付属資料 7.3.2-1

ＲＯテストプラントの運転マニュアル

CONTENTS

Page

CHAPTER I. GENERAL

1.0 Introduction 1

2.0 General Description 1

CHAPTER II. DESIGN BASE

1.0 Equipment Design 3

2.0 Seawater Quality 3

3.0 Seawater Temperature 3

4.0 Water Recovery Rate of RO 3

5.0 Permeate Water Quality and Quantity 3

6.0 Flow Balance 4

7.0 Overall Figures of Reverse Osmosis Process Test Plant 4

8.0 General Process Description 6

CHAPTER III. OPERATION PROCEDURE

1.0 Operation Summary 12

2.0 Check List Prior to Plant Start Up 14

3.0 Start, Stop Procedure 15

4.0 Chemicals Handling 29

5.0 List of Setting 29

CHAPTER IV. MAINTENANCE PROCEDURE

1.0 Trouble Shooting for Membrane 32

2.0 RO Membrane Cleaning 40

3.0 Membrane Element Replacement 41

Page

CHAPTER V. WHAT IS RO?

1.0 Principle of Reverse Osmosis 55

2.0 Characteristics of Membrane 60

3.0 Chemicals Compartibility 68

4.0 Spiral Wound Element and Hollow Fiber Element 70

CHAPTER VI. MEMBRANE DETERIORATION AND REJUVENATIONS

1.0 Membrane Deteriorations 74

2.0 Rejuvenation 81

CHAPTER VII. LOG TAKING AND COMMUNICATION

1.0 Necessary Communications 83

2.0 Minimum Log Taking 83

ATTACHMENT

1. Engineering Drawing

1) Basic and Mechanical Drawings

JICA Dwg. No.	Kurita Dwg. No.	Title
SAJ-R1001 1	0T9A-2N03-Y001 1	Plot plan of MSF & RO test plants
" -R1002(1/4) 1	" -Y002(1/4) 1	Pretreatment section-piping & instrument diagram
" -R1002(2/4) 1	" -Y002(2/4) 1	Spiral wourd type RO equipment section-piping & instrument diagram
" -R1002(3/4) 1	" -Y002(3/4) 1	Hollow fiber type RO equipment section-piping & instrument diagram
" -R1002(4/4) 1	" -Y002(4/4) 1	Chemical and utility-piping & instrument diagram
" -R4003 1	" -Y003 1	Plot plan indoor & side view
" -R4004 2	" -Y004 2	Foundation of pipe & cable trench in test plant facility

JICA Dwg. No.	Kurita Dwg. No.	Title
SAJ-R4005 2	0T9A-2N03-Y005 2	Foundation of control panel
" -R4006 1	" -Y006 1	Foundation of pre-treatment skid & chemical feeder skid
" -R4007 1	" -Y007 1	Foundation of tanks
" -R4008 1	" -Y008 1	Connection point of utilityies in RO test plant facility
" -R4009	" -Y009	Foundation of RO skid
" -R4101	" -Y101	Pretreatment skid assembly-section-100
" -R4111	" -Y111	Spiral wound type RO equipment skid assembly-section-200
" -R4112	" -Y112	Hollow fiber type RO equipment skid assembly-section-300
" -R4113	" -Y113	Chemical feeder skid assembly-section-500
" -R4201	" -Y201	Piping assembly
" -R4203(1/3)	" -Y203(1/3)	Detail of piping assembly in pipe trench
" -R4203(2/3)	" -Y203(2/3)	Detail of piping assembly in pipe trench

CHAPTER I. GENERAL

1. INTRODUCTION

This volume provides the system level operation and maintenance instructions for "Reverse Osmosis Process Test Plant". And the original equipment manufacturer's operation and maintenance manuals for purchased system compornents are provided in the Attachment.

Engineering drawings for system assemblies are included in this volume.

2. GENERAL DESCRIPTION

The plant is "Reverse Osmosis Process Test Plant" for Japan-Saudi Arabia Reseurch Project of Seawater Desulination organized by Japan International Cooperation Agency.

In order to investigate, evaluate and establich the most suibable and economical "Reverse Osmosis Desalination System" considering the climatic and environmental conditions and the quality of the seawater in Saudi Arabia, the plant is operated in the various condition and the following confirmation will be made:

1) Performance of Pretreatment System
   (Disinfection, coagulation and filtration)

2) Performance of the membrane elements against the feed seawater salt content, operating pressure, recovery rate.

3) Life of the membrane against high temperature and or high pressure operation.

4) Performance and establishment of Reverse Osmosis Desalination System.

5) Eualuation and establishment of the operation and maintenance for "Reverse Osmosis Desalination System".

CHAPTER II. DESIGN BASE

1.0 Equipment Design

Ambient temperature	:	5 ∿ 45°C
Humidity	:	56 ∿ 70%
Rain fall	:	0 ∿ 47.8 mm/year (in 1976∿1980) (10% voltage fluctuation or less)

2.0 Seawater Quality

pH	:	8.2
TDS	:	44,500 ppm
Cℓ	:	25,000 ppm
Turbidity	:	less than 2

3.0 Seawater Temperature

21°C ∿ 32°C

4.0 Water Recovery Rate of RO

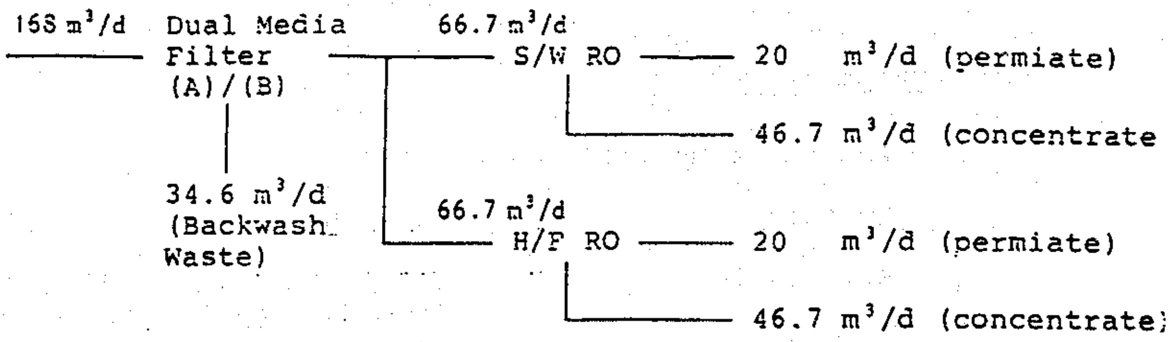
30%

5.0 Permeate Water Quality and Quantity

(Spiral wound type RO and Hollow fiber type RO)

Permeate TDS	:	500 ppm
Permeate productivity	:	20 m^3/d

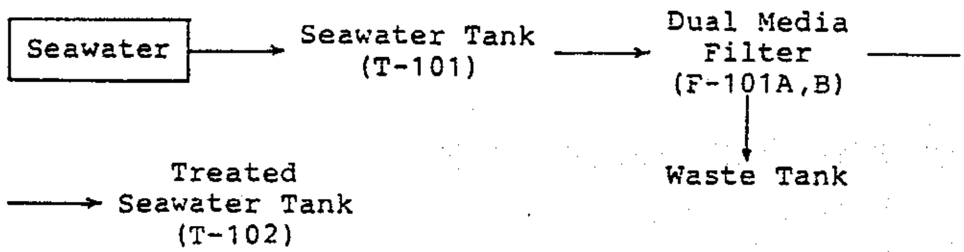
6.0 Flow Balance



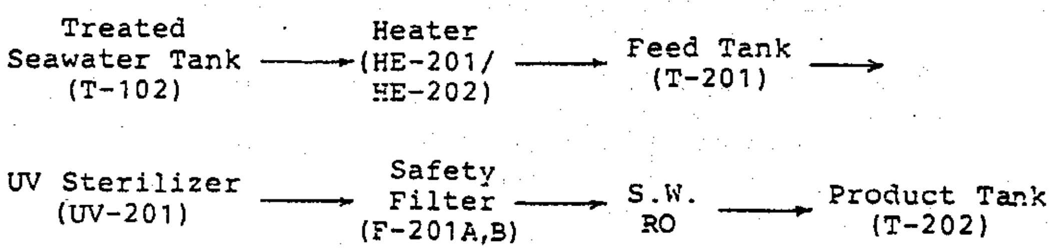
Required water at filter inlet	:	168 m^3/d
RO product water	:	20 m^3/d each
Brine water to dispose	:	46.7 m^3/d
Backwash waste water	:	34.6 m^3/cycle

7.0 Overall Figures of Reverse Osmosis Process Test Plant

1) Pre-treatment Equipment Section



2) Spiral Wound Type RO Equipment Section



3) Hollow Fiber Type RO Equipment Section

Treated Seawater Tank (T-101) → Heater (HE-301/HE-302) → Feed Tank (T-301) →

UV Sterilizer (UV-301) → Safety Filter (F-301A,B) → H.F. RO → Product Tank (T-302)

4) Chemical and Utility

a) Chemical Dosing Equipment Section

Chlorine Tank (T-501) — (NaCℓO) → Pre-treatment Equipment
→ H.F. Type RO Equipment

Coagulant Tank (T-502) ($FeC\ell_3$) → Pre-treatment Equipment

Acid Tank (T-503) (H_2SO_4) → S.W. Type RO Equipment
→ H.F. Type RO Equipment

Deoxidant Tank (T-504) (SBS) → S.W. Type RO Equipment

b) Steam

Steam → Heater for S.W. Type RO Treatment (HE-201/HE-202)
→ Heater for H.F. Type RO Treatment (HE-301/HE-302)

c) Instrument Air

Compressor (P-401) → Air Dehumidifier → RO Plant
→ MSF Plant

8.0 General Process Description

The plant can be divided into four sections constituting pretreatment, S.W. type RO system, H.F. type RO system and chemical dosing system.

1) Pre-treatment Equipment

Suspended matters in the seawater shall be removed before application to the RO equipment.
The pre-treatment equipment section is accommodated to remove the aforementioned suspended matters.
Seawater received in Seawater Tank (T-101) is transferred to Dual Media Filter (F-101A and F-101B) by Seawater Pump (P-101A,B).
Two Dual Media Filters are installed in series.
Suspended matters are removed by 1st Dual Media Filter (F-101A).
2nd Dual Media Filter is accommodate to polish the seawater.
The treated seawater is stored in Treated Seawater Tank (T-102).
Sodium hypochlorite (NaCℓO) and ferric chloride are fed to the seawater at the inlet of Dual Media Filter.
Sodium hypochlorite (NaCℓO) is fed for sterilization, and ferric chloride is fed for coagulant.
Pre-treatment equipment is mounted on the skid.
The 1st Dual Media Filter (F-101A) will be automatically operated by programing timer mounted in control

panel. Therefore, service and regeneration of the 1st Dual Media Filter (F-101A) will be automatically carried out by the programming timer.

The 2nd Dual Media Filter (F-101B) will be manually operated.

Treated seawater is monitored by Automatic Fouling Index Monitor located in test plant control room, and in case of more than 4.0 of fouling index, the alarm will be annunciated.

2) Spiral Wound Type RO Equipment Section

Treated seawater is desalinated by Spiral Wound Type RO Equipment.

Treated seawater stored in Treated Seawater Tank (T-102) is transferred to Feed Preheater (HE-201) and Feed Heater (HE-202) to adjust temperature of the seawater by Feed Pump (P-201), and stored in Feed Tank (T-201). Heating media for Feed Pre-heater (HE-201) and Feed Heater is steam and concentrate respectively. And then, seawater is transferred to UV Sterilizer (UV-201) and Safety Filter (F-201A,B) and to S.W. type RO Module through High Pressure Pump (P-203) to desalinate.

Permeate (Product) is stored in Product Tank (T-202) and concentrate is discharged to trench.

Safety Filter (F-201A,B) is provided with 10 micron polypropylene tube and replacement of filter tube shall be required when the pressure drop will reach approx. 2.45 kg/cm². Spare tubes are provided.

UV Sterilizer (UV-201) is provided for disinfection of the seawater.

Life of germicidal lamp will be within 4000 Hrs. Replacement of germicidal shall be required accordingly.

Spare lamps are provided.

Sulfuric acid and SBS (deoxidant) is fed for pH adjusting and removing residual chlorine and dissolving oxygen respectively.

S.W. Type RO Equipment is mounted on the skid.

3) Hollow Fiber Type RO Equipment Section

Treated seawater is also desalinated by Hollow Fiber Type RO Equipment. Treated seawater in Treated Seawater Tank (T-301) is transferred to Feed Preheater (HE-301) and Feed Heater (HE-302) to adjust temperature of the seawater by Feed Pump (P-201) and stored in Feed Tank (T-301).

Heating media for feed heater and feed pre-heater is steam and concentrate respectively. And then, the seawater is transferred to UV Sterilizer (UV-301) and Safety Filter (F-301A,B), and to H.F. type RO Module through High Pressure Pump (P-302) to desalinate.

Safety Filter (F-301A,B) is provided with 10 micron

polypropylene tube and replacement of filter tube shall be required when the pressure drop will reach approx. 2.45 kg/cm^2. Spare tubes are provided.
UV Sterilizer (UV-301) is provided for disinfection of the seawater.
Life of germicidal lamp will be within 4000 Hrs.
Replacement of germicidal shall be required accordingly.
Spare lamps are provided.
Permeate (Product) is stored in Product Tank (T-302) and concentrate is discharged to trench.
H.F. Type RO Equipment is mounted on the skid.

4) Chemical Dosing Equipment Section
Total four (4) chemical feeders are accommodated in the chemical dosing equipment skid.

a) Chlorine Feeder
Sodium hypochlorite 1.0% solution is used for pretreatment process, and H.F. type RO system.
The dosage of chlorine is adjusted so as to keep 0.2 to 1.0 ppm of residual chlorine at filter inlet and RO inlet respectively.
The dosage of chlorine may vary depending on the contamination of the seawater. One Chlorine Tank (T-501) and two Dosing Pump (P-501A,B) are provided.

b) Coagulant Feeder

Ferric chloride 5% solution is used for pretreatment process in regards to coagulation of suspended matters in the seawater, so that Dual Media Filter can well filtrate.

Dosage of ferric chloride will be manually adjusted in 2 to 3 ppm against the seawater.

One Coagulant Tank (T-502) and one Dosing Pump (P-502) are provided.

c) Acid Feeder

70% of sulfuric acid is used for pH adjustment for both S.W. type RO and H.F. type RO dosage of acid is monitored by pH Recorder (PHRA-301) mounted on the control panel located in Test Plant Control Room and adjusted manually by changing pump stroke. High and low pH alarms are set in pH recorder for annunciation.

One Acid Tank (T-503) and two acid Dosing Pump (P-503A,B) are provided.

d) Deoxidant Feeder

SBS (Sodium Bisulfite) is dosed to scavenge residual chlorine and dissolved oxygen which is contained in the seawater for disinfection.

S.W. type RO Module is not permitted to contain 0 ppm of residual chlorine and 0.5 ppm of dissolved oxygen.

One Deoxidant Tank (T-504) and three Dosing Pump (P-504A,B,C) are provided. Two Dosing Pump (P-504A,B) are operated normally and one Dosing Pump (P-504C) are operated as shock treatment for stopping S.W. type RO treatment.
Dosage of SBS will be monitored by ORP indicator (Oxygen reduction potential indicator - ORP-201) mounted on the control panel located in Test Plant Control Room and adjusted manually by changing pump stroke.

e) Spare Dosing Pump

Eight (8) complete chemical dosing pumps are provided as spare to the event such that failure of any chemical dosing system and/or dosing of rejuvenating chemical for the R.O. membranes when necessary.

CHAPTER III. OPERATION PROCEDURE

1.0 Operation Summary

The followings are summary of important practices to be observed during normal operation.

1) Pretreatment

The flow adjustment for Dual Media Filter (FIL-101A,B) should be checked.

Feed water flow	7.0 m^3/Hr
Backwash water flow	25.0 m^3/Hr
Backwash air flow	36.0 m^3/Hr

2) S.W. Type RO System

2-1) The flow adjustment for S.W. type RO system should be checked.

