction time** : 12 min
- Backwash method** : Anthracite and sand
- 1) With sea water for 5 min at the LV* specified for the filter medium concerned.
 - 2) With air for 10 min by operating a backwash
 - 3) With sea water for 10 min at the LV* specified for the filter medium concerned.

Polymer

- 1) With sea water for 120 min at the specified LV*.

*: This is the LV at the flow rate that gives the optimum permeation rate derived from the porosity of the filter medium, as shown below:

- Anthracite : 30 m/hr
- Polymer : 10 m/hr
- Sand : 50 m/hr

GC-MS analytical conditions

- Analyzer** : HP 5880-GC-MSD HP 5970B
- Recorder** : HP Plotter 7475A
- Column** : HP 1, 25 m capillary
- Injection volume** : 3 μ l
- Column oven temperature** : 50°C
- Interface temperature** : 280°C
- Injection temperature** : 250°C
- Rate of column temperature increase** : 1) Initial temperature : 50°C, 3min
2) Rate : 10°C/min
3) Final temperature : 280°C, 10min

Measuring conditions

Slit width : Excitation 10 nm, fluorescent 10 nm
Excitation radiation wavelength : 306 nm
Fluorescent emission wavelength : 336 nm

Filtration by membrane filter

Filter : ϕ 47mm, pore diameter 0.2 μ m (Millipore GTTPO4700)
Filtration method : Filtration under pressure with the sample introduced

1.3 Results and discussions

(1) Removal of oil content by means of direct filtration

Figs. 3, 5 and 7 show changes in the oil content removal performance over time of the direct filtration method using anthracite + sand, polymer + sand, and sand + sand as filter media, respectively. Figs. 4, 6, and 8, on the other hand, show changes in oil content and pressure differential in Column No. 1, while Fig. 7 shows the GC-MS chromatogram of each sample.

From these data, the following became clear:

- While almost all the oil content was removed by the direct filtration method, some components, considered to be soluble (mainly low-molecular-weight aromatic compounds), could not be removed with anthracite or sand, a typical ordinary filter medium.
- When a polymer filter medium was used, both the dispersed and soluble oil components were removed.
- With a brand new filter medium, time to breakthrough was about 22 hours for anthracite, 10 hours for the polymer and more than 90 hours for sand.
- The rise in pressure differential was greater with sand than anthracite or with the polymer. Unlike with the anthracite and sand, it also flattened out at a certain point with the polymer.

As far as oil removal is concerned, sand and Anthracite performed identically (see Fig. 4 and 8).

From Fig. 4 and 6, the difference in oil content removal performance between anthracite and the polymer is quite noticeable. However, compared to the feed, most

(8.2.3)

oil components were more or less completely removed in all the cases as shown in Fig. 9 (A, B, C, D), the major difference being whether low-molecular-weight aromatic compounds, considered to be soluble, remained in the filtrate (Fig. 9 (E and F)). GC-MS results of sand and Anthracite were found quite similar.

As shown in Fig. 3, the oil content still remains at 1 ppm after filtration through anthracite and sand. Although this is relative large compared to the feed, which had a 6 - 10 ppm oil content, it consists mainly of low-molecular-weight aromatic compounds, as shown in Fig. 9 (E), and since the component distribution of this sample was fairly similar to that of the filtrate sample obtained by passing oil-dispersed sea water through a membrane filter, it is also considered to be attributable to soluble oil components. It should be noted that, in this experiment, the samples, mainly containing aromatic compounds, were analyzed via fluorescence spectroscopy using a calibration curve obtained from a diesel oil, and this appears to be the reason why measurements were on the high side, as explained in "3.4. Measurement of Soluble Oil Components".

Knap has reported that, according to the results of a GC-MS analysis of oil-dispersed sea water, naphthalene was the most abundant polynuclear aromatic hydrocarbon, considered to be traceable back to this oil content, along with 1,2-dimethylbenzene, 1-ethyl-3-methylbenzene. Attributes the absence of other polynuclear aromatic hydrocarbons to decomposition via photosynthetic oxidization by ultraviolet light contained in the sun's radiation. It is also reported that, apart from oil content, various hydrocarbons derived from marine microorganisms were detected³⁷. Although the results of our experiment generally agree with this report, there is a small difference in detected components. This is considered to be due to the fact that, in this experiment, the oil-dispersed sea water was used relatively soon after its preparation, so that its volatile components as well as those substances which would normally have been decomposed as a result of photosynthetic or biological oxidization remained.

Although direct filtration proved to be capable of removing most oil components regardless of the filter medium, those considered to be soluble (mainly low-molecular-weight aromatic compounds) could only be removed with polymer. The following are probable reasons for this:

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Diesel oil is a mixture of various compounds, most of which are insoluble and dispersed in sea water. However, a small number of components (mainly low-molecular-weight aromatic compounds) dissolve in sea water, though in small quantities. With the direct filtration method, insoluble oil components, which are dispersed in the sea water as oil droplets, are captured by flocs created by a flocculant and removed by the filter medium together with the flocs, while soluble components are considered to pass through it intact as in the case of anthracite and sand. Polymer, on the other hand, is not only a filter medium but also an adsorption medium, with which those components which passed through anthracite can also be removed. In this regard, however, the experiment merely confirmed the existence of such a function and therefore the assessment of polymer as a filter medium should be made only after various basic characteristics (adsorption mechanism, oil content removal performance, regeneration method, regeneration performance, etc.) are investigated.

