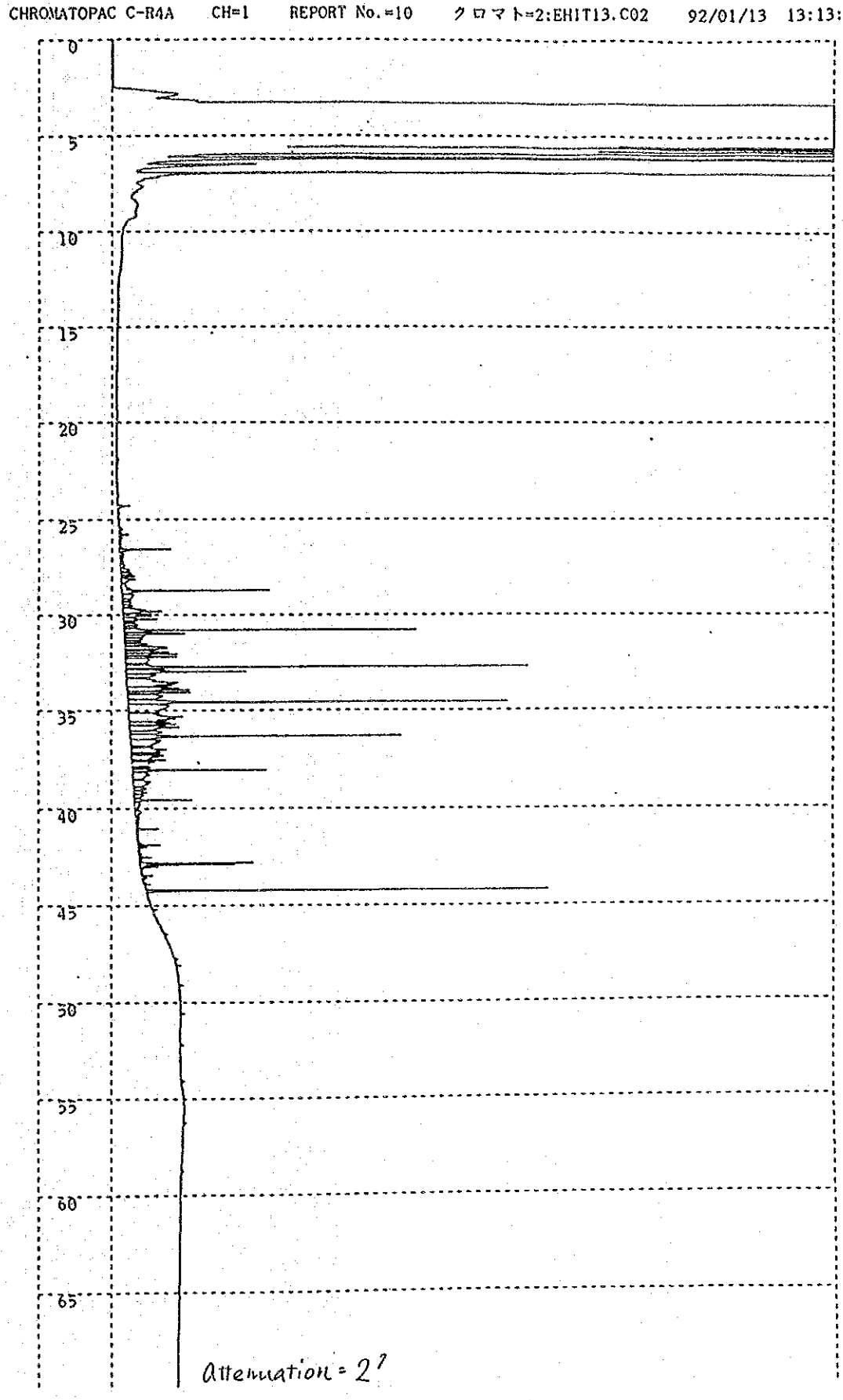
Fig. 3 のピーク情報 その3

158	46.291	56238	9621	V	0.1329
159	46.448	280331	12815	V	0.6627
160	46.883	59411	8819	V	0.1404
161	47.01	97834	8474	V	0.2313
162	47.187	64619	8184	V	0.1528
163	47.294	77866	8154	V	0.1841
164	47.5	57022	7227	V	0.1348
165	47.721	90357	8813	V	0.2136
166	47.843	55073	7110	V	0.1302
167	47.994	74261	6749	V	0.1755
168	48.125	69893	6593	V	0.1652
169	48.391	115634	6441	V	0.2734
170	48.645	106493	6095	V	0.2517
171	49.056	73489	6106	V	0.1737
172	49.2	62638	4977	V	0.1481
173	49.419	79132	4628	V	0.1871
174	49.721	44178	4305	V	0.1044
175	49.919	47423	4387	V	0.1121
176	50.144	82073	3950	V	0.194
177	50.522	74232	4073	V	0.1755
178	50.992	66234	2773	V	0.1566
179	51.292	18121	2295	V	0.0428
180	51.639	92073	3393	V	0.2177
181	52.125	23959	1925	V	0.0566
182	52.317	9803	1403	V	0.0232
183	52.439	17893	1473	V	0.0423
184	52.772	41789	2115	V	0.0988
185	53.333	10688	772	V	0.0253
186	53.467	2767	724	V	0.0065
187	53.645	10695	779	V	0.0253
188	53.965	13152	793	V	0.0311
189	54.142	4920	478	V	0.0116
190	54.395	6866	820	V	0.0162
191	54.775	3299	228	V	0.0078
193	55.265	1703	124	V	0.004
194	56.067	1003	191		0.0024
195	56.529	1678	321		0.004
196	56.794	1249	221		0.003
197	57.05	761	114	V	0.0018
198	57.187	1971	177	V	0.0047
199	57.4	480	80	V	0.0011
200	57.543	403	66		0.001
202	58.395	657	98		0.0016
203	58.546	711	107	V	0.0017
204	58.946	12133	489	V	0.0287
205	59.567	704	33		0.0017
206	59.875	751	81	V	0.0018
208	60.34	361	53		0.0009
212	60.973	1425	137	V	0.0034
213	61.514	4827	240	V	0.0114
214	61.75	1527	132	V	0.0036