Permeate flow	0.84 m^3/Hr
Concentrate flow	1.95 m^3/Hr

(The above figures base on 30% of water recovery rate.)

2-2) The pressure adjustment for S.W. type RO system should also be checked.

Operating pressure will be 50 to 60 kg/cm^2G.

2-3) Residual chlorine and dissolving oxygen in RO feed

Keep 0 ppm of residual chlorine and max. 0.5 ppm of dissolving oxygen.

(This will be caused the perfect remedy for operating S.W. RO system.)

2-4) pH in RO feed

Keep RO feed pH 4.0 to 8.5.

2-5) Fouling index

Keep RO feed fouling index 4 or less.

Always ensure pretreatment operation.

2-6) Temperature in RO feed

Keep 21 to 50°C of temperature in RO feed.

3) H.F. Type RO System

3-1) The flow adjustment for H.F. type RO system should be checked.

Permeate flow	0.84 m^3/Hr
Concentrate flow	1.95 m^3/Hr

(The above figures base on 30% of water recovery rate.)

3-2) The pressure adjustment for H.E. type RO system should be checked.

Operating pressure will be 50 to 65 kg/cm^2G.

3-3) Residual chlorine in RO feed

Keep 0.2 to 1.0 ppm of residual chlorine.

3-4) pH in RO feed

Keep 4.0 to 7.0 of pH in RO feed.

3-5) Fouling index

Keep RO feed fouling index 4 or less.

Always ensure pretreatment operation.

3-6) Temperature in RO feed

Keep 21 to 50°C of temperature in RO feed.

2.0 Check List Prior to Plant Start Up

1) Verify that all high pressure victaulic couplings are properly tightened.
2) Verify that all electric sequences are healthy.
3) Verify that pretreatment system are producing adequate water quality which meet membrane requirement.
4) Verify that instrument air system is healthy.
5) Verify that no leak on the water, air and chemical dosing lines.
6) Verify that all necessary chemicals are properly prepared with adequate freshness and dilution.
7) Verify that all chemical metering pumps are healthy and well stroke-adjusted.
8) Verify that all membrane elements are properly loaded.
9) Verify that high pressure pump is well prepared in the alignment, V-belt tension, oiling, etc.
10) Verify that all automatic valves are properly functioning.

11) Verify that all timers are properly set.

12) Verify that correct electric source is wired.

3.0 Start, Stop Procedure

1) Start up Procedure

a) Verification for manual valves

Verify that the manually operated valves located throughout the system have been set as follows. (For valve number, refer to the attached "Piping and Instrument Diagram, DWG No. SAJ-R-4002 (1/4 ∿ 4/4)".

Valve No.	Situation	Remarks
V-101	Open	
V-102	Close	
V-103	Open	Note-1
V-104	Open	
V-105	Open	
V-106	Open	
V-107	Close	
V-108	Close	Note-2
V-109	Close	
V-110	Close	
V-111	Close	
V-114	Close	Note-3
V-115	Open	
V-116	Open	
V-117	Close	Note-4
V-118	Open	
V-120	Close	
V-121	Close	)
V-122	Close	) Note-5

Valve No.	Situation	Remarks
V-123	Open	
V-124	Open	
V-201	Open	
V-202	Open	Note-6
V-203	Close	
V-204	Open	
V-205	Open	Note-6
V-206	Open	
V-207	Open	
V-208	Close	
V-209	Open	Note-6
V-210	Open	
V-211A,B	Open or Close	
V-212A,B	Close or Open	
V-216	Throttled and adjusted	
V-218	Open	Note-7
V-219	Close	
V-220	Close	Note-8
V-221	Close	
V-222	Open	
V-224	Open	Note-9
V-225	Close	
V-226	Close	
V-227	Close	Note-5
V-228	Close	
V-229	Close	
V-230	Open	Note-7
V-231	Close	
V-232	Open	
V-233	Close	
V-240	Close	Note-11
V-301	Open	
V-302	Open	Note-6
V-303	Close	
V-304	Open	

Valve No.	Situation	Remarks
V-305	Open	Note-6
V-306	Open	
V-307	Open	
V-308	Close	
V-309	Open	Note-6
V-310	Open	
V-311A,B	Open or Close	
V-312A,B	Close or Open	
V-316	Throttled and adjusted	
V-318	Open)	Note-7
V-319	Close)	
V-320	Open)	Note-8
V-321	Close)	
V-322	Open	
V-324	Open)	Note-9
V-325	Close)	
V-326	Close	
V-327	Close)	Note-5
V-328	Close)	
V-329	Close	
V-330	Open	Note-7
V-331	Close	
V-332	Open	
V-333	Close	
V-340	Close	
V-401	Open	
V-402	Close	
V-403	Open	
V-404	Close	
V-501A,B	Open	
V-502	Open	
V-503A,B	Open	
V-504A,B,C	Open	

Valve No.	Situation	Remarks
V-505	Close	
V-506	Close	Note-6
V-507	Close	
V-508	Close	

Note-1 When UV Sterilizer (UV-101) will be out of order, by-pass of UV-101 shall be used. In the case, sodium hypochlorite shall be dosed by P-501A.

Valve No.	Situation
V-102	Open
V-103	Close
V-104	Close

Note-2 Dual Media Filter - B (F-101B) for polishing shall be manually operated.

Machine No. Valve No.	Service	Backwash					
		Drainage	Air Bubbling	Pause	Back-washing	Pause	Flashing
V-105							
V-106							
V-107							
V-108							
V-109							
V-110							
V-111							
P-101							
P-102							
P-103							
Operating Time	*1	*2	10 min.	3 min.	15 min.	5 min.	15 min.
Flow Rate	7 m^3/Hr	-	36 m^3/Hr	-	25 m^3/Hr	-	7 m^3/Hr

*1. Backwash shall be manually carried out once a week or when pressure drop of F-101B will reach 0.5 kg/cm at the service.

*2. Drainage shall be made until water level in Dual Media Filter (F-101B) will reach at the surface of the anthracite.

Note-3 When no operation of Dual Media Filter (F-101A,B) will be done, by-pass line of F-101A,B shall be operated. (V-114 open)

Note-4 When the quality of the treated seawater will be over the limit (fouling index will be more than 4.0), the treated seawater of Dual Media Filter (F-101A,B) shall be re-treated by Dual Media Filter (F-101A,B).

Note-5 When water in the tank will be drained out, these valves shall be open.

Note-6 The by-pass line of the respective machine or unit such as HE-201, HE-301, UV-201, UV-203 will be operated, by-pass valves such as V-203, V-208, V-303, V-308 shall be open.

Note-7 When no operation of Heater (HE-201, 202, 301, 302) will be done, the following procedure shall be carried out.

V-218, 318, 230, 330	Close
V-219, 319	Open

Note-8 See "Chemical Cleaning of RO Module".

Note-9 When the permeate of S.W. RO system and/or H.F. RO system will be recirculated, the permeate shall be flowed back to the respective "Feed Tank" (T-201 or T-301).

b) Verification for automatic valves

Valve No.	Situation	Remarks
AV-101	Open	
AV-102	Open	
AV-103	Close	
AV-104	Close	Note-1
AV-105	Close	
AV-106	Close	
AV-107	Close	
AV-108	Close	
TSU-401	Open	Note-2

Note-1 Automatic operation of Dual Media Filter (F-101A) is as follows:

Machine No. Valve No.	Service	Backwash					
		Drainage	Air Bubbling	Pause	Back-washing	Pause	Flashing
AV-101	—						—
AV-102	—						
AV-103					—		
AV-104					—		
AV-105			—				
AV-106		—	—				
AV-107							—
AV-108		—					
P-101	—						—
P-102					—		
P-103			—				
Operating Time	24 Hrs	3 min	10 min	3 min	15 min	5 min	10 min
Flow Rate	7.0m³/Hr	-	36m³/Hr	-	25m³/Hr	-	7.0m³/Hr

The above processes are automatically operated by pre-setting timer.

All selector switches for automatic valves are in AUTO positions.

3) Verification for instrumental setting points

Verify all set points and timer setting based on 6.0 "LIST OF SETTING".

4) Turn on the main circuit breaker for incoming power and then turn on all circuit breakers located in control panel.

Confirm air compressor start working.

5) Turn selector switches for chemical dosing pumps to AUTO position.

6) Turn on push button for air compressor.

7) S.W. type RO system start procedure

When all equipments are ready for automatic operation, S.W. type RO system is operated as follows:

Push button of S.W. type RO system ON

P-201, P-202, P-503A, P-504A, P-504B,
UV-201 ON

(10 sec.)

P-203 ON

8) H.F. type RO system start procedure

When all equipments are ready for automatic operation, H.F. type RO system is operated as follows:

Push button of H.F. type RO system ON

P-301, P-302, P-501B, P-503B, UV-301 ON
(10 sec.)
P-303 ON

9) Check and adjust all necessary items and correct based on "Operation Summary".

2) Stop Procedure

(1) Stop RO System

1-1) S.W. Type RO System

Automatic stop procedure is as follows:

Push STOP button

P-504C ON
(2 min.)
P-203, P-504C STOP
(3 sec.)
P-201, P-202, P-503A, P-504A,
P-504B, UV-201 STOP

1-2) H.F. Type RO System

Automatic stop procedure is as follows:

Push STOP button

P-303 STOP
(4 sec.)
P-301, P-302, P-501B, P-503B,
UV-301 STOP

(2) Pretreatment

Turn on the selection switches of all equipment to "AUTO".

P-101 start, AV-101, AV-107 open

(Flashing of Dual Media Filter - A (F-101A) is carried out before service.)

10 min.

AV-102 open, AV-107 close

(Then, Dual Media Filter is in service.)

24 Hrs or pressure drop of F-101A will reach 0.5 kg/cm^2.

Backwash process is automatically carried out.

(Drainage - Air bubbling - Pause - Backwashing - Pause - Flashing)

Service

4) Turn off all circuit breakers on the control panel and close all valves, when the next start is not expected soon.

3) Emergency Stop

(1) S.W. Type RO System

1-1) Abnormal condition of Oxygen Reduction Potential Meter (ORPA-201).

P-203 stop, P-504C start, Annunciator
(2 min.)
P-504C stop
(3 sec.)
P-202, P-503A, P-504A, P-504B stop

1-2) Abnormal low pressure at the suction of P-203 (PSA-201).

P-203 stop, P-504 start, Annunciator
(2 min.)
P-504C stop
(3 sec.)
P-202, P-503A, P-504A, P-504B stop

1-3) Low water level in Feed Tank (T-201) (LSA-201)

P-504C start
(3 min.)
P-203 stop
(3 sec.)
P-202, P-503A, P-504A, P-504B, P-504 stop

1-4) Booster Pump (P-202) motor trip

P-503A, P-504A, P-504B, UV-201 stop
(2 sec.)
P-203 stop, P-504C start
(2 min.)
P-504C stop

In any emergency stop, RO system does not start automatically.
After checking, correcting and readjusting, restart of RO system shall be made.

(2) H.F. Type RO System

2-1) Abnormal pH (PHRA-301 pH < 4 or pH > 7)

Annunciator 9pH low or pH high)

P-303 stop
(4 sec.)
P-302, P-501B, P-503B, UV-301 stop

2-2) Abnormal low pressure at the suction of P-303 (PSA-301)

Annunciator (P < 0.1 kg/cm^2)

P-303 stop
(4 sec.)
P-302, P-501B, P-503B, UV-301 stop

2-3) Low water level in Feed Tank (T-201)

(LSA-301)

P-303 stop

(4 sec.)

P-302, P-501B, P-503B, UV-301 stop

2-4) Booster Pump (P-302) motor trip

P-501B, P-503B, UV-301 stop

(2 sec.)

P-303 stop

The above chemical dosing rate is estimated value. The actual dosing rate shall be decided based on the field test.

4.0 Chemicals Handling

1) Handling

As a rule, polyethylene hand gloves and eye-glasses must be utilized for safety when handling chemicals. Dilution, mixing, conveying and container opening. Washing water must always be prepared for emergency.

2) Storage

Store any chemicals in cool and dark place completely closing chemical containers, bags, cans.

3) Spills

Wash with water to any chemical spills immediately. Splashing to human skin, machines, floor and any part of articles must immediately be flushed with water.

5.0 List of Setting

1) Pretreatment

Item	Setting Value (Estimated)
PI-101	2.0 kg/cm^2G or more
PI-102A	1.8 kg/cm^2G or more
PI-102B	1.2 kg/cm^2G or more
PI-103A	1.2 kg/cm^2G or more
PI-103B	0.6 kg/cm^2G or more
PI-104	0.8 kg/cm^2G or more
FI-101	5 ∿ 8 m^3/Hr

Item	Setting Value (Estimated)
FI-102	25 m^3/Hr
FI-103	36 m^3/Hr

2) S.W. Type RO System

Item	Setting Value (Estimated)
PI-201	2.5 kg/cm^2G or more
PI-202	3.0 kg/cm^2G or more
PI-203	1.0 kg/cm^2G or more
PI-204	50 kg/cm^2G or more
PI-205	50 kg/cm^2G or more
PSA-201	0.1 kg/cm^2G or less (annunciator)
FI-201	0.84 m^3/Hr
FI-202	1.25 ∿ 2.5 m^3/Hr
TI-201	21 ∿ 32°C
TI-202	20 ∿ 50°C
TI-203	20 ∿ 50°C
TI-204	20 ∿ 50°C
TA-201	55°C (annunciator)
ORPA-201	150 mV (annunciator)

3) H.F. Type RO System

Item	Setting Value (Estimated)
PI-301	2.5 kg/cm^2G or more
PI-302	3.0 kg/cm^2G or more
PI-303	1.0 kg/cm^2G or more
PI-304	50 kg/cm^2G or more
PI-305	50 kg/cm^2G or more

Item	Setting Value (Estimated)	
PSA-301	0.1 kg/cm^2G or less (annunciator)	
FI-301	0.84 m^3/Hr	
FI-302	1.25 m^3/Hr ∿ 2.5 m^3/Hr	
TI-301	21 ∿ 32°C	
TI-302	20 ∿ 50°C	
TI-303	20 ∿ 50°C	
TI-304	20 ∿ 50°C	
TA-301	55°C	(annunciator)
PHRA-301	4 or less	(annunciator)
	7 or more	(annunciator)

4) Utility

Item	Setting Value (Estimated)
PI-401	4.0 kg/cm^2G or more
PI-402	6.9 kg/cm^2G or more
PI-403	6.8 kg/cm^2G or more

CHAPTER IV. MAINTENANCE PROCEDURE

1.0 Trouble Shooting for Membrane

1) Introduction

It is recommended to evaluate always the membrane performance of running RO system for finding out future troubles, if it is going to happen, in early age.

The log sheet (see SHAPTER - will be forwarded to the agent and KURITA for this purpose.

The various figures in the log sheet will be plotted graphically for identification.

Salt rejection and the flux are main parameters to chase operating performance. Other various figures in the log sheet are giving the background of main parameters.

Depending on the fed-back log sheet, the evaluation of membrane performance is made.

2) On-line Evaluations

2-1) Performance Classifications

The performance of the membrane will be then classified to four (4) possible cases regarding behaviors of salt rejection and the flux.

The possible classifications (refer to CHAPTER -).

a) Case - 1

Salt rejection	:	Going constant
Flux	:	Going constant or on the line of reasonable compaction

Possible reason	Remedies
Stable operation	None

b) Case - 2

Salt rejection	:	Going down
Flux stable	:	Going constant or on the line of reasonable compaction

Possible reason	Remedies
(1) O-ring leaks	(1) Permeate check Check permeate conductivity on every pressure tube to find out leaks. Then, O-ring correction.
(2) Early age of membrane deterioration	(2) Permeate check For classification of deterioration

c) Case - 3

Salt rejection	:	Going down
Flux	:	Going down

Possible reason	Remedies
(1) Fouling	(1), (2) See CHAPTER -

(2) Scale precipitation

(3) Big salinity increase in raw water

d) Case - 4

Salt rejection :	Going down
Flux :	Going up

Possible reason	Remedies
(1) Physical deterioration	(1), (2), (3) See CHAPTER -
(2) Biological deterioration	
(3) Chemical deterioration	
(4) Released compaction	(4) Give compaction to membrane
(5) Serious O-ring leak	(5) Correct O-ring

2-2) Permeate Check

The permeate conductivity from individual pressure tubes occasionally suggest imporatnt aspect of membrane performance.