The difference in the time until breakthrough is attributable to a difference in the porosity of the packed filter medium and the unevenness of its surface, with sand recording a longer time as a result of a larger space available for it to hold flocs. Also, the rise in pressure differential was greater with sand than anthracite and polymer, because sand had a larger packing density than anthracite and polymer, while the flattening out of the rise in pressure differential with the polymer seems to be due to the presence of column sections with a lower packing density, through which flocks escaped.

Based on the above results, the combination of filter media for the actual pretreatment system will be determined taking the following into account:

- A guard column is needed to protect the RO membrane from being blocked by flocs in the event of the breakthrough of the filter medium.
- Direct filtration is capable of removing at least dispersed oil components irrespective of the filter medium used. If this is sufficient for the RO membrane, no further high level treatment is necessary.
- In cases where soluble oil components adversely affect the RO membrane, the use of polymer or activated carbon may be considered.
- The actual filter configuration will be selected from the medium combinations shown in Fig. 10, considering such factors as water quality required by the RO membrane, regeneration performance and oil content removal capacity of various

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filter media, and associated costs.

A microscopic observation of these samples revealed the following:

Feed

Oil droplets are captured by flocs.

Filter Column No. 1 (anthracite or polymer or sand) outlet

Neither flocs nor oil droplets are observed. This does not change whether the filter medium is anthracite or polymer or sand.

Filter column No. 2 (sand) outlet

Neither flocs nor oil droplets are observed.

(2) Backwash regeneration performance

Each filter medium was backwashed after breakthrough and regeneration performance was compared. As a result, the following differences were observed:

- With anthracite and sand, the performance was almost completely regenerated by backwash with water + air, with its characteristics such as the time until breakthrough and pressure differential more or less restored to the original state. However, in the case of polymer, backwash with water alone could not achieve adequate regeneration, with the time until breakthrough and pressure differential becoming longer and larger, respectively. (Figs. 4 and 6)**

The difference in backwash regeneration performance is considered to be attributable to the following: partly due to limitations in the equipment, effective backwashing, unlike the case with anthracite and sand, was not possible with polymer; as a result, flocs accumulated in the filter medium could not be removed completely, and the space to hold them was reduced compared to the initial state. With this filter medium, however, an improvement in regeneration performance through an improved backwashing method seems possible.

(3) The effect of sand (introduced as a second stage) in the direct filtration method

The oil content removal performance of a sand filter column installed after the anthracite or sand(or polymer) column was as follows:

- Oil components which cannot be removed by direct filtration can hardly be**

removed using a sand filter column. However, in the event of flocs overflowing as a result of the breakthrough of the filter medium in the previous stage, the sand filter column will be able to remove them, as well as most of the oil components. (Figs. 3 and 5)

This is considered to be attributable to the fact that sand, like anthracite, only functions as a filter so that it is not effective in removing oil components which could not be removed by direct filtration in the previous stage, replicating the experiment results obtained in Japan.

(4) Examination of the characteristics of oil-dispersed sea water before and after direct filtration experiments

The oil content of feed and filtrate samples of direct filtration operations was measured, both with and without providing further membrane filtration. The results are as follows (Fig. 11):

- By providing the feed with membrane filtration, the oil content was reduced to about 10%.
- A filtrate sample of anthracite direct filtration retained approximately 10% of the oil content of the feed. Providing this sample with membrane filtration was only able to further reduce the oil content slightly.
- With a filtrate sample of polymer direct filtration, the oil content dropped almost to the background level for sea water. Providing this sample with membrane filtration hardly reduced the oil content any further.

Diesel oil is a mixture of various hydrocarbons and the relative abundance of its soluble components is considered to be more or less constant, about 10% according to a fluorescence spectrometric analysis. Therefore, where the relative abundance of aromatic compounds is higher than that, measurements taken using a calibration curve based on diesel oil appear to turn out higher than they should, as the fluorescence spectrometric method used in this experiment selectively measures fluorescent light emitted by aromatic rings. In fact, according to GC-MS chromatograms, almost all oil components have been removed with direct filtration, as shown in Fig. 9 (A - D).

The filtrate sample of direct filtration using anthracite had the same level of residual oil content relative to the feed as that of membrane filtration, presumably because

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dispersed oil components were almost completely removed by direct filtration, with only soluble components remaining. The slight decrease in oil content after providing this filtrate sample with a further membrane filtration treatment is considered to be attributable to a loss of part of these soluble components, which occurred as they were caught by the apparatus used in the filtration operation.

On the other hand, the reason why the residual oil content in the filtrate sample of direct filtration using polymer was reduced to the background level for sea water seems to be the fact that soluble components were removed through adsorption action by the polymer filter medium. An additional membrane filtration to this sample hardly reduced the oil content any further, presumably because the detected substances were not oil components, but amino acids and other aromatic organic compounds, which, as intrinsic components of sea water, were not caught by the apparatus and lost.

These observations agree with the GC-MS diagrams (Fig. 9).

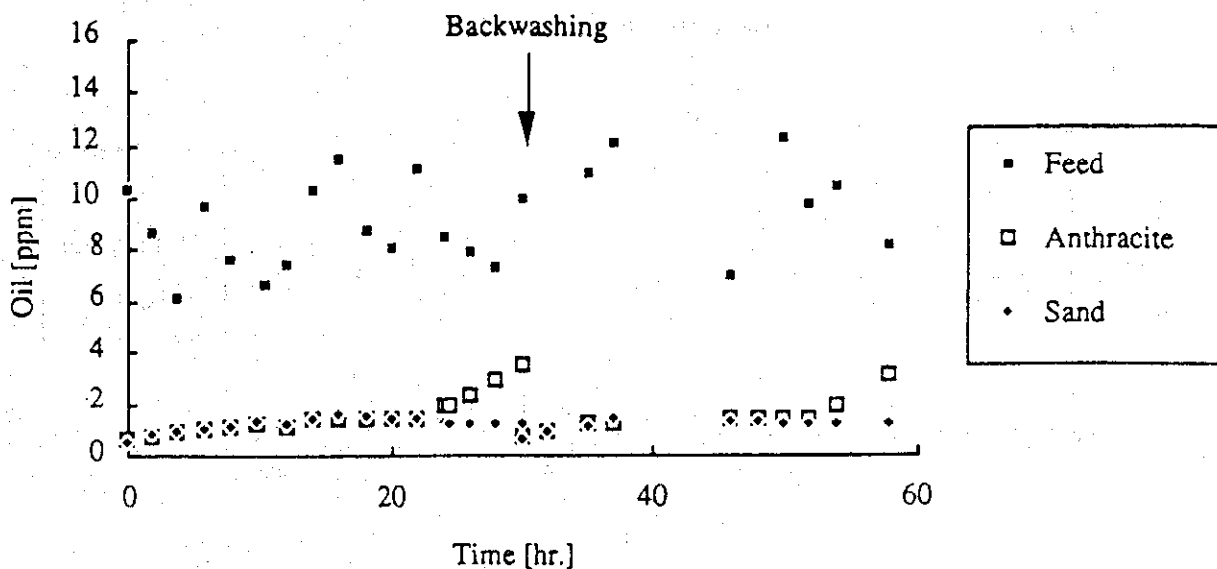


Fig. 3 Direct Filtration based on Anthracite + Sand

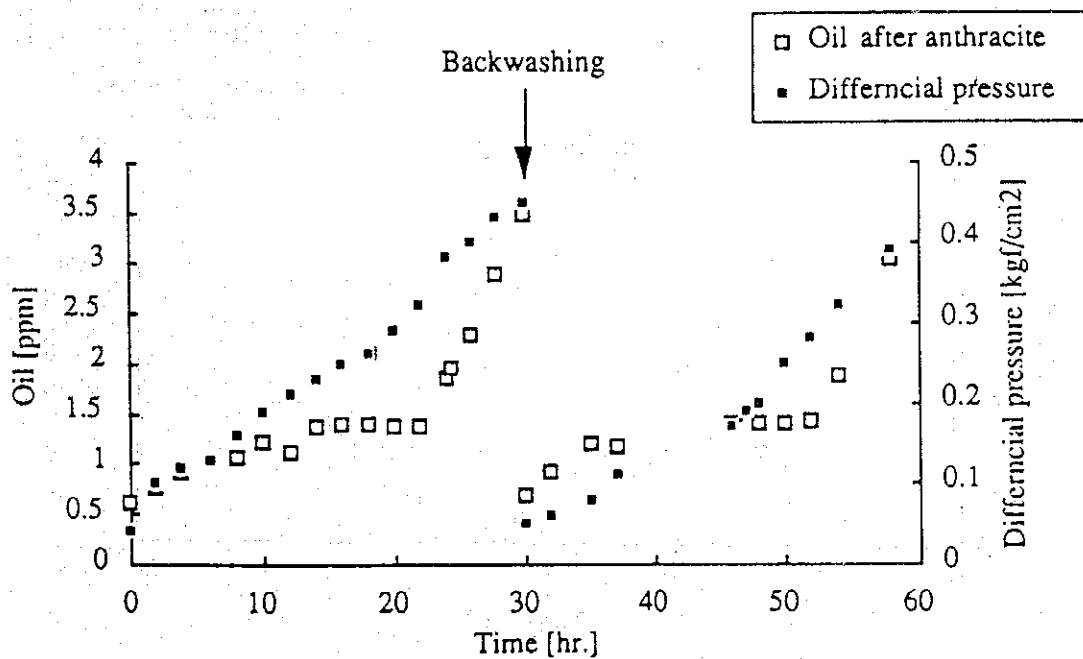


Fig. 4 Relationship between Outlet Oil Content and Pressure Differential for Anthracite Filter