Not only O-ring leaks but also other kind of deterioration of membranes are identified by "Permeate check".

The evaluation will always be made by comparison of current values with original value of each permeate conductivity.

There are some classifications.

Case P-1

Almost same electric conductivity are seen in all 1st bank pressure tubes. Higher than 1st tank and almost same conductivity are seen in all 2nd bank pressure tubes.

Possible aspect	Remedies
Normal operation	No

Case P-2

Extremely high electric conductivity are seen on one or more pressure tubes irregularly.

Possible aspect	Remedies
(1) O-ring leaks	(1) Correct O-ring
(2) Biological attack	(2) See CHAPTER -
(3) In-correct brine seal setting or damage on brine seal	(3) Replace brine seal (if any)

Case P-3

Relatively high conductivity and or increasing conductivity are seen in all 1st bank pressure tubes while 2nd bank conductivity are stable.

Possible aspect	Remedies
(1) Physical deterioration is going to take place	(1) See CHAPTER -

Case P-4

High conductivity are seen on all banks pressure tubes.

Possible aspect	Remedies
(1) Chemical deterioration	(1) See CHAPTER -

Case P-5

High electric conductivity are seen on all 2nd bank pressure tubes while all 1st bank conductivity are normal.

Possible aspect	Remedies
(1) Scale precipitation	(1) See CHAPTER -

2-3) Off-line Evaluations

Off-line evaluation will be done after on-line evaluation through both of performance classification and permeate check.

Off-line evaluation necessitates removing membrane elements from pressure tubes for identification.

The following will be clarified by this off-line evaluation at site.

a) Weight measuring

Extremely heavy membrane is accumulating fouling or scale deposit.

Usually weight measuring is carried out after 1 hr drainage of water from membrane element standing feed side up.

Scale accumulating membrane must be replaced.

b) Visual check

Deformations like telescoping of membrane element, U-seal (brine seal) mis-setting or damage, O-ring damage, and etc. are checked and corrected at site.

2-4) Labo-evaluations

For further evaluation, KURITA carryes out labo-investigation to the troubled membrane sent back to Japan in the case that above field evaluation was not enough for trouble shooting.

2) Cleaning frequency

Two times of routine cleanings per year is expected for normal operation. It is recommended to do this two times cleaning per year without reason.
The frequency of cleaning will be increased when upset on the pretreatment occur and or other disturbances.

3) When is RO cleaning made?

a) Six (6) months elapsed since last RO cleaning
b) When salt passage is increased more than 20% not due to O-ring leaks or other obvious reason than fouling.
c) When permeate flux is decreased more than 10% not due to membrane compaction and or high osmotic pressure.

When RO system is in the state of above 1), b) and c) clean RO system without waiting delta-P developes across RO membranes.

d) When delta-P increased more than 30% accompanying salt rejection down and flux down not due to flows, water temperature changes.

4) Procedure of cleaning system

The following cleaning equipment and chemicals are provided.

- 0.5 m^3 of Cleaning Tank (T-601) with necessary nozzles and flexible hoses

· SBS	100% crystal
· Citric acid	100% powder
· Ammonia	25% sol
· Formalin	37% sol
· Sodium hexa-meta phosphate	100% powder

The following procedure for chemical cleaning shall be performed:

a) Connect the necessary line from cleaning tank to RO system (permeate, concentrate and chemical feed line).
   Necessary nozzles for the above connection have been provided.

b) Prepare chemical solutions.
   pH of chemical solution must be checked and corrected by adding ammonia solution.

c) Booster Pumps (P-202, P-302) are used for chemical recirculation. Recirculation time will be 20 to 30 min. so that all membrane elements are completely soaked to the cleaning chemicals.

d) Stop recirculation by stopping Booster Pumps (P-202, P-302) and leave it for 8 to 12 Hrs.
   The fouling matters, metal oxides and/or biological matters whatever will be dissolved and/or removed during this soaking time.

Then, RO system will be flushed by RO feed water. The flushing is carried out without high pressure pump running but RO booster pump. Flushing water will be drained through cleaning outlet valve. The flushing will be continued for more than 2 hours until pH value of flushing water become same as RO feed.

After flushing, RO system is then put into running however, permeate water must be drained at least one hour since start up.

2.0 RO Membrane Cleaning

1) Cleaning Chemicals

a) S.W. type RO system

Name of Chemicals	Necessary for One Time Cleaning
1. Citric acid (powder)	10 kg
2. Ammonia (25% sol) (For adjusting pH 4)	18 kg
3. Formalin (37% sol)	6.8 kg
4. Water (permeate)	0.5 m^3 (net)

In addition to above chemicals, sodium hexa-meta phosphate will be utilized for removal of scale fouling when necessary.

b) H.F. type RO system

Name of Chemicals	Necessary for One Time Cleaning
1. Citric acid (powder)	10 kg
2. Ammonia (25% sol) (For adjusting pH 4)	18 kg
3. SBS (crystal)	250 g
4. Water (permeate)	0.5 m^3 (net)

In addition to above chemicals, sodium hexa-meta phosphate will be utilized for removal of scale fouling when necessary.

3.0 Membrane Element Replacement

More than 2 gangs are recommended when removing RO membrane elements.

Remove the membrane elements from each pressure tube as follows:

1) S.W. Type RO Membrane Element (Rotary SP-120)

1.1) Removal

a) Disconnect victaulic couplings of feed/concentrate connecting tubes (Fig. 7-1) and permeate connecting tubes at each end of the pressure tube.
The flexible tubing may remain attached to the end plates. Mark or tag all items removed for return to the same location.

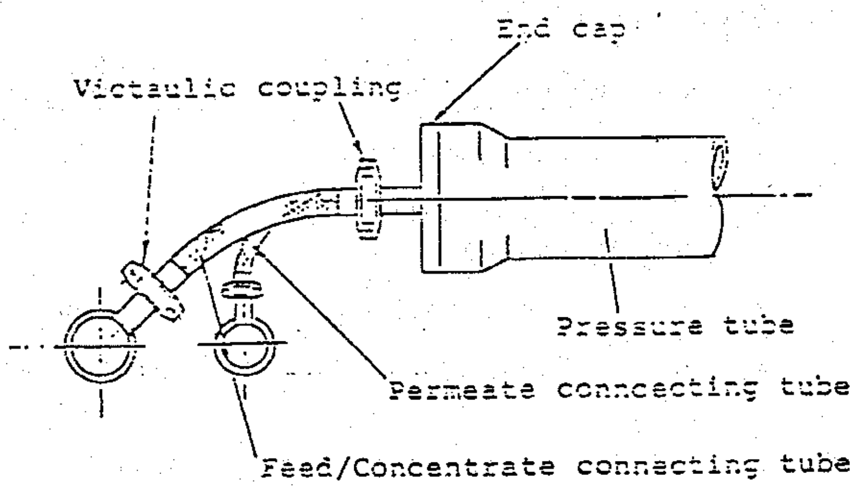


Fig. 7-1

b) Remove clamp nuts and then remove end cap assembly (end plate and end cap) from each end of pressure tube as follows:

i) Remove all clamp nuts from end plate

ii) Carefully withdraw end cap assembly from end of pressure tube. Screw driver is not recommended for jack up. Thin and wider plate are used.

c) Push elements from feed end side with bar like a plastic pipe with one end swab.
The elements should be pushed out one at a time. And each element should be supported at the brine end side until free of the pressure tube as it is being pushed out from the opposite end. (Fig. 7-2)

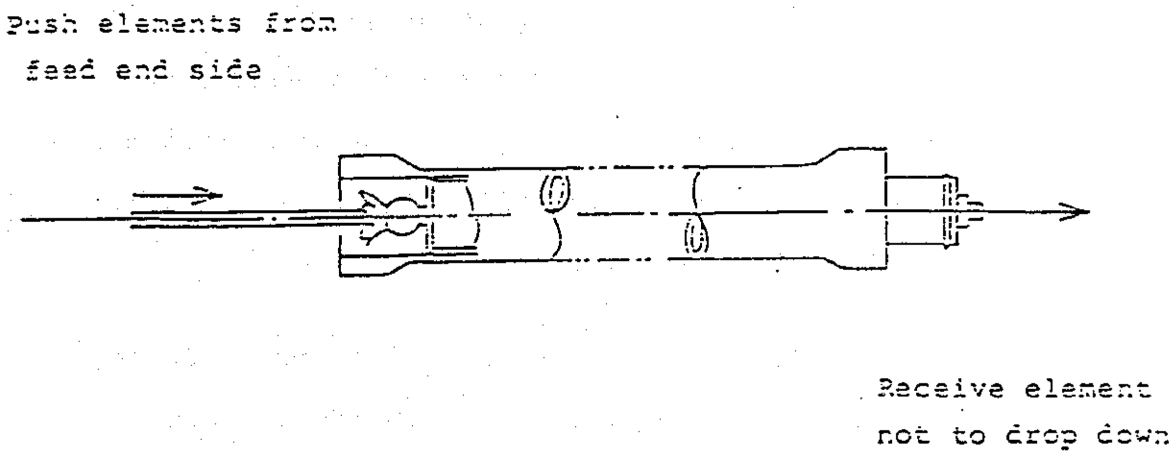


Fig. 7-2 Membrane Removal

d) Each removed element has to be marked as it was loaded. And has to be kept on clean desk or others. The permeate tube end must be protected so that O-ring can seal pressure with tube end surface.

1.2) Loading

a) Remove end cap assembly as stated in item 1.2 in para. 3.0 Membrane elements removal.

b) Spray clean water through the open pressure tubes to remove any dust or other forign matter.

Note: In the event that additional cleaning is needed, improvise a swab large enough to fill the inside diameter of the pressure tube. Soak the swab in a glycerine water solution and force it through the pressure tube until clean.

c) Set O-ring to the all necessary positions Lubricate all O-ring seals with vaseline or glycerine. When setting, do not roll into positions. Expand O-ring slightly to set. Do not pull O-ring over any sharp edges.

d) Ensure all brine seals (U-seals) for membrane elements are properly located and lubricated with vaseline.

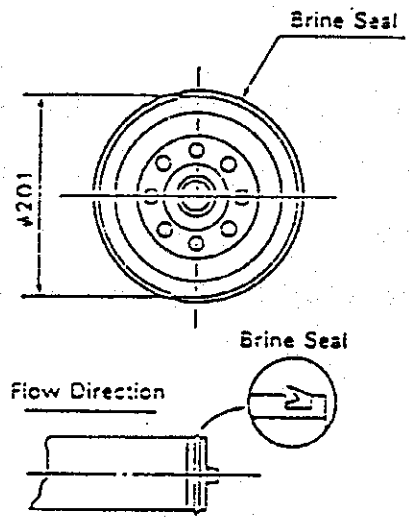


Fig. 7-3

e) Confirm direction of membrane elements loading not to insert reversely. (Insert brine end and push feed end). Set brine end-connector into permeate tube of membrane element on brine side with lubricated O-ring.

f) Place the brine end of the tail end element in the pressure tube and slide it in about three quarters of the element.

g) Set inter-connector with 2 lubricated O-rings into permeate tube of tail end element on feed side.

h) Lift the next RO membrane element into position and insert its brine-end permeate tube into the inter-connector on the tail end elements.
The tail end element in this moment must be fixed not to travel. The two elements (tail end element and next element) must be aligned and levelled each other for correct connection.
Be very careful to hold the next element so that the weight is not supported by the inter-connector.

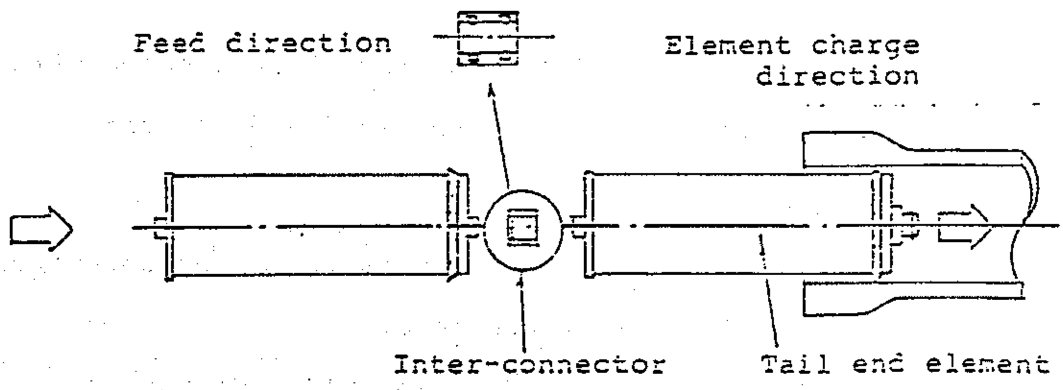


Fig. 7-4 Membrane Loading

i) Push the element into the pressure tube until about one quarter of the second element extends from pressure tube.

j) Repeat steps g), h) and i) until all membrane elements are loaded into the pressure tube.

k) Push again the elements-train to the concentrate side so that tail end element extends approx. 2 to 3 cm from pressure tube brine-end.

l) Set the end cap assembly into the end-connector located on tail end element after lubrication.

m) Push back elements-train with end cap assembly sliding on the guide-stud bolts. And set all clamp nuts with finger-tightening.

Note: Do not over tighten.
Finger tightening plus half turn is the most recommended.

n) Set the feed-end-connector with lubricated O-ring into permeate tube of the lead end element.

Notes:

(1) Feed-end-connector is the blind connector. Not hollow connector.

(2) Set internal O-ring only. Do not set the external O-ring. (important)

(3) Feed-end-connector is also named the feed-end-adopter.

o) Measure the clearance between the shoulder of feed end connector and the inner face of end cap.
Put the adopter spacers to avoid clearance if there is.

p) Set the end cap assembly after lubrication.
Tighten clamp nuts with finger plus half turn.
Do not over tighten.

Note: Set end cap assembly to assure proper alignment with connecting tubes

q) Set the each victaulic couplings properly.

Note: Do not make any clearance between each half of victaulic coupling.
Do not pressurize RO if there is clearance between each half of victaulic coupling.

1.3) Reducing Membrane Numbers in Pressure Tube for S.W. Type RO System

Reduce membrane element numbers in pressure tube as following procedure (when necessary by some reasons).

a) It is necessary to unload all membranes out from pressure tube even when reduce one or two elements.

b) Repeat same procedure as stated 3.0 for membrane replacement.

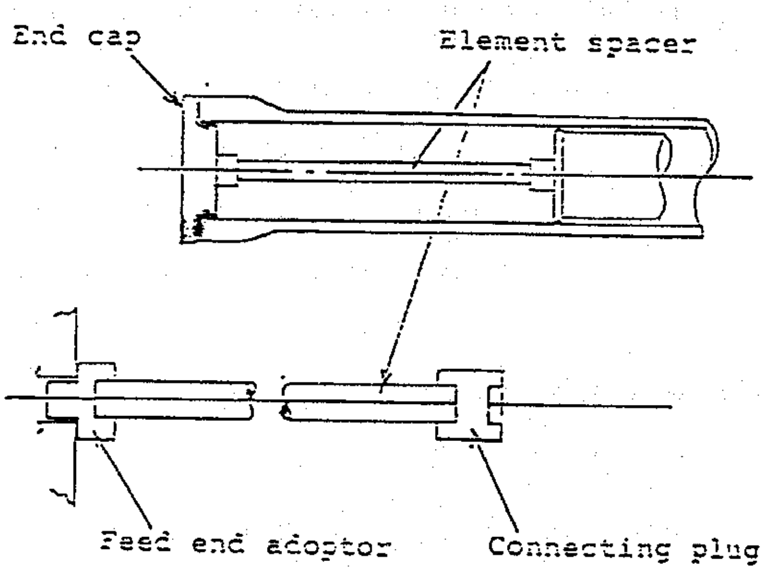
c) Delete membrane element from lead and position which is feed-inlet side.

c) Load elements as stated turn but only for last element (lead end element), place "Element spacer" instead of the last element.