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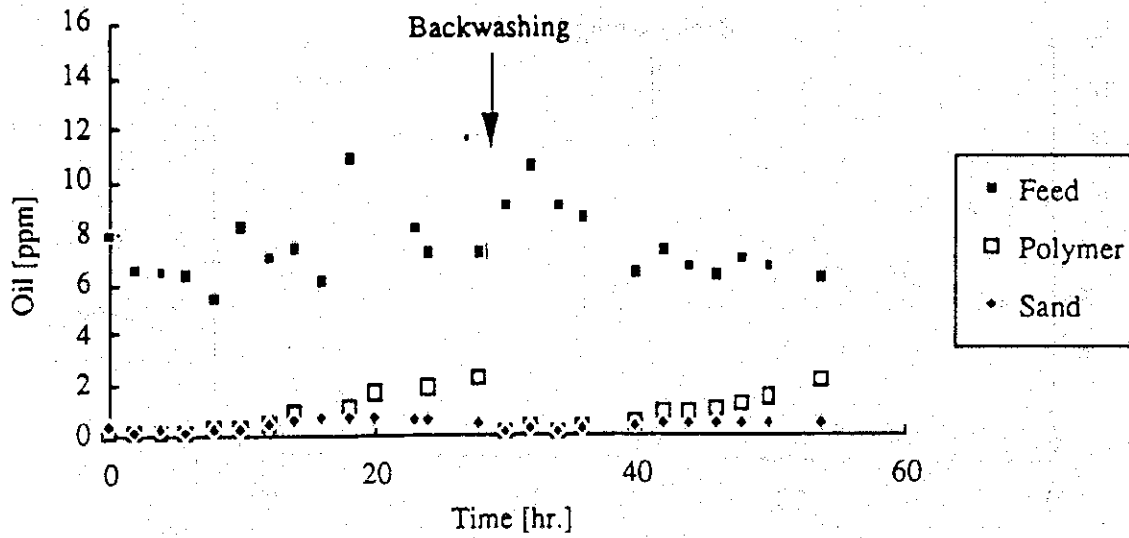


Fig. 5 Direct Filtration based on Polymer + Sand

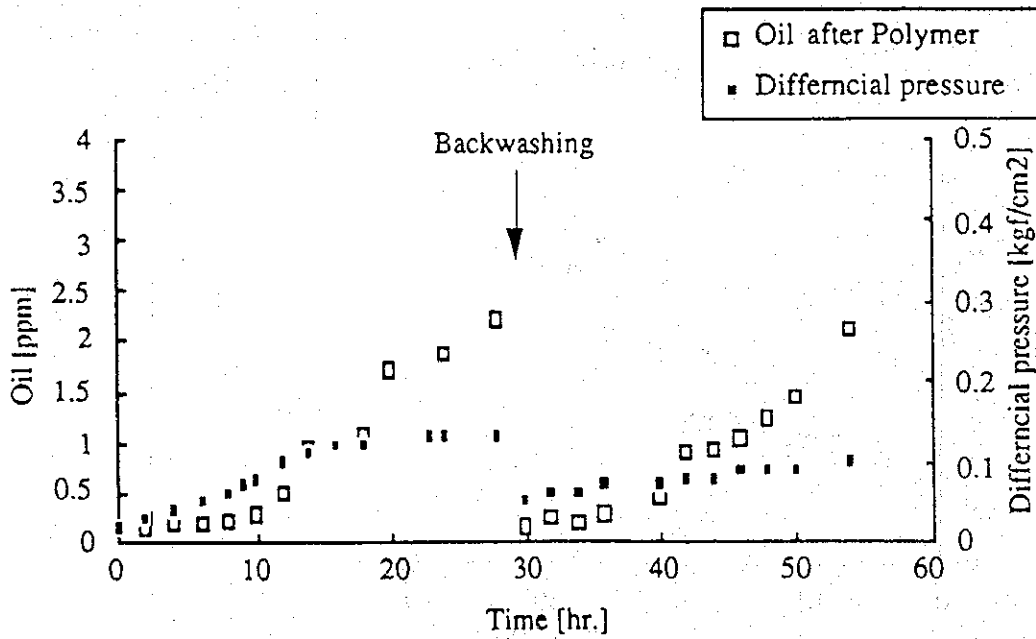


Fig. 6 Relationship between Outlet Oil Content and Pressure Differential for Polymer Filter

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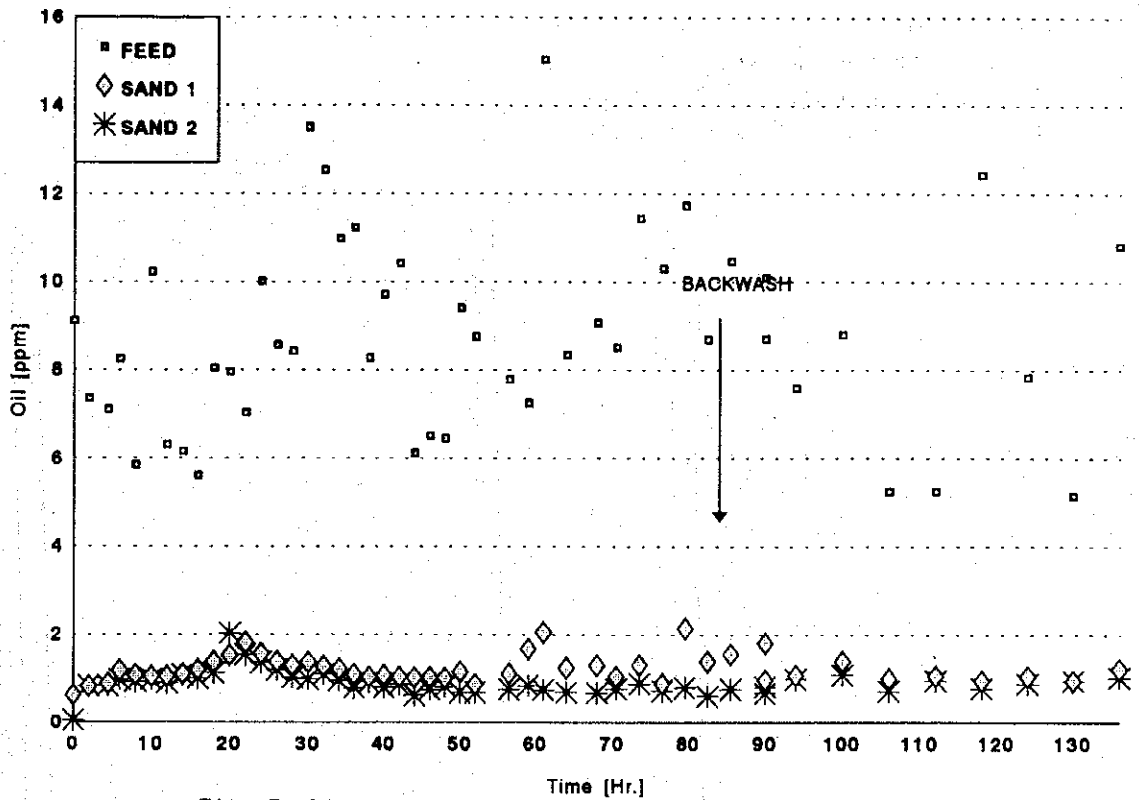


Fig. 7 Direct filtration based on Sand + Sand

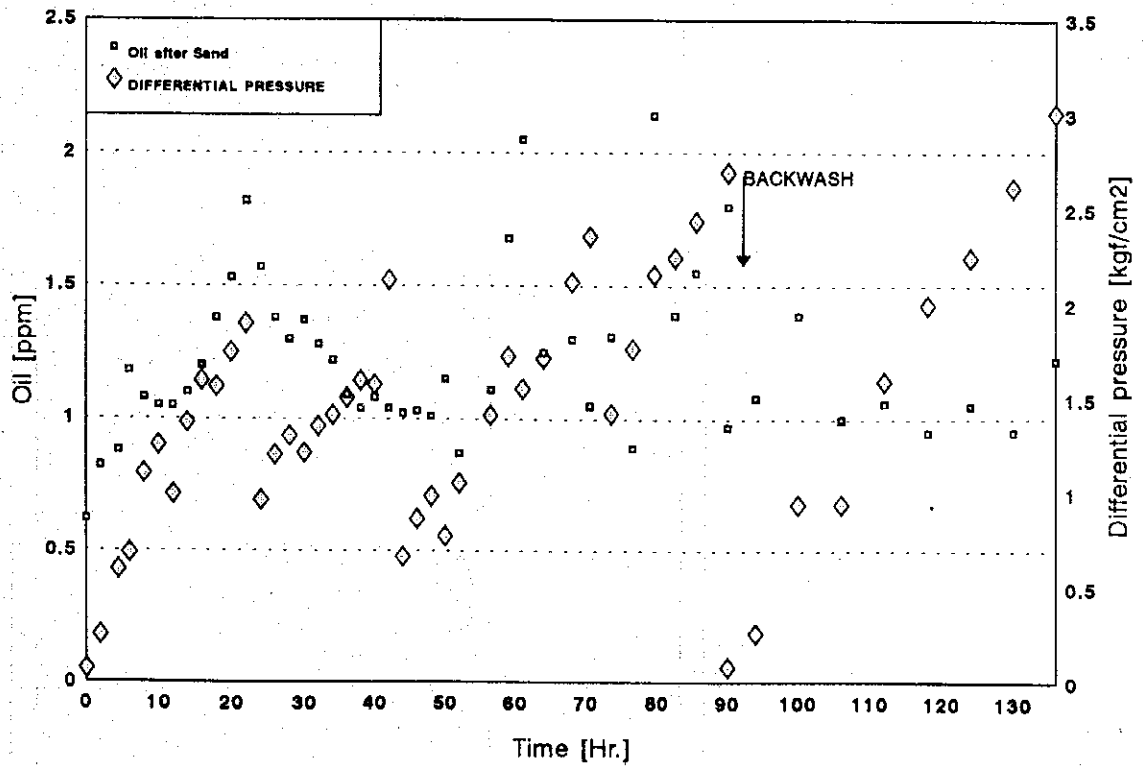


Fig. 8 Relationship between Outlet Oil Content and Pressure Differential for Sand Filter