Notes:

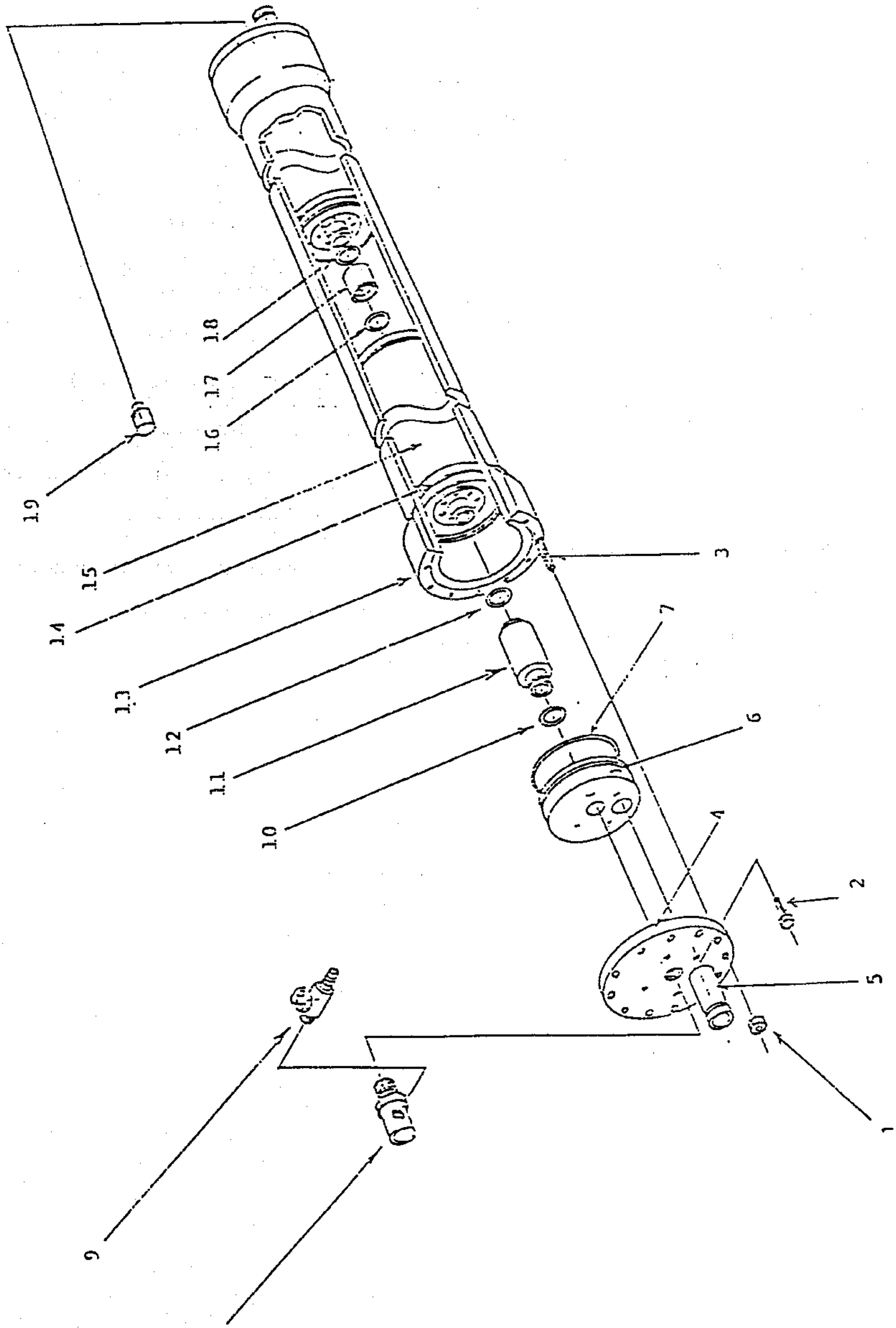
(1) Set the connecting plug (blind inter-connector) instead of inter-connector at feed end of 2nd element.

(2) The same procedure is taken for feed end adopter.



Adoptor, spacer may be necessary to avoid clearance.

Fig. 7-5



1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19

Pressure Tube Assembly

1. Clamp nut
2. End cap fixing bolt
3. Pressure tube stud bolt
4. End plate
5. Feed/connectrate nozzle
6. End cap
7. End cap O-ring
8. Permeate hose connector
9. Permeate sample cock
10. External O-ring for end connector end connector
11. End connector
12. Internal O-ring for end connector
13. Pressure tube
14. Brine seal (U-seal)
15. Membrane element
16. O-ring for interconnector
17. Inter-connector
18. O-ring for interconnector
19. End plate plug

2) H.F. Type RO Membrane Element

(Toyobo HM8255, HR8355)

2.1) Removal

a) End plate A (product outlet side) shall be removed, and then the support plate on tube sheet shall be taken out of the shell.

b) The element shall be push out of the shell up to the point where O-ring #2 appears by feed water pressurized at 3 kg/cm².

c) The element shall be pulled out from the shell by hands. When tube sheet is caught by the stopper ring, the element can be turned in the shell slightly in order to be pulled out easily.

d) End plate B (feed water inlet side) shall be removed as the above manners.

e) Inside wall of the shell shall be washed by a sponge with soap.

2.2) Loading

a) A new element is sealed by laminate films filled with 0.8 wt% formalin and packed in a carton box.

b) Before unsealed, the element shall be investigated if there is no defect on the surface or O-ring of the element.

Then new element shall be pulled out from the films.

c) Insert of new element into the shell

- O-ring #2 shall be covered slightly with silicon grease and it shall be fitted with O-ring slit on tube sheet.
- The element shall be put into the shell by hands with care to kept streight on.
- When the element could not reach smoothly the point illustrated in Figure 1, use an element insertion flange (exclusive jig) for insertion. In this case, be careful not to hit the element at the center.
- Tube sheet would be damaged by colliding with the stopper ring, if the element would be forced to be put into the sheel exceeding the point indicated in Figure 1.

d) A supporting plate shall be attached on the open end of the tube sheet.

e) Assembly of end plate A
After checking O-ring #4 has no defect, end plate A shall be fastened.
The torque must be kept less than 200 kg-cm.

f) Assembly of end plate B
After checking O-ring that O-ring #3 is fitted in a connector and that O-ring #1 has no defect, end plate B shall be set on the shell by using a guide rod in the manner illustrated in Figure 2.

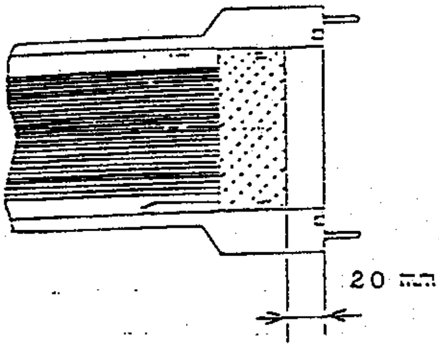


Figure 1

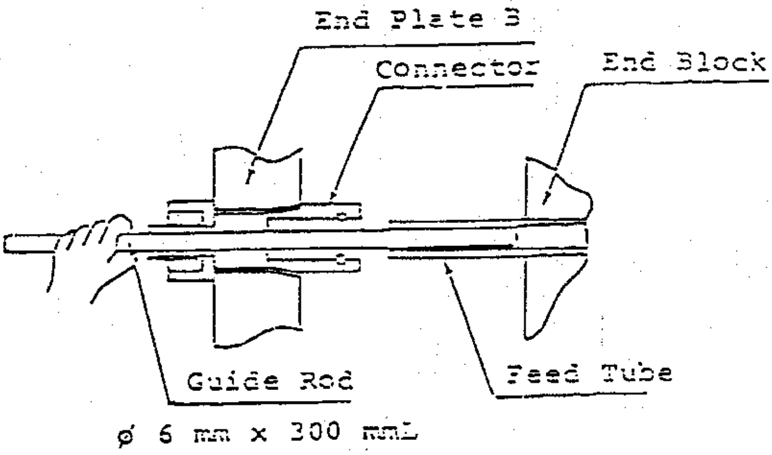
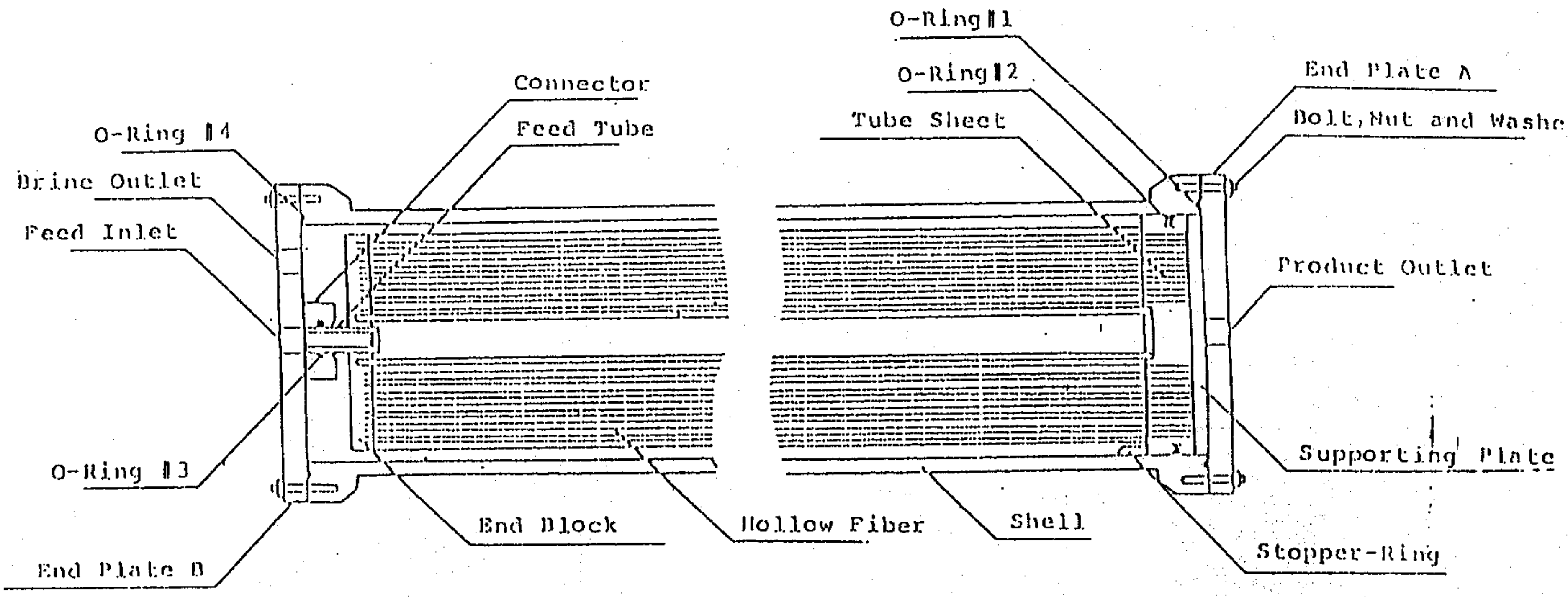
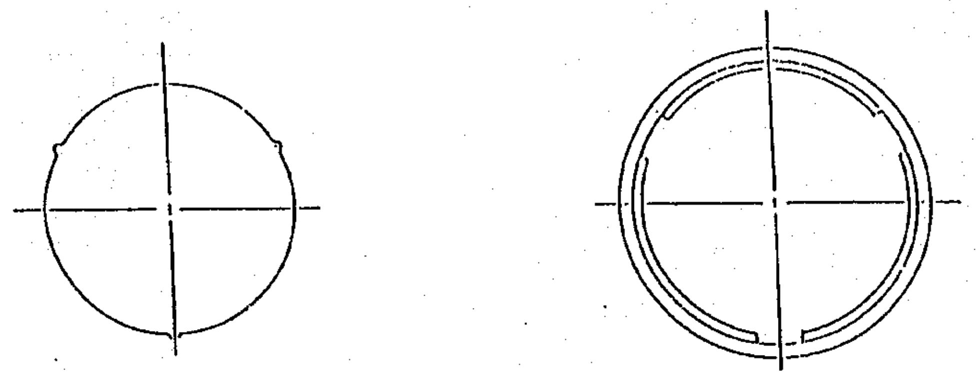


Figure 2

Figure 3 Structure of HOLLOSEP





Cross Section's Outline of End Block

Cross Section of Shell on Stopper-Ring part

CHAPTER V. WHAT IS RO?

1.0 Principle of Reverse Osmosis

1) Introduction

In this section, we will attempt to provide a basic description of the Reverse Osmosis (RO) theory briefly. We hope clients understand the theory logically and utilize the principles reasonably.

2) RO Principles

RO is one of the membrane permeation processes for separating pure water (or other solvent) from a less pure solution. A semipermeable membrane and an appropriate pressure are indispensable, that is, the solution is passed over the surface of an appropriate semipermeable membrane at a pressure in excess of the effective osmotic pressure of the feed solution and only pure solvent permeate through the membrane and then solutes are rejected and concentrated at the feed solution side. Solvent is mostly water but any other liquid is applicable, and solutes are mostly inorganic salts but any other organic salts or organic compounds are to be applied.

To understand how RO works, consider natural osmosis. In Figure 1.a, when fluids of different degrees of concentration are separated by a semipermeable membrane, the water (or solvent) permeates through the membrane from the dilute solution into the concentrated solution. This is known as "Osmosis".

The osmotic flow continues until a state of equilibrium is reached. Once equilibrium is attained, the difference in fluid level becomes equal to what is known as "Osmotic Pressure" of the solution (See Fig. 1.b).

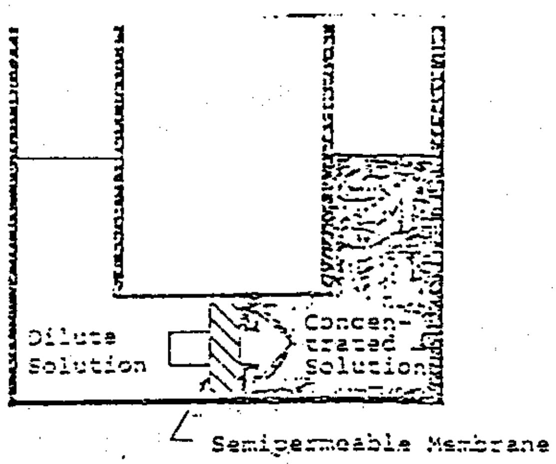


Fig.1a Osmosis

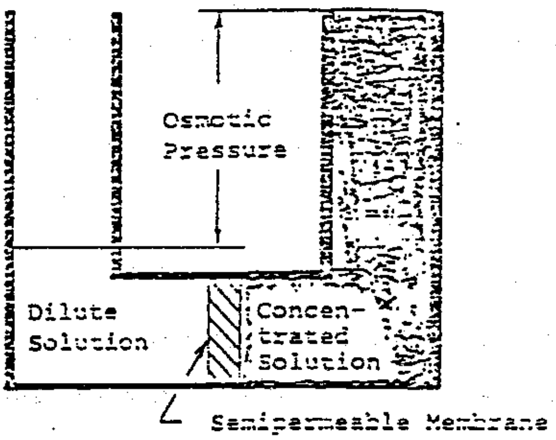


Fig.1b Osmotic pressure

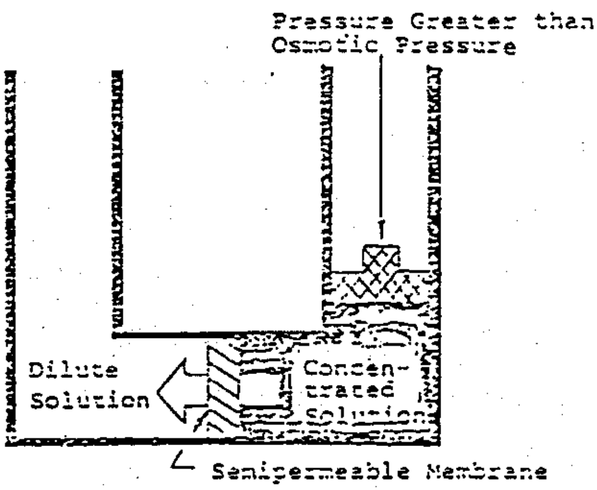


Fig.1c Reverse osmosis

Fig.1 Reverse osmosis principle

The term of "Rejection" is defined by equation 3.

$$\text{Ref.} = \left(1 - \frac{C_P}{C_M}\right) \times 100 \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (3)$$

where,

Rej.: rejection of solute (%)

C_P : solute concentration in permeate (g/ℓ, mol/ℓ)

C_M : solute concentration on membrane surface (g/ℓ, mol/ℓ)

The term "Recovery" is defined by equation 4.

$$R_C = \frac{Q_P}{Q_F} \times 100 \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (4)$$

where,

R_C : recovery or conversion (%)

Q_P : product solution flow rate ($g/cm^2 \cdot sec$)

Q_F : feed solution flow rate ($g/cm^2 \cdot see$)

And the ideal "Osmosis Pressure" is defined by equation 5.

$$\Pi_{id} = R\, T \sum_s C_s \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (5)$$

where,

Π_{id} : ideal osmotic pressure (atm)

R : gas constant ($atm \cdot \ell/mol \cdot {}^\circ K$)

T : absolute temp. of solution (°K)

C_s : concentration of solutes (mol/ℓ)

$\sum_s$: summation

If the osmotic pressure is overcome, as in Fig. 1.c, by the application of an external pressure to the concentrated solution, the water (or solvent) in this solution will permeate into the dilute solution. This is called "Reverse Osmosis".