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- (A) Sea water
- (B) Oil-dispersed sea water
- (C) After anthracite direct filtration
- (D) After polymer direct filtration

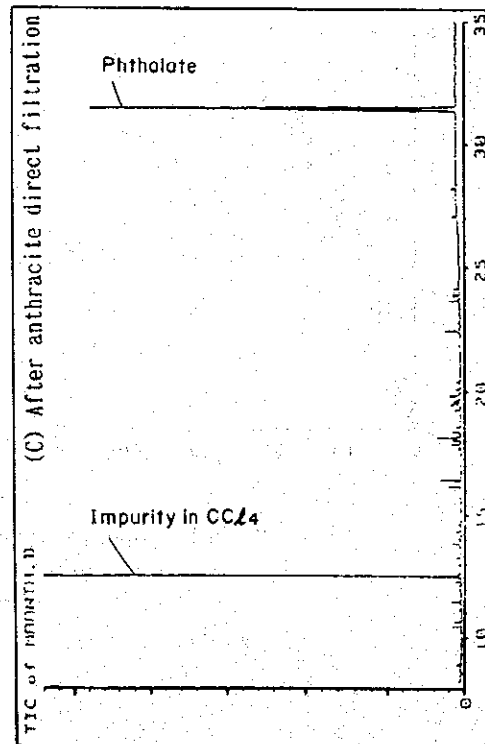
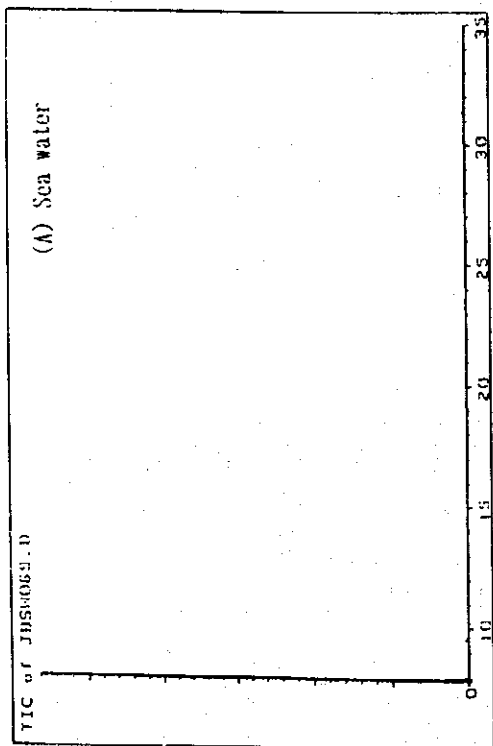
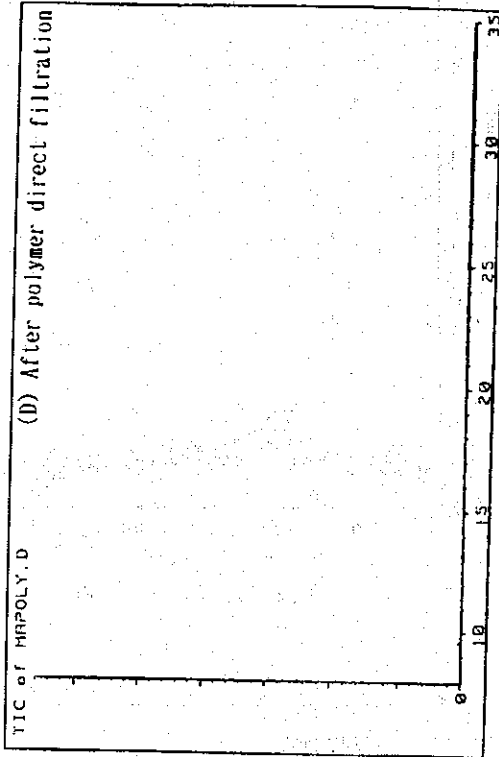
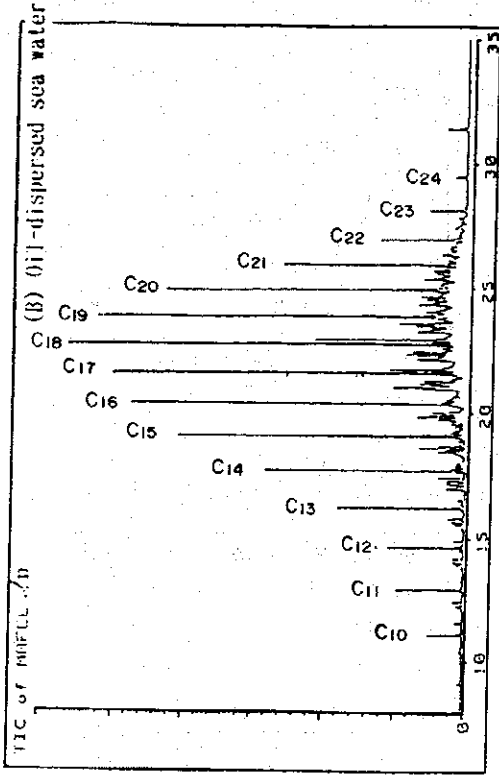