As mentioned above, RO is a process involving neither phase transition nor chemical reactions. So, RO process has a merit especially in saving energy and can be applied to any water purification process or solutes recovery process requiring the separation of fluids into permeate and concentrate forms.

3) Performance Variables

To a first approximation, two equations define the passage of solvent (water) and solute (dissolved solids) through the semipermeable membrane:

$$Q_{solvent} = A\ (\Delta P - \Delta \Pi) \qquad (1)$$

$$Q_{solvent} = B \cdot \Delta C \qquad (2)$$

where,

Q :	solvent or solute flow through membrane	($g/cm^2 \cdot sec$)
A :	membrane permeability coefficient for solvent	($g/cm^2 \cdot sec \cdot atm$)
B :	membrane permeability coefficient for solute	(cm/sec)
ΔP:	applied pressure differential	(atm)
$\Delta \Pi$:	osmotic pressure differential	(atm)
ΔC:	solute concentration differential	(g/cm^3)

2.0 Characteristics of Membrane

1) Net Effective Pressure

The most significant driving force to the Reverse Osmosis membrane performance is the pressure applied to the membrane among various factors which are giving effects to membrane performance.

Generally, the applied pressure to the membranes are illustrated in following equation:

$$Pa = Pn + Po + \frac{1}{2}\text{ delta-P} + Pb$$

where,

Pa : applied pressure to the membranes and shown as reading of RO inlet pressure gauge (kg/cm^2G)

Pn : net effective pressure which gives performance to the membranes

Po : mean osmotic pressure of concentrated water in the RO membrane system (kg/cm^2G)

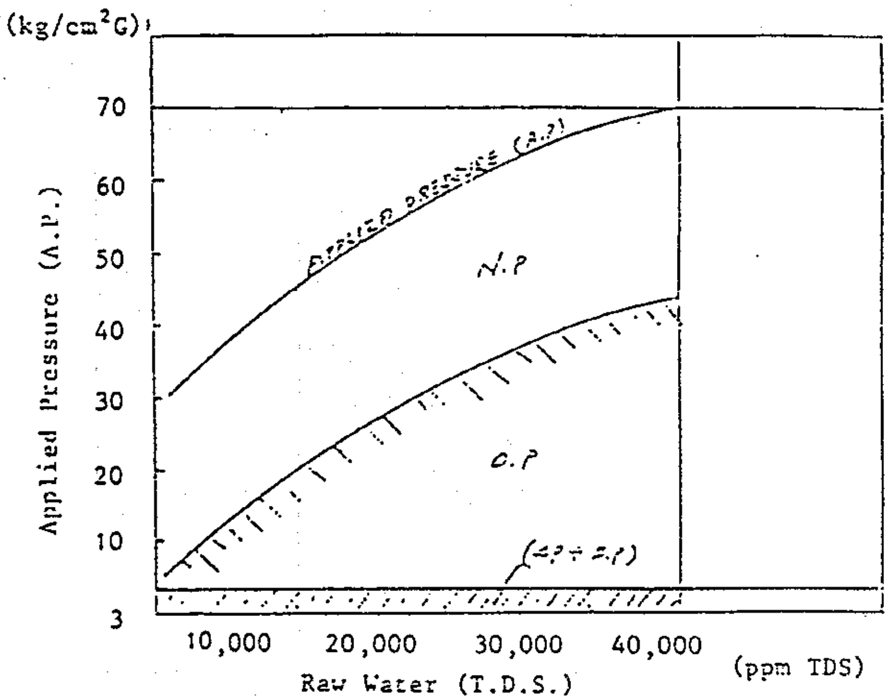
$\frac{1}{2}$ delta-P : 50% of delta-P across membrane system and can be obtained from the pressure difference between applied pressure and outlet pressure of concentrate water (kg/cm^2G)

Pb : the back pressure on the permeate water line which is given by the locations and piping resistance (kg/cm^2G)

As shown in above equation, the applied pressure Pa is the total sum of various component of pressure. And the net pressure Pn is actual driving force to the performance of membrane. Besides, standard performance of the membrane is designed and defined by each membrane manufacturer as of under the standard operating conditions which give certain amount of net effective pressure.

Applied pressure Pa would be constant to give constant performance to membranes when Po and delta-P are constant.

- See Applied Pressure - Raw Water TDS Curve -



Note: Delta-P and back pressure on permeate line are almost constant as they are matters of pretreatment and the plant piping design.

2) Membrane Flux

Membrane flux (permeate flow rate) varies proportionally to net effective pressure under the constant water temperature. And also membrane flux varies with power to water temperature.

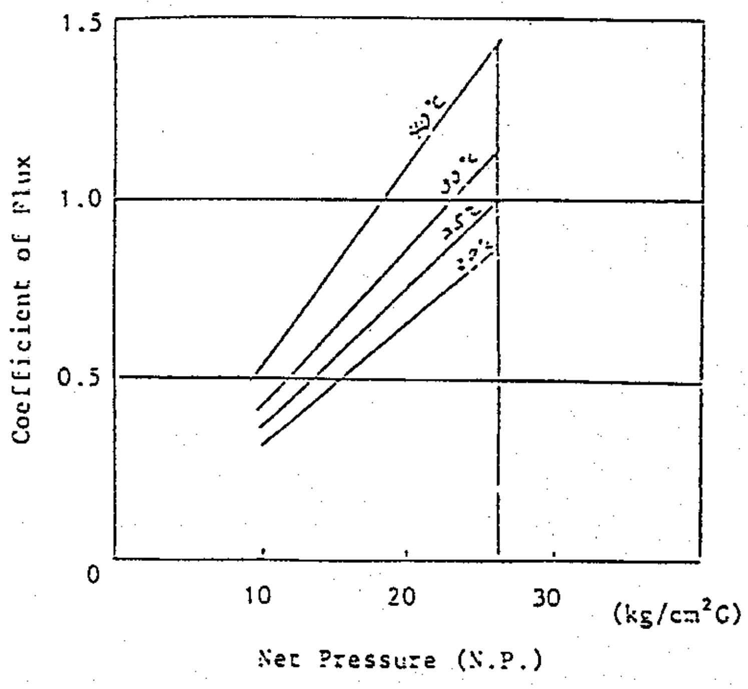
Fough equation:

$$\text{Flux multiplier} = 1{,}028^{(t-25)}$$

where, t: water temperature

Thus, the flux is the function of net effective pressure and water temperature.

- See Coefficient of Flux VS Net Pressure -



3) Salt Rejection

The salt rejection (%) of membrane also varies reverse-proportionally to net effective pressure.

- See Salt Rejection VS Net Pressure -

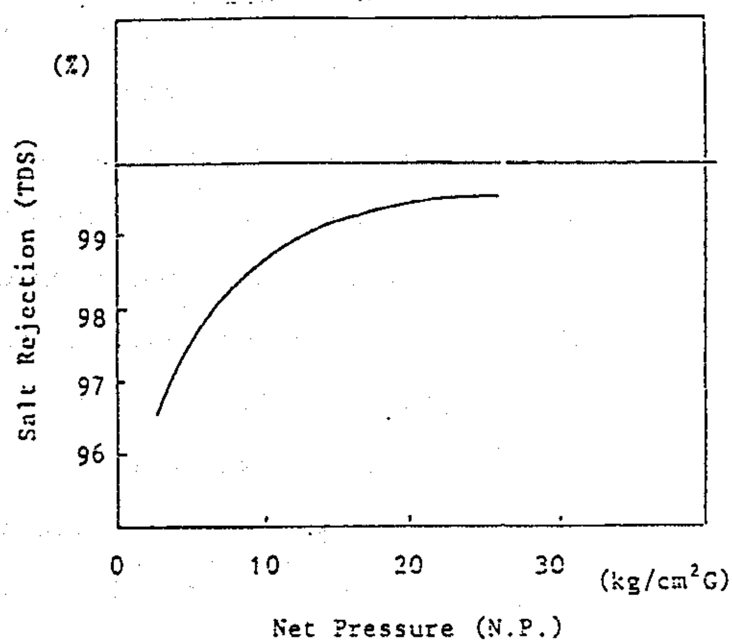


Fig. 3 N.P. - Salt Rejection

4) Water Recovery Rate

The water recovery rate (conversion rate) determines the concentration of water in RO membrane system and give directly effect to permeate water quality (TDS ppm) in the constant salt rejection condition.

For higher TDS feed water, RO system will be operated in a lower recovery rate to keep requested permeate quality (TDS ppm). And for lower TDS feed water, higher recovery rate can be applied within the range of scale precipitation inhibition.

5) Feedwater Treatment Required for Membrane

Pretreatment Considerations

The dissolved salt content of most water sources varies from near zero for precipitation in remote areas to approximately 3.5 percent in seawater. Among the more imporatnt materials dissolved in natural water are oxygen and carbon dioxide from the atmosphere, as well as ions of calcium, magnesium, sodium, iron, manganese, silica, sulfates and chlorides. The equilibrium between carbon dioxide in the atmosphere and bicarbonate and carbonate ions in solution provides a powerful buffering system, so that natural water nearly always fall in a fairly neutral pH range of 5 to 9.

Pretreatment of the feed is necessary in all desalination methods, and reverse osmosis is no exception. In general, feed and brine streams are treated for the following purposes:

i) To remove excess turbidity or suspended solids.

ii) To adjust and control the pH and temperature of the feed.

iii) To inhibit or control the formation of compounds, which when precipitated will plug the water passages or coat the membranes.

iv) To disinfect and prevent slime growths or prevent contamination of the equipment.

v) To remove emulsified and unemulsified oil or similiar organics.

Flux decline with time is the most serious operational problem. It is common to all reverse osmosis design concepts and other membrane processes as well. Flux decline is the sum of membrane compaction and fouling. At low operating pressures (35 kg/cm or less and 30°C feed water temp.), the latter effect usually predominates.

The major pretreatment requirement for any RO device is the prevention of membrane fouling. Fouling, a complex phenomenon, affects all types of membranes and devices, although the degree of severify may vary. Five types of fouling should be considered, (a) membrane scaling, (b) fouling by metal oxides, (c) device plugging, (d) colloidal fouling, and (e) biological fouling. Membrane fouling can sometimes be minimized by periodic cleaning. The proper amount of pretreatment (and cleaning), therefore, is a major decision in reverse osmosis design.

The quality of feed water required by membrane processes is shown below. All membrane configurations, except tubular, have limited capabilities for treatment of feed water containing suspended solids. The smaller the clearance between the adjacent membranes, the tighter the requirements are for turbidity and silting index - plugging factor - of the feed water as noted also, below:

a) Feed water identification

   Fouling index 4.0 or less

b) Allowable SDI (Silt Density Index)

   SDI < 5.0

Each membrane process has established limits on most of the dissolved solids which tend to form insoluble precipitates within the membrane elements. Among these are iron, manganese, strontium, barium and silica. Iron and manganese seem to be of major concern to each membrane process, with maximum levels much lower than the other dissolved solids. Chlorine is used to inhibit organic growths in feed water. The membrane systems, however, have a limited tolerance to residual chlorine. The maximum permissible and minimum suggested residuals are,

0.3 - 1.0 ppm

However, non-cellulose acetate membrane (composite membrane) is strictly inhibited residual chlorine and dissolving oxygen.

The maximum permissible and minimum suggested values are,

Residual chlorine	0 ppm
Dissolving oxygen	Max. 0.5 ppm

The membrane processes recommend (a) addition of 2 to 20 ppm of sodium hexametaphosphate (SHMP) to the feed water to suppress scale precipitation, and (b) adjusting pH to the values 4 to 6 to prevent calcium carbonate formation and/or to prevent hydrolysis of the cellulose acetate membranes. The maximum recommended operating water temperature is 35°C. Short period of higher water temperature is also acceptable unless it is over 40°C. Operation at temperature above these limits results in accelerated deterioration of membrane performance.

3.0 Chemicals Compartibility

This Chapter discusses the chemical compartibility of the membrane.

1) Effect of Low pH

Very low pH adversely affects membrane performance and integrity by hydrolysis. With respect to low pH, the recommendation is,

a. The pH in the membrane element must be 4.0 or more for continuous operation.

b. Membrane elements must not be exposed to pH 2.0 or less for more than 60 minutes at such time of chemical cleaning and/or start up time.

2) Effect of High pH

High pH adversely affects the membranes. With respect to high pH the recommendation is,

a. The pH in the membrane elements must be 6.5 or less for continuous operation.

b. The membrane accept higher pH than 6.5 up to 7.5 for short time but limited by $CaCO_3$ scale precipitation.

c. The membrane must not be exposed to higher pH than 8.0.

3) Long Term Exposure to Following Oxidants

a. The cellulose acetate membranes are degraded by oxydants.
   H_2O_2, KM_3O_4, O_3, Na_2SO_5, CH_3CO_3H, etc.
   Residual chlorine of more than 1 ppm is not recommended for long term operation.

b. The non-cellulose acetate membrane (composite membranes) are strictly inhibited oxidants.

4) Long Term Exposure to the Solvents Containing Water
When RO feed water contains the solvents to membrane, membrane performance will be degraded. The solvents will be numerous kinds such as aromatic substances in the chemical processes and others.
In general, water soluble organic compounds must be individually tested for compartibility.
Waste water from aromatic chemical processes must be avoided for safety.

5) Effect of Scale Inhibitors
Following scale inhibitors are chemically compartible to the membranes:

a. SHMP (Sodium Hexa Metha Phosphate)
b. Sodium polyacrylate
c. Organic polyphosphonate
d. Chlorine max. 1.0 ppm as $C\ell_2$

6) Cleaning Agent

a. Citric acid (But compartible pH range only)

b. EDTA (But compartible pH range only)

c. Ammonia (But compartible pH range only)

d. Formalin (But compartible pH range only)

Max. 5,000 ppm

e. SHMP

f. Triton X-100 (as detergent)

7) Lubricants

a. Vaserine

b. Glycerine

4.0 Spiral Wound Element and Hollow Fiber Element

1) Introduction

Currently more than 4 kinds of commercially available membranes are utilized in Reverse Osmosis application field.