Fig. 9 GC-MS Chromatograms

- (E) After anthracite direct filtration (enlarged)
- (F) After polymer direct filtration (enlarged)
- (G) Sea water used for polymer backwashing (enlarged)

Fig. 9 GC-MS Chromatograms (continued)

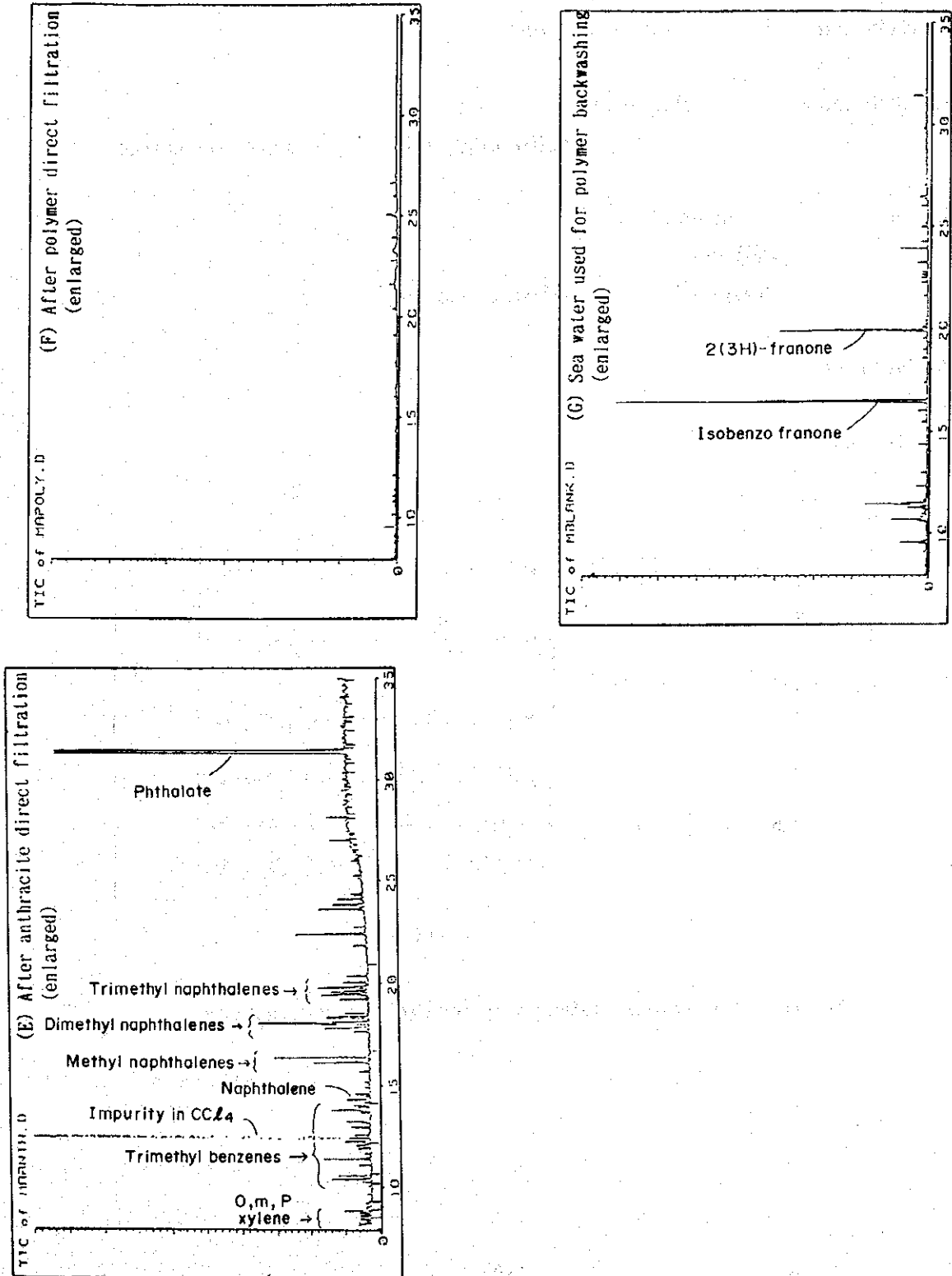


Fig. 9 GC-MS Chromatograms (continued)

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**Oil-contaminated
sea water**

Flocculant (ferric chloride)

Anthracite/sand

Anthracite/sand*

Anthracite/sand

Polymer*

Anthracite/sand*

Activated carbon

Polymer

Anthracite/sand*

Polymer*

Polymer*

Activated carbon

RO membrane

***: Guard column**

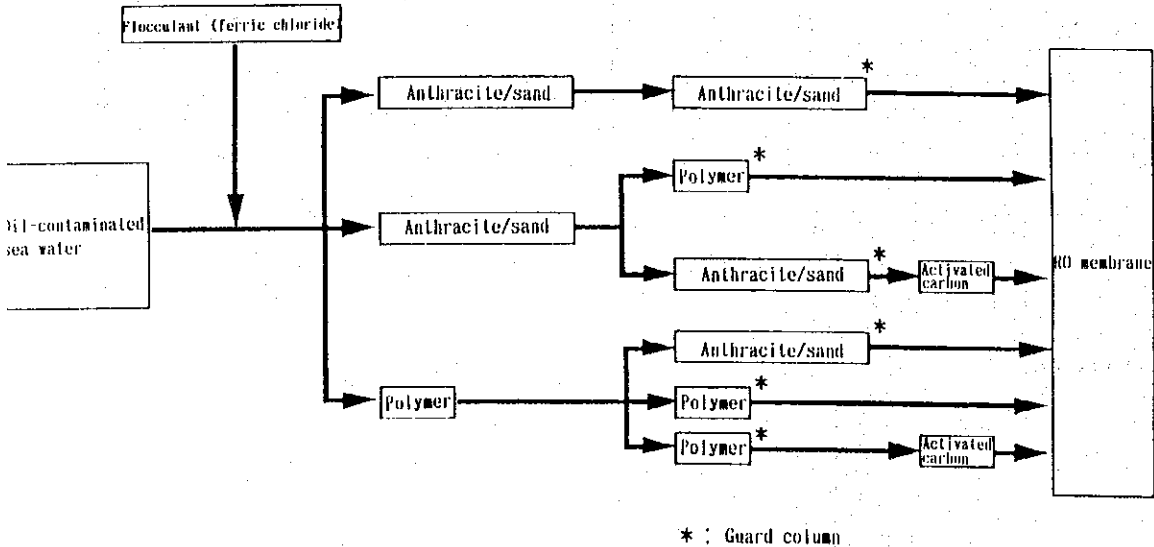


Fig. 10 Oil-contaminated sea water Pretreatment Scheme

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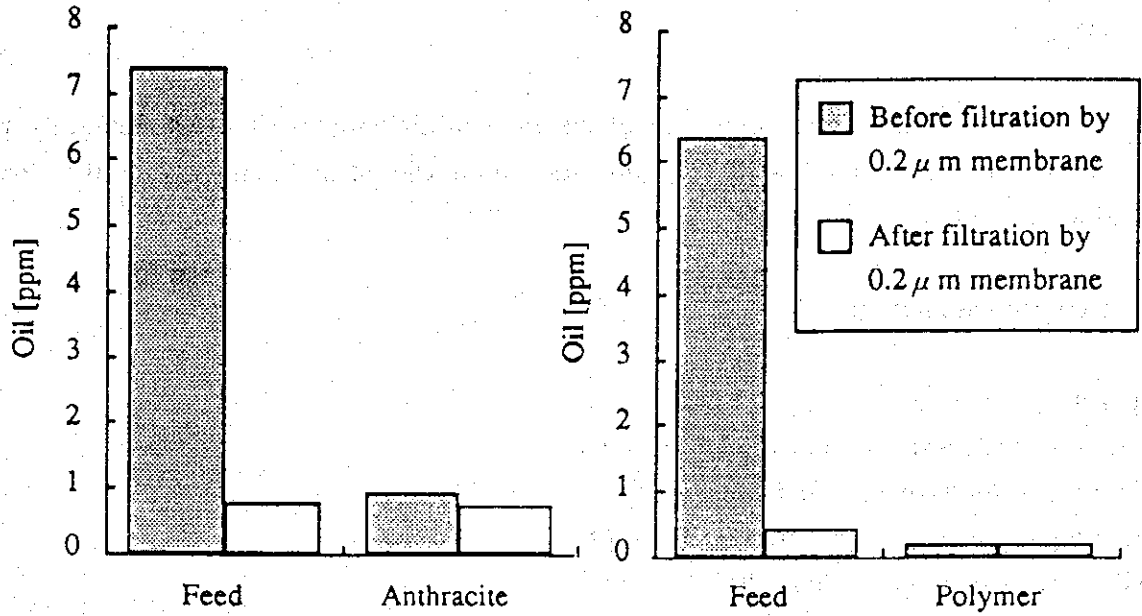


Fig. 11 Oil Content Removal Performance of the Direct Filtration Method using Different Filter Media

2. Removal of oil content by activated carbon

2.1 Objectives

This investigation aims to determine the effectiveness of activated carbon adsorption for the removal of low concentration oil components which cannot be removed by the direct filtration method.

2.2 Experiment method

Apparatus and reagents

Oil-added sea water preparation apparatus

Oil adsorption removal apparatus

Fluorophotometer (Nihon Bunko FP-770)

Microscope (Olympus BHS-PC-B-SW)

Separating funnel (100 ml)

Glass funnel

Separating funnel shaker (Iwaki KM-SHAKER, V-SN)

Filter paper (Advantec 5A, ϕ 11 cm)

Volumetric flask (25 ml)

Sea water

Fuel oil A

Isooctane (Kanto Chemical, for fluorophotometric analysis)

Anhydrous sodium sulfate (Wako, special grade)

Sand (0.6 mm)

Anthracite (1.0 mm)

Activated carbon (Palm trees GW 20 - 42)

Ferric chloride solution (Sankei Kasei)

Oil content measuring method

Fluorophotometry

Procedure

The experiment was conducted in accordance with the following procedure:

- (1) Preparation of oil-dispersed sea water