They are,

a. Spiral wound type

b. Hollow find fiber type

c. Tubular type

d. Plate and frame type

Aboves are the classification based on the membrane configurations. The spiral wound type and hollow fine fiber type are mainly utilized for both brackish and seawater desalination. The tubular and the plate and frame type are utilized mainly for waste water reclamation and/or foods-concentration.

a) Comparison of spiral wound type and hollow fine fiber type

The comparison on the membrane configuration is represented by the comparison of S.D.I. (Silt Density Index) value required for RO feed water.

SDI for spiral wound : Max. 5.0

SDI for hollow fiber : Max. 3.0

Obviously spiral wound membrane accept wider range of pretreatment. And hollow fiber membrane requires more severe pretreatment.

b) Membrane materials

Generally membrane materials can be classified into two groups.

i) Cellulose acetate group

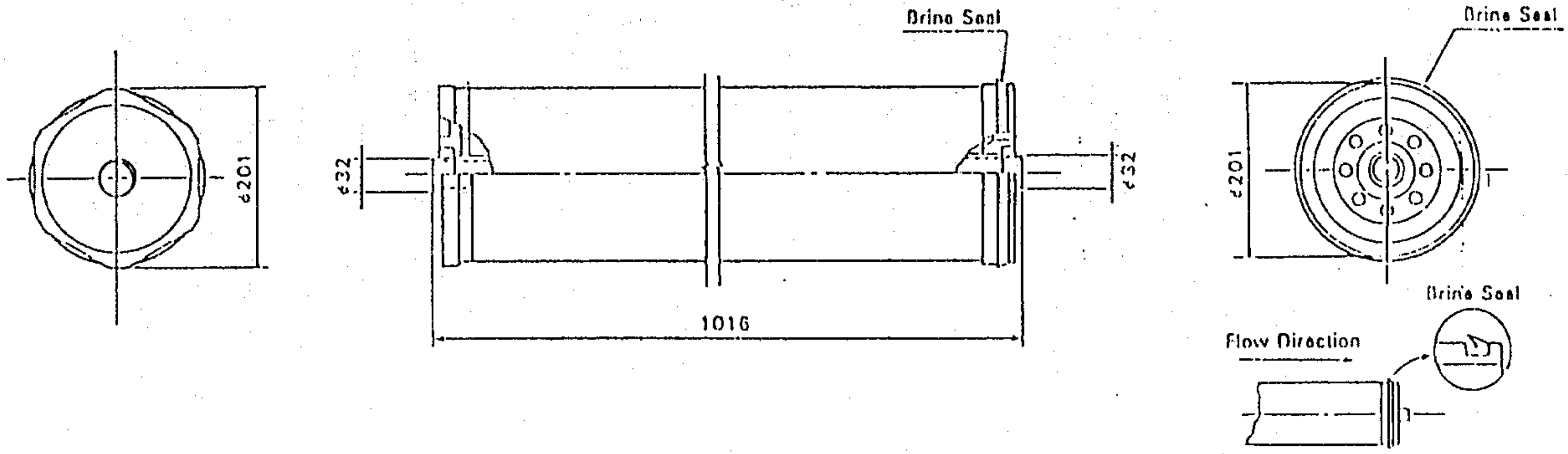
ii) Aromatic polyamide group

Each material has its advantage and disadvantage as follows:

	CA	PA
Allowable pH range	4 ~ 7.5	4 ~ 9
Residual chlorine tolerance	3.0 ~ 1.0 ppm as Cl_2	0.0 ppm as Cl_2
Max. water temp. acceptable	40°C	35°C

Usually, membranes are selected by the plant requirements and circumstances. Such as salt rejection, pretreat, water temperature acceptability, and etc. Then RO system is designed and is operated so as to meet membrane requirement.

TYPE SC-3200 ELEMENT



CHAPTER VI. MEMBRANE DETERIORATION AND REJUVENATIONS

1.0 Membrane Deteriorations

1) Physical Deterioration

When membranes are physically deteriorated by some reasons, membranes gradually lose their salt rejection and increase simultaneously permeate flux.
In this deterioration usually, the load end elements in the first bank pressure tubes start to deteriorate in a short time after commissioning of the RO system. And in the extreme case, the lead end element may completely lose salt rejection in one week time accompanying permeate flux increasing and has no more ability of desalination.
Not only the lead elements in the first bank pressure tubes but also 2nd elements in the same pressure tubes will be deteriorated next, 3rd, 4th, as well.
This happens under certain physical conditions created by high pressure valve throttling, feed/concentrate velocities, high pressure pump selection, delta-P across pump discharge valve and etc. And will proceed very swiftly once it happens.
It is very important accordingly and highly recommended to follow strictly the guide lines to protect deterioration of membranes.

Guide Lines

a. Do not throttle pump discharge valve more than allowable limitation 10 kg/cm^2G delta-P across valve.

b. 60 kg/cm^2G or higher pressure be applied.

c. Delete and add membrane elements so as to meet above conditions a. b.
When necessary, delete lead and elements in first bank pressure tubes and put element spacers for fixing.

d. Do not give higher feed flow rate than max. allowable 3.4 m^3/Hr to the individual pressure tubes.
Above guide lines should be followed unless otherwise indicated.
High pressure pipes and valves are sized and selected so as to meet specified flow rate and pressure. The big percentage of nominal capacity change (capacity decreasing) are not recommended.

2) Biological Deterioration

When membranes are biologically deteriorated, membranes gradually lose their salt rejection and increase simultaneously permeate flux.
This biological deterioration may happens either in running RO or in long term - shut down RO when conditions are met.

Membranes exposed to biological attack lose salt rejection in a short time and increase permeate flux. And generally deteriorations happen at random and difficult to find out certain relation between location of membrane and deterioration.
It is very important accordingly and highly recommended to follow strictly the guide lines to protect deteriorations of membranes.

<u>Guide Lines</u>

- For Running RO -

a. Keep chlorine residual 0.3 to 1.0 ppm all the time. (This will be the perfect remedy for operating RO.)

b. Lower RO feed pH in the case that residual chlorine cannot be kept by some reasons.
(This is temporary remedy only.)

c. Minimum two times RO cleaning annually adding formalin solution.

- For Shut down RO -

a. Soak membranes in 0.5% formalin solution pH controlled 4.0 to 4.5.
(For long term shut down)

b. Replace soaking solution of 0.5% formalin pH controlled 4.0 to 4.5 monthly. (For long term shut down)

The monthly replacement of formalin solution must be strictly followed.

c. It is not necessary to remove membrane elements during long term shut down unless ambient temperature goes up over acceptable limit.

d. Shut down less than 2 weeks doesn't require formalin solution treatment but weekly or more frequent flushing using conditioned water is necessary. This must also be strictly followed.

3) Chemical Deterioration

Chemical deteriorations including hydrolysis of membranes happen in following cases:

a. Long term operation on unusual RO feed pH

The recommended feed pH range is 4 to 6 (6.5). And even in allowable pH range for membrane 3.0 to 4.0 and 6.5 to 8.5, hydrolysis may take place in long term operation. For higher pH operation, not only hydrolysis but also $CaCO_3$ precipitation may take place.

As summary, pH control on RO feed must be strictly followed.

b. Long term exposure to following oxidants

The cellulose acetate membranes are degraded by oxidants.

H_2O_2, KM_3O_4, O_3, Na_2SO_5, CH_3CO_3H, etc.
Residual chlorine of more than 1 ppm is not recommended for long term operation.

c. Long term exposure to the solvent containing water
When RO feed water contains the solvent to cellulose acetate membrane, membrane performance will be degraded.
The solvents to cellulose acetate will be numerous kinds such as aromatic substances in the chemical processes and others.
In general, water soluble organic compounds must be individually tested for compartibility.
Waste water from aromatic chemical processes must be avoided for safety.
When chemical deterioration happens, membranes gradually lose salt rejection and increase simultaneously permeate flux. And deterioration takes place almost all membranes exposed to the same kind of water.

4) Fouling/Scale Precipitation
When membranes are accumulating fouling matters and/or scale precipitations on their surface, the performance of the membrane will be degraded.
The degradation will appear on salt rejection and flux. That is, flux decrease and salt rejection decrease. And in extreme case, delta-P across membrane will be increased.

a. Fouling

Fouling is not deterioration of membrane and it is possible to restore membrane performance by cleaning. The most popular fouling matters will be ferric iron and/or organic matters. As described above, salt rejection goes down accompanying flux decreasing. This phenomena can be foreseen by routine millipore check and others.

To avoid fouling, following guide lines must be followed:

<u>Guide Lines</u>

i. Always ensure pretreatment operation and keep millipore check colour grade less than 10 and less than 5 minutes filtration time.

ii. If fouling happens or going to happen, don't wait delta-P across membranes increase.
Chemical cleaning should be done to restore salt rejection and flux. And correct pretreatment.

b. Scale precipitation

It is important and be underlined that it is impossible to remove scale precipitation once it is precipitate and deposit on membrane surface. And scale deposit will grow up once it is formed.

No chemical cleaning, no flushing will help to remove except $CaCO_3$ scale.

$CaCO_3$ (calcium carbonate) scale will be formed under high pH operation. And can be removed by acid cleaning and/or low pH operation.

Other precipitations such as $CaSO_4$, $BaSO_4$, SiO_2, etc. cannot be removed completely by any chemical cleaning.

Perhaps membrane elements must be disposed and replaced once scale precipitation and deposition take place on membrane surface, no way to restore.

Following guide lines therefore strictly followed:

<u>Guide Lines</u>

i. Ensure dosing of scale inhibitor in accordance with instruction.

ii. Make flushing using conditioned feed water at every shut down to displace brine water from RO system.

iii. Do not increase water recovery rate than specified unless otherwise indicated.

iv. Lower pH operation necessary for silica saturation water.

2.0 Rejuvenation

It is possible to restore performance which was deteriorated during operation or shut down unless it was degraded due to scale precipitation.

1) RRC

Rejuvenating chemical RRC will be used for this purpose after correcting source of deteriorations.

The RRC is the chemical, food additive grade and is dosed to RO feed line by chemical feeder while RO is in running.

Then performance of membranes will be restored in a short time. RRC is so called "Hole plugging rejuvenation" and will be effective to the deterioration of membranes which have decreased salt rejection and increased flux.

2) Method of RRC Dosing and Rejuvenation

Dissolve RRC 10% or less in filtered water (not in permeate water, usually in neutral pH water) and dose to running RO system using chemical feeder.

The injection point will be up stream of micron filter.

Dosing rate to RO feed is in the wide range of 1 ppm - 100 ppm.

Watching to permeate conductivity, flux and pressure is necessary during dosing. And stop dosing immediately when target performance are restored.

Generally, flux will gradually decrease, permeate conductivity will also be gradually decreased and operating pressure will increase by this RRC dosing.

3) Post Treatment for RRC Dosing

It is standard procedure to lower pH 4.0 - 3.0 at RO feed for stabilization of RRC.

The permeate flux will be decreased extremely by this 4.0 - 3.0 feed pH.

Perhaps flux will drop down to 20 to 30% of designed flux in this moment. Keep lower pH for 2 to 3 Hrs. to stabilize RRC.

Adjust RO feed pH to normally operating value 6.0 to 5.8 after stabilization. Permeate flux will come up to normal value.

4) pH Sensitivity

RRC treated RO membranes are extremely pH sensitive.

In 5.5 or less pH, flux decreases extremely (to 20 - 30% of original value). Usually 6.0 pH is the most adequate operating pH.

An automatic pH control loop must not be used when RRC is applied to membranes. Manual set pH control only.

5) Storage

RRC solution will be stored in cool and dark place.

And the life will be more than 2 years unless container is opened and exposed to air.

CHAPTER VII. LOG TAKING AND COMMUNICATION

1.0 Necessary Communications

It is essential to communicate each other; plant user and supplier, or plant user, local agent and plant supplier.
It is strongly recommended for the plant user to forward operation log sheet at least monthly filling up necessary items and in turn, plant supplier give comments of the users report.
In the case that local agent service net work is available, the communications will be done through local agent or firm.

The followings are normal communications summarized:

1) Operation log sheet forwarding monthly (through agent)
2) Comments on operation from supplier (through agent) Telex or letter whichever
3) Telephone/telex communication when urgent on any problem happend.

Above communications is necessary from start up time for long life of the plant.

2.0 Minimum Log Taking

1) Log Sheet

The operating log sheet describes the performance of the RO system. The operating data must be routinely filled up in log sheet during the life of the plant.

They must include:

- Flows (product and brine)
- Pressures (high pressure pump suction, discharge, RO inlet, and brine outlet)
- Water temperature (RO feed)
- pH (RO feed)
- Conductivity (RO feed, permeate and brine)
- Chemical consumptions (chlorine, scale inhibitor, acid SBS)
- Milli-pore check (colour grade, filtration time)
- Hours of operation
- Unusual incidents (up sets in pretreatment, shut down, pH, pressure, etc.)
- Permeate check

2) Recommended Frequency of Data Collection

The minimum figures are as follows:

	Per Shift	Daily	Monthly
a. Flows	2 times		
b. Pressures	2 times		
c. Water temperature	2 times		
d. Conductivity & pH (panel)	2 times		
e. Residual $C\ell_2$ (RO feed)	2 times		
f. Milli-pore-check		1 time	
g. Running hrs		1 time	

	Per Shift	Daily	Monthly
h. Conductivity (potable)		1 time	
i. pH (potable)		1 time	
j. Salt rejection		1 time	
k. Chemical consumption		1 time	
l. Permeate check			2 times
m. Water analysis (raw permeate)			1 time
n. Unusual incident - on occurrence			

3) Maintenance Log

a. Start up record

Record keeping must cover all supplied equipment such as lubricant changed, brand name, volume, bearing temperature, etc.

For RO membranes, it is recommended to record serial numbers of individual membrane elements charged to pressure tubes. Locations of elements, tag No. of pressure tube and serial number of elements must be clearly recorded for future maintenance.

b. Maintenance log

Maintenance record must be kept on the plant.

The logs include

- Routine maintenance
- Mechanical failure
- Membrane replacement
  Serial numbers, locations, etc.

- Cleanings (chemicals and conditions)
- Long term shut down (conditions)
- Frequency of micron filter backwash

付属資料 8.1.1-1

逆浸透法によるトリハロメタンの除去

逆浸透法による海水淡水化実験（１）[2]

１．はじめに

逆浸透法（ＲＯ）海水淡水化では、生物障害の防止を目的として、取水部において海水に塩素を注入している。しかし海水中には臭素が含まれており、また微量の有機物もあるため、塩素の添加によりブロモホルムを主としたトリハロメタン（ＴＨＭ）が生成する。そこで、酢酸セルロース系とポリアミド系の２種類の海水淡水化用逆浸透膜モジュールを用いて、実海水での淡水化実験を行い、ＴＨＭの挙動を調査した。また、トリハロメタンを除去できない逆浸透膜を使用する場合について、塩素代替滅菌剤としてクロラミン処理の検討も行った。

２．実験方法

(1)実験装置の処理フロー

テストプラントのフローシートを図１に示す。海水は水深3ｍより取水した。塩素処理では取水点で次亜塩素酸ソーダを添加し、遊離残留塩素を0.3mg/ℓとした。クロラミン処理では、原水槽出口で塩化アンモニウムと次亜塩素酸ソーダを添加し、クロラミンを生成した。凝集剤としては塩化第２鉄を添加し砂ろ過を行った。ＲＯ処理では、ポリアミド膜はカートリッジフィルターの前で重亜硫酸ソーダを添加し残留塩素を除去した。ＲＯでの透過水の回収率は40％、操作圧力は酢酸セルロース膜では54～57kgf/cm²、ポリアミド膜では57～59kgf/cm²とした。水温は23～32℃であった。

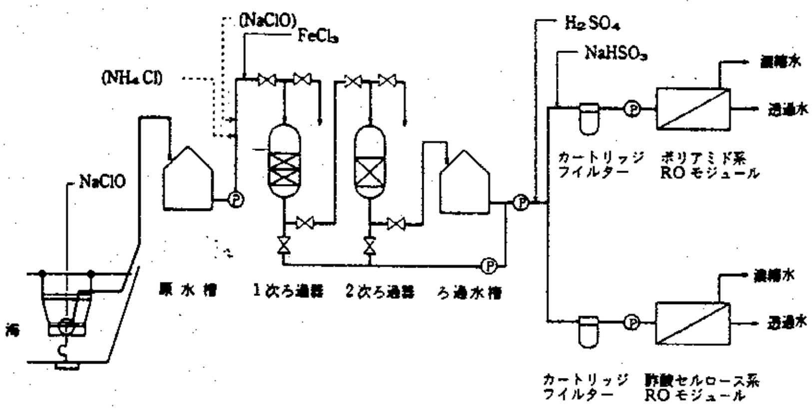


図１　フローシート

(2)ＲＯモジュール

実験では酢酸セルロース系とポリアミド系の２種類のＲＯ膜を使用した。酢酸セルロース膜は中空糸タイプであり、１エレメントで10m³/dの透過水が得られる。塩素を添加する事により常に0.2～1.0mg/ℓの残留塩素を保持することで、エレメント内を殺菌できるが、逆に残留塩素がないとバクテリアにより膜が劣化する可能性がある。ポリアミド膜はスパイラルタイプであり、１エレメントで4m³/dの透過水を得られる。この膜は酸化剤により劣化するため、供給水は残留塩素をゼロにする必要がある。本実験ではエレメント３本を使用し、回収率40％において9m³/dの透過水を得ることを目標とした。

(3)水質分析

トリハロメタンの分析は、ガスクロマトグラフを使用し、溶媒抽出法により分析した。

３．実験結果と考察

3-1.脱塩性能

水質分析結果の一例を表１に示す。海水の水質は日本沿岸の海水としては一般的な水質である。ＲＯ供給水（ろ過水）のＳＤＩ値は常に３以下であり、２種類のＲＯエレメントの供給水条件を満足していた。酢酸セルロース膜は脱塩率が非常に高く、回収率40％において脱塩率は99.7％であった。一方、ポリアミド膜では99.5％であり、酢酸セルロース膜に比較すると低かったが、透過水の蒸発残留物は200mg/ℓ程度であり、水道水水質基準を満足している。また、ポリアミド膜は酢酸セルロース膜に比較して、硫酸イオン、カルシウム、マグ

ネシウムなどの2価イオンの除去率が高かった。これは膜面の荷電の相違によるのもと考えられる。なお、ポリアミド膜は供給水中に微量の重金属が含まれる場合には、急激に脱塩率の低下を起こすことがあり、十分な運転管理が必要である。

3-2. THMの除去

(1)塩素処理

塩素処理でのTHMの分析結果を表2、3に示す。塩素添加後の滞留時間2～4時間では、RO供給水のTHMは15～25μg/ℓとなりそのほとんどがブロモホルムであった。

RO処理では、膜によりTHMの除去性能は大きく異なった。酢酸セルロース膜では、透過水のTHMは供給水の1.2～1.5倍になっておりRO膜で除去されず、逆に透過水側の濃度が高くなった。これはTHMが酢酸セルロース膜との親和性が強く、水よりも透過性が高かったためと考えられる。一方ポリアミド膜では、透過水のTHMは供給水の約10～20%に低下しており、RO膜により約80～90%が除去された。なお、ポリアミド膜の濃縮水に除去されたTHMが含有されており、THMはポリアミド膜に吸着されているのではなく、ポリアミド膜により分離されることを示している。

(2)クロラミン処理

クロラミン処理でのTHMの分析結果を表4に示す。クロラミン処理ではTHMはほとんど生成せず、供給水では1μg/ℓ未満であったが、酢酸セルロース膜の透過水、ポリアミド膜の濃縮水では、1μg/ℓ程度存在することがあった。

(3)トリハロメタン生成能(THMFP)

THMFPの分析結果を表2、3に合わせて示す。THMFPは海水では37～55μg/ℓ、RO供給水では33～42μg/ℓであった。これに対して酢酸セルロース膜透過水のTHMFPは22～33μg/ℓであり、供給水より約20～40%低下していた。これは生成したTHMは酢酸セルロース膜の透過側で濃度が高くなるが、THMFPの原因となる前駆物質については膜で除去されるためと考えられる。なおTHMの水質基準値は100μg/ℓ以下であり、酢酸セルロース膜透過水のTHMFPはこの値の1/4～1/3であった。

表1　水質分析結果

項目	単位	海水	RO供給水	RO透過水 CA膜(*1)	RO透過水 PA膜(*2)
pH	−	8.2	6.6	4.9	5.8
電気伝導率	μS/cm(25℃)	50400	50400	170	374
蒸発残留物	mg/l	35600	35600	88	184
SDI値	−	>6	1.6		
残留塩素	mg/l		(*3)0.3	0.3	0
総アルカリ度	mg/l as$CaCO_3$	115	62.4		
塩素イオン	mg/l	18700	18700	46.1	106
硫酸イオン	mg/l	2530	2560	2.9	1.8
臭素イオン	mg/l	69			
カルシウム硬度	mg/l as$CaCO_3$	980	980	3.5	2.0
マグネシウム硬度	mg/l as$CaCO_3$	5460	5460	6.9	4.4
鉄	mg/l	0.10	<0.02		
COD	mg/l	2.1	0.5		

(*1):酢酸セルロース膜　(*2):ポリアミド膜　(*3):ポリアミド膜では0mg/l

表2　トリハロメタン(存在量)とトリハロメタン生成能

運転時間 (hr)	トリハロメタン(μg/l) 上段:存在量　下段(カッコ内):生成能 海水	供給水	酢酸セルロース膜 透過水	酢酸セルロース膜 濃縮水	ポリアミド膜 透過水	ポリアミド膜 濃縮水
140		25.0	29.0	18.0	2.7	39.0
	(54.5)	(41.8)	(32.5)	(48.2)	(2.7)	(63.7)
500		15.0	22.0	14.0	2.0	24.0
	(43.2)	(39.0)	(23.3)	(42.0)	(4.8)	(49.2)
1150		16.0	22.0	13.0	3.0	24.0
	(37.0)	(33.0)	(22.0)	(40.0)	(3.2)	(37.0)
2260		21.5	30.0	18.0	2.6	27.0
	(61.5)	(38.6)	(32.4)	(56.7)	(4.8)	(36.1)

表3　トリハロメタン(存在量、生成能)の内容

	トリハロメタン(μg/l) 供給水	CA膜 透過水	PA膜 透過水	トリハロメタン生成能(μg/l) 海水	供給水	CA膜 透過水	PA膜 透過水
クロロホルム	<1.0	<1.0	<1.0	12.0	<1.0	<1.0	<1.0
ブロモジクロロメタン	<1.0	<1.0	<1.0	<1.0	<1.0	1.0	<1.0
ジブロモクロロメタン	<1.0	<1.0	<1.0	1.5	1.8	2.5	<1.0
ブロモホルム	25.0	29.0	2.7	41.0	40.0	29.0	2.7
総トリハロメタン	25	29	3	55	42	33	3

CA膜:酢酸セルロース膜　PA膜:ポリアミド膜

表4　クロラミン処理でのトリハロメタン

運転時間	トリハロメタン(μg/l) 供給水	酢酸セルロース膜 透過水	酢酸セルロース膜 濃縮水	ポリアミド膜 透過水	ポリアミド膜 濃縮水
80	<1.0	1.0	<1.0	<1.0	1.1
270	<1.0	<1.0	<1.0	<1.0	1.0
580	<1.0	<1.0	<1.0	<1.0	<1.0

2）小島，日下部，畠山：逆浸透法による海水淡水化実験（1）
第45回全国水道研究発表会要旨集　平成6年5月　p128

付属資料 8.1.2-1

蛍光光度法による油分測定マニュアル

蛍光光度法による油分測定マニュアル

I. 本マニュアルの背景及び目的

油分測定法について各種検討を行なった結果、蛍光光度法は、数10 ppb オーダーの低濃度領域まで精度良く測定できることがあきらかになった。今期の実験は非常に低濃度の油分を測定することが必要であるため、油分の測定に蛍光光度法を適用する。

本マニュアルは蛍光光度法を用いて低濃度油分の測定を行なうための手順及び機器の操作、メインテナンスを実験担当者が行なえることを目的としたものである。

II. 操作方法

A. 蛍光光度計使用法

1. 用意するもの

- 蛍光光度計（島津RF-1501）
- 蛍光測定用セル
- ビーカー（100 ml）
- メスフラスコ（50 ml）
- ピペット
- 精密天秤
- イソオクタン（蛍光分析用グレード）
- A重油又はその相当品

2. 手順

2.1 標準試料の作成

1) A重油約 0.5 g を 100 ml ビーカーに正確に量りとり、これをイソオクタン約 40 ml にて溶解した後 50 ml メスフラスコに定容する。これは約 10000 ppm の濃度となる。
2) 上記試料をイソオクタンにて 50 ml メスフラスコを用いて順次希釈し、10 ppb~100 ppm 程度までの各種濃度の試料を作成する。

2.2 蛍光分光光度計の操作

（注意事項）

- 電源コンセントを差し込む前に、コンセントの電圧と装置の設定電圧が一致していることを必ず確認する。

本機の操作はメニュー方式になっており、画面に表示されている項目の中から必要な項目を選んで、その番号を入力すると対話が実行される。

対話を途中で中断するときや、画面を一つ前に戻すときは[Return]キーを使用する。

2.2.1 装置の立ちあげ

1) 電源スイッチをONにする。液晶ディスプレイが「初期設定」画面、次いで「メニュー」画面になる。

2.2.2 スペクトル測定

(1) 測定条件の設定

1) 「メニュー」画面のとき、[1]キー（波長スキャン）を押す。

「波長スキャン」と表示された「スペクトル測定の測定条件設定」画面が表示される。

2) スキャンする側の波長（蛍光又は励起波長）を決定し、もう一方の波長を固定値として入力する。

・固定値として入力する側の波長を[EX λ GOTO]キーまたは[EM λ GOTO]キーを押して選択し、次いで波長を数値キーで入力し、[ENTER]キーを押す。このとき入力する数値は文献データなどを参考にして、とりあえずそれを入力してみる。

3) 励起又は蛍光側スキャン範囲を設定する。両者とも設定値は220~900 の範囲とする。

励起側スキャン範囲（nm）を設定する場合

・[2]キー（励起側スキャン範囲）を押す。

・スキャン開始波長を数値キーで入力し、[ENTER]キーを押す。次に同様にして、スキャン終了波長 を数値キーで入力し、[ENTER]キーを押す。

蛍光側スキャン範囲（nm）を設定する場合

・[3]キー（蛍光側スキャン範囲）を押す。

・スキャン開始波長を数値キーで入力し、[ENTER]キーを押す。次に同様にして、スキャン終了波長 を数値キーで入力し、[ENTER]キーを押す。

(2) 装置条件の設定

1) 「波長スキャン」と表示された`「スペクトル測定の測定条件設定」画面のとき、[F4]キー（装置の条件）を押す。

「装置の条件」と表示された「スペクトル測定の装置条件設定」画面が現われる。

2) 励起側分光器スリット幅 を設定する。

[1]キー（励起側バンド幅）を押すたびに10 nmと20 nmが切り替わる。

3) 蛍光側分光器スリット幅 を設定する。

[2]キー（蛍光側バンド幅）を押すたびに10 nmと20 nmが切り替わる。

4) 光信号の変化に対する装置の追従性を設定する。

[3]キー（レスポンス）を押す。各種選択肢が現われ、これらのなかから適当なものを選択する。

5) 光電管への印加する電圧を設定する。

[4]キー（感度）を押すたびごとにHighとLowが切り替わる。

6) スペクトルの表示形式を設定する。

[5]キー（表示形式）を押すたびごとに更新書きと重ね書きが切り替わる。

7)シャッターの開閉を設定する。

[6] キー（シャッター制御）を押すたびごとにONとOFFが切り替わる。
・ONのときは通常シャッターは閉じており、測定時のみシャッターが開く。これにより試料の光化学変化を防止できる。
・OFFのときは自動的にシャッターの開閉はできない。[SHUTTER] キーを押して開閉する。

(3) スペクトル測定の実行

1) 「波長スキャン」と表示された「スペクトル測定の測定条件設定」画面のとき、[F3] キー（測定画面）を押す。
「スペクトルの測定」画面が現われる。
2) 試料をセルにいれ、ホルダーにセットする
3) [START/STOP] キーを押す。
測定が開始され、画面にスペクトルが表示される。
4) ピーク位置の波長を決定する。
・カーソルキー（[<] または [>]）を押し、カーソルをピーク位置にあわせる。
・画面上部に表示される波長と蛍光強度を読み取り、ピーク位置周辺で蛍光強度が一番大きくなる波長を読み取る。

以上の操作にて片方のピーク波長が決定されたらその波長を固定波長としてもう一方の波長をスキャンし、同様の方法にて、もう一方の側のピーク波長を決定する。

以上の操作にて最適な励起、蛍光波長が決定される。

2.2.3 検量線の作成

(1) 測定条件の設定

1) メニュー画面のとき、[2] キー（定量測定）を押す。
「定量測定の測定条件設定」画面が表示される。
2) [1] キー（標準試料数）を押した後、標準試料数（1~10）を数値キーにて入力し、[ENTER] キーを押す。
3) 励起波長を設定する。
・[2] キー（励起波長）を押す。
（[EX λ GOTO] キーを押してもよい。）
・励起波長を数値キーで入力し、[ENTER] キーを押す。設定値は0または220~900の範囲とする。
4) 蛍光波長を設定する。
・[3] キー（蛍光波長）を押す。（[EM λ GOTO] キーを押してもよい。）
・蛍光波長を数値キーで入力し、[ENTER] キーを押す。設定値は0または220~900の範囲とする。
5) 濃度の単位を設定する。
・[4] キー（単位）を押す。

・用いる単位についている番号を画面から選択し、数値キーで入力した後、[ENTER]キーを押す。

(2) 装置条件の設定

1)「定量測定」と表示された「定量測定の測定条件設定」画面のとき、[6]キー（装置の条件）を押す。

「装置の条件」と表示された「定量測定の装置条件設定」画面が現われる。

2) 励起側分光器バンド幅を設定する。

[1]キー（励起側バンド幅）を押すたびに10 nmと20 nmが切り替わる。

3) 蛍光側分光器バンド幅を設定する。

[2]キー（蛍光側バンド幅）を押すたびに10 nmと20 nmが切り替わる。

4) 光信号の変化に対する装置の追従性を設定する。

[3]キー（レスポンス）を押す。各種選択肢が現われ、これらのなかから適当なものを選択する。

5) 光電管へ印加する電圧を設定する。

[4]キー（感度）を押すたびごとにHIGHとLOWが切り替わる。

6)シャッターの開閉を設定する。

[5]キー（シャッター制御）を押すたびごとにONとOFFが切り替わる。

・ONのときは通常シャッターは閉じており、測定時のみシャッターが開く。これにより試料の光化学変化を防止できる。

・OFFのときは自動的にシャッターの開閉はできない。[SHUTTER]キーを押して開閉する。

(3) 検量線の作成

1) メニュー画面のとき、[2]キー（定量測定）を押す。

「定量測定」と表示された「定量測定の測定条件設定」画面が表示される。

2) [1]キー（試料数）を押す。

3) 試料数を数値キーにて入力し、[ENTER]キーを押す。

4) [F2]キー（検量線）を押す。

「多点検量線」と表示された「標準試料テーブル」画面が現われる。

5) 濃度値を数値キーにて入力し、[ENTER]キーを押す。

6) 蛍光強度 (FI)の入力方法を数値キーにて選択する。選択は入力する方法の示されている数値キーを押す。

(1: キー入力, 2 : 測定入力)

<u>キー入力によるとき</u>

・蛍光強度を数値キーにて入力し、[ENTER]キーを押す。

この操作を設定した試料数だけ繰り返す。

<u>測定入力によるとき</u>

・ 標準試料をセットし、[START/STOP]キーを押す。測定が終了すると、蛍光強度が

表示される。

この操作を試料を変え、入力した試料数だけ繰り返す。

7) [F1] キー（検量線）を押す。

画面に検量線のグラフが表示される。また、画面左側には検量線の近似式が表示される。

(4) 検量線／測定条件の保存

1) 「定量測定」と表示された「測定条件設定」画面で[F1]キー（条件記憶）を押す。
「記憶条件リスト」が現われる。

2) [F2] キー（記憶）を押す。

3) 記憶させたいファイル番号を数値キーにて入力し、[ENTER] を押す。
「文字入力」画面が現われる。
入力したファイル番号がすでに埋まっている場合には更新するか否かを尋ねてくる。更新する場合には[ENTER]を、しない場合には[RETURN] を押す。

4) 「文字入力」画面でカーソルキー（[<]または[>]）で画面に現われているアルファベットなどの文字を順次選択し、[ENTER] を押す。

5) [F1] キー（終了）を押す。

2.2.4 定量測定

1) 測定条件での検量線を「2.2.3 検量線の作成」に従って作成する。

2) メニュー画面のとき,[F4] キー（条件呼出）を押す。
「条件記憶リスト」画面が現われる。

3) [F2] キー（設定）を押す。

4) 呼び出したいファイル番号を数値キーにて入力し、[ENTER] を押す。
指定したファイルの測定条件に設定され、自動的にその測定条件での測定モードになる。このとき「定量測定」という、測定条件の表示された画面が現われる。

5) 試料をセットし、[START/STOP] キーを押す。
測定が開始され、結果が表示される。[START/STOP] キーを1回押す毎に試料 No. が1ずつ増えていく。

6) 次の試料をセットし、[START/STOP] キーを押す。

2.2.5 メインテナンス

「メニュー」画面のとき、[4] キー（保守・設定）を押す。

「保守・設定」画面が表示される。

(1) ランプの点灯時間

1) 「保守・設定」画面のときに [1] キー（ランプの点灯時間）を押す。
「ランプの点灯時間」画面が現われる。この表示時間によりそのランプの通算使用時間を知ることができる。
ランプを交換したときは、[F4]キー（リセット (Reset)）を押して、表示時間を 0 に

戻す。

（注意事項）

・装置本体を移動するときは、必ずキセノンランプを取り外し、専用保護ケースに入れる。
キセノンランプに衝撃が加わると、キセノンガスの封入圧力が高いため爆発することがある。

・キセノンランプのバルブ部には手を触れないようにする。もし触れてしまった場合には添付クリーナーかエタノールをしみ込ませたガーゼなどで汚れをきれいにふき取る。
汚れが付着したままで点灯するとバルブが焼きつき、破損の原因となる。

・キセノンランプの寿命（メーカー保証期間）は500時間である。1000時間を越える使用はランプが破損し装置内部に大きなダメージを与えることがあるので絶対にしないこと。

・交換したキセノンランプは爆発の恐れがあるのでそのまま廃棄せず、厚手の布などでくるんだ後に、ハンマーなどで割ってから廃棄する。

B. 試料（油分散海水）の前処理

1. 用意するもの

・分液漏斗（100ml , 足を短くカットしたもの）
・分液漏斗振盪器
・分液漏斗用スタンド
・ガラス製漏斗
・濾紙（5A）
・薬さじ
・メスフラスコ（25 ml）
・ピペット（10 ml）
・精密天秤
・イソオクタン（蛍光分析用グレード）
・無水硫酸ナトリウム
・廃液受け用ビーカー（1L 程度，海水及びイソオクタン用）

2. 操作

1) 試料 50 ml を分液漏斗に直接受ける。分液漏斗には前もって 50 ml の標線を描いておく。
2) 試料の入った分液漏斗にイソオクタン 10 ml を添加し、栓をした後、振盪器で 5 分間激しく振盪する。
この間にガラス製漏斗に濾紙を敷き、約 1 g の無水硫酸ナトリウムを置く。また、漏斗出口には 25 ml のメスフラスコを置く。（試料の数だけ用意する）
3) 振盪後、数分間静置し、水相とイソオクタン相を分離する（水相が下になる）。
4) 分離後、分液漏斗の栓を開け、水相を排出する。
5) イソオクタン相を無水硫酸ナトリウムに通して脱水した後、下に置いてあるメスフラスコにいれる。
6) 上記無水硫酸ナトリウムをイソオクタン 10 ml で洗い、試料中の油分を完全にメスフラスコに回収する。
7) メスフラスコにイソオクタンを加え、25 ml に定容する。
8) 蛍光測定用セルに入れ、「2.2.4 定量測定」に従って蛍光強度を測定する。

（注意事項）
・使用後の分液漏斗、漏斗、メスフラスコはイソオクタンで洗浄し、次の測定時の妨げにならないようにする。

以上

付属資料 8.2.3-1

油分吸着実験マニュアル

油分吸着実験マニュアル

I. 本マニュアルの目的

本マニュアルはRO-2（含油海水の前処理）実験を行なうための手順及び機器の操作、メインテナンスを実験担当者が行なえることを目的としたものである。

II. 各実験実施法

A. 試料調製法

1. 用意するもの

・超音波ホモジナイザー（超音波工業 USH-300Z20S）
・ペリスタリックポンプ（コールパーマー 7524-00）
・シリコンチューブ（コールパーマー；Master Flex 80SL(Size: 18)）
・ガラス瓶(1L)
・ガラス製注射筒
・海水
・メスシリンダー(500 ml)
・25L ふた付きポリボトル
・海水（実験所要量，約 20 L，上記ボトルに前もって入れておく）
・TOC分析計（島津TOC-500，ハロゲンスクラバー付き）
・マイクロシリンジ
・ガラス製ピペット(5 ml 程度のもの)
・ガラス製試験管

2. 操作

2.1 油分散濃厚試料の調製

1) シリコンチューブをペリスタリックポンプのヘッドに巻き付けヘッドカバーをかぶせ、ポンプ本体に固定する。

2）ポンプ本体に巻き付けたシリコンチューブの片側を超音波ホモジナイザー乳化室の液入口に接続する。

3）超音波ホモジナイザー乳化室の出口にシリコンチューブを接続する。

4）ポンプ本体に巻き付けたシリコンチューブの片側（超音波ホモジナイザー乳化室の液入口に接続していない側）および超音波ホモジナイザー乳化室の出口に接続したシリコンチューブを1Lの海水をいれたガラス瓶に差し込む。

5）1Lの海水をペリスタリックポンプで超音波ホモジナイザー内を循環させながら、2.5mlのA重油を海水の循環しているシリコンチューブにガラス製注射筒を用いて注入し、そのまま10分間超音波処理を行なう。

(注意事項)

・ 超音波ホモジナイザー作動時はその発生する音に気を付ける。

・ 超音波処理後は乳化室内を純水1Lにて洗浄する。

（濃厚試料調製時のシステムを使用。但し、循環させず出口からの洗浄液は排出する）

2.2 油分散試料濃度の調整

1）油分散濃厚試料をメスシリンダーにはかりとり、これをポリボトルに前もって入れておいた海水に添加する。

2）ポリボトルにふたをしてから、容器を激しく揺り動かし、油分散濃厚試料を混合・希釈する。

3）希釈した試料の一部をガラス製ピペットにて容器の中央部からサンプリングし、油分をTOC分析計にて測定する。

4）油分が希望している濃度と異なっている場合には、濃厚試料をさらに添加または逆に希釈し、希望濃度に調整する。

B. 油分吸着実験法

1. 用意するもの

・ペリスタリックポンプ（コールパーマー 7524-00）

・シリコンチューブ（コールパーマー；Master Flex 80SL(Size: 18)）

・ガラス製カラム（内径 20 mm，長さ 30 cm）

・スタンド（カラム支持用）

・三方コック

・ピンチコック

・濃度調整済み油分散試料（25L ふた付きポリボトルに入れてあるもの）

・各種充填剤

・ビーカー

・漏斗

・海水

・洗瓶

2. 操作

2.1 装置組立

1) シリコンチューブをペリスタリックポンプのヘッドに巻き付け、ヘッドカバーをかぶせ、ポンプ本体に固定する。

2) 同チューブのカラム側先端に三方コックを接続する。

3) ビーカーに所要量の充填剤を入れ、水を注ぎ湿潤させる。

4) カラム 出口側の充填剤支持部に、外部リングに通したピンチコック付きのシリコンチューブを接続する。

5) カラム出口側の充填剤支持部にステンレスネットを敷き、0リング、ガラス筒を接続する。

6) ピンチコックを閉めた後、純水を洗瓶にてカラム底から約 5 cm の高さまで入れる。

7) ガラス筒上端に漏斗を置き、洗瓶から純水を流しながらビーカーにいれた充填剤を所要量流し込む。

この際、水がガラス筒上端に近づいたらピンチコックを開き、水位を充填剤の

少し上までに調整する。

8) 純水をガラス筒上端までつぎたす。

9) ガラス筒から漏斗を外し、上端（カラム入口側）に0リング、三方コック付きシリコンチューブを接続した充塡剤支持部、外部リングを接続する。

10) カラム出口側のシリコンチューブ先端を排水容器に導く。

11) カラム入口側に接続されているシリコンチューブの先端を海水の入っている容器にいれ、ポンプにて通水し、カラム内を平衡化する。

（現在は、充塡剤体積の約 30 倍以上を通水している）

2.2 吸着実験

1) カラム入口側に接続されているシリコンチューブの先端を濃度調整済み油分散試料（25L ふた付きポリボトルに入れてあるもの）に入れる。

2) ポンプにて上記試料をカラム内に通水する。

3) カラム入口直前の試料を三方コックから、出口試料を出口側シリコンチューブの先端から試験管に採取する。

4) 試料の油分濃度をTOC分析計にて測定する。

C. 油分測定法

油分測定には四塩化炭素抽出法、TOC法、ガスクロ法など各種あるが、現在は当地における四塩化炭素の品質、サンプル数の多さ、実験スケールの大きさに由来するサンプル量の制約などにより、現在の状況下でもっとも適していると思われるTOC法を使用している。

但し、TOC法は試料の一部のみをサンプルとしているので本法を油分測定に用いる際には以下の条件を満たしていることが必要である。

・試料中の油分ができる限り均一に分散していること。

現在、試料は超音波法により分散させており、その油滴径は1μmよりも小さく、ほぼ均一に分散していると考えてよい。

1. 用意するもの

・メスフラスコ

・フタル酸水素カリウム（特級）

・炭酸水素ナトリウム（特級）

・炭酸ナトリウム（特級）

・高温炉（600。Cまで上がるもの）

・デシケーター

・高純度空気（ CO2 , CO , HC 各 1ppm 以下 ）

・TOC分析計（島津TOC-500 , ハロゲンスクラバー付き）

・マイクロシリンジ（ 10 μl , 50 μl , 100 μl ）

・0.6 N 塩酸

・パラフィルム

・ビーカー

2. 操作

2.1 標準液の作成

2.1.1 TC標準液の作成

1) フタル酸水素カリウム（特級）2.125gを正確にはかり、ゼロ水にとかし、1Lメスフラスコに定容する。この溶液はTC=1000ppmに相当する。これをTC標準原液とする。

2) TC標準原液を正確にゼロ水で希釈し、標準溶液とする。

2.1.2 IC標準液の作成

1) 炭酸水素ナトリウム（特級）3.50gと、あらかじめ500~600。Cで30分間加熱した後デシケーター中で放冷した炭酸ナトリウム（特級）4.41gを正確にはかり、ゼロ水にとかし、1Lメスフラスコに定容する。この溶液はIC=1000ppmに相当する。これをIC標準原液とする。

2) IC標準原液を正確にゼロ水で希釈し、標準溶液とする。

2.2 装置の立ちあげ

1) MAIN及びFURNACEスイッチをONにする。

2) キャリアガスのバルブを開ける。

キャリアガスのボンベ二次圧は1kg/cm2 (1.42psi)、流量は150ml/min.に設定する。キャリブレーション以後は動かさないようにする。

3) 炉の温度が上がり、装置が安定化する（READYランプ点滅）まで待つ。

2.3 キャリブレーション

2.1 にて調製した標準液を用いてキャリブレーションを行なう。

1) 推定される試料濃度より、用いる標準液濃度及び測定レンジ、試料注入量を決定する。

測定レンジ、試料注入量は以下の式によって選定される。

測定レンジ(ppm F.S)×注入量(μl)／RANGE(×1or3or10)=300 *1

2) STD LOW→数値→ENT　とキーを押し、低濃度試料（通常ゼロ水）の濃度をインプットする。

3) READYランプが点燈するのを確認してから、TCを押し、直ちにTC注入口のバルブを開け、シリンジパッキンを注入口に押し当てながら、低濃度試料（通常ゼロ水）を一気に注入する。

4) そのまま、約5秒間してから針を抜き、直ちに注入口のバルブを閉じる。

5) 必要回数だけ、2)~4)の操作を繰り返す。

6) 測定値のなかにたの測定値とかけ離れた異常データがあるときは、CLEAR→数値→ENTER　とキーを押し、その数値を除外する。

7) ENTを二回押す。*2

8) 続いて高濃度側標準液を2)~6)と同様の方法で注入する。

9) ENTを二回押す。*3

この操作により、CAL. FACTOR　AおよびBが印字される。

（注意事項）

*1: この式により求められた注入量が中途半端な場合には、その数字より少なめのきりの良い注入量の方が好ましい。

*2: この操作により、CAL. FACTOR　AおよびBが印字されるが、ここでの数字は正しい値ではない。

*3: ここで得られた数値は、一連のキャリブレーション操作により得られたものであり、今後、この数値を測定前にインプットすることにより標準液を用いてのキャリブレーションを行なわなくても、直ちに測定が可能となる。2.4 測定

1) SAMPLE NUMBER→数値→ENT　とキーを押す。

2) READYが点燈するのを確認してから、TC（またはIC）を押し、直ちにTC（またはIC）注入口のバルブを開け、シリンジパッキンを注入口に押し当てながら、試料を一気に注入する。

3) 2.3 4)~6)と同様の操作を行なう。

4) ENT　を押す。

この操作により、測定値の平均が算出される。

5) ENT　を押す。

この操作により、試料の希釈倍率をインプットすることができる（通常は必要なし）。試料を希釈した場合には、ここで希釈倍率を入力する。

6) ENT　を押す。

この操作により、READYが点滅し、次の試料を測定することができる。

7) 1)~6)を繰り返す。

2.5 測定終了

1) FURNACEスイッチをOFFにする。

2) 15分以上経過後、キャリアガスボンベのバルブを閉止する。

3) MAINスイッチをOFFにする。

2.6 メインテナンス

2.6.1 TC触媒の活性化

以下のような場合にはTC触媒の活性化が必要となる。

・触媒や燃焼管を交換したとき

・長期休止後測定したとき

・塩分含有試料、ICを多く含む試料、アルカリ性試料、無機性SSなどを含む試料などを測定した後

活性化操作は以下の手順にて行なう。

1) IC反応管にIC充塡剤が充塡されていることを確認する。

2) 装置前とびら右側にある、流路切り替え部のTC-IC継手に接続されている配管を外し、パラフィルムをかぶせて、ガスの流れを止める。

3) 約0.6Nの塩酸を約80μlを約2分以上の間隔で2回注入する。

4) 同量のゼロ水を1~2回注入し、15分以上放置する。

5) 流路切り替え部及びドレンチューブをもと通りに戻す。

6) READYランプが点滅するのを待つ。

（注意事項）

・以上の操作時にはキー操作は不要であり、READYランプが点灯していなくても構わない。

・ICおよびVOC回路にはこの操作を行なわないこと。

2.6.2 IC反応液の補充

1) IC注入口を外す（IC反応管は装置に装着したままで良い）。

2) 約1.5mlのIC反応液をピペットなどにて添加する。

（多く加えた場合には過剰分がドレンへ排出され、ラインが閉塞する可能性があるので注意すること）

（注意事項）

・本操作は一週間に一度を目安とする。

2.6.3 加湿器水位の点検

1） 加湿器水位が2本の標線内にあることを確認する。

2） 水位が下の標線以下の場合には、精製水を補給口から補給する。

（注意事項）

・年1回程度は新しい0.3%NaOHと交換する。

JICA