215	62.25	1100	56	V	0.0026
216	62.389	697	77	V	0.0016
220	64.117	1491	108		0.0035
221	64.483	495	31	V	0.0012
222	64.992	326	49		0.0008
223	66.671	515	42		0.0012
225	67.509	6542	297		0.0155
226	68.509	329	42		0.0008
	TOTAL	42301688	4992028		100

4.23×10⁷

重油1000μg/mlの海水50℃蒸留水のn-ヘキサン抽出液　Fig. 4



** 定量計算結果 **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	2	10.796	1877	206	V		0.048	
	3	11.078	5923	217	V		0.1515	
	4	11.417	2523	196	V		0.0645	
	5	12.792	621	98			0.0159	
	8	21.266	383	55	V		0.0098	
	9	21.915	2131	279			0.0545	
	10	23.267	2083	114			0.0533	
	11	23.494	1286	202	V		0.0329	
	12	23.667	1108	187	V		0.0283	
	13	23.81	1623	340	V		0.0415	
	14	24.297	11418	2209			0.292	
	15	24.597	1293	216	V		0.0331	
	16	24.733	883	185	V		0.0226	
	17	24.883	518	106	V		0.0132	
	18	25.209	2238	181	V		0.0572	
	19	25.483	5858	648	V		0.1498	
	20	25.794	9268	1715	V		0.237	
	21	25.983	4184	792	V		0.107	
	22	26.247	1879	248	V		0.0481	
	23	26.584	33010	8461			0.8443	
	24	26.841	5119	721	V		0.1309	
	25	26.942	3742	558	V		0.0957	
	26	27.143	3148	428	V		0.0805	
	27	27.269	2743	490	V		0.0701	
	28	27.437	6162	715	V		0.1576	
	29	27.643	13329	1617	V		0.3409	
	30	27.783	6477	1686	V		0.1657	
	31	27.834	11349	1717	V		0.2903	
	32	27.998	9441	2317	V		0.2415	
	33	28.165	20583	2378	V		0.5264	
	34	28.754	98810	24035	V		2.5272	
	35	28.986	25672	2157	V		0.6566	
	36	29.263	10025	1462	V		0.2564	
	37	29.366	12736	1378	V		0.3257	
	38	29.535	10626	1711	V		0.2718	
	39	29.839	58998	6821	V		1.509	
	40	29.979	42344	4946	V		1.083	
	41	30.247	31265	5668	V		0.7996	
	42	30.37	13199	2140	V		0.3376	
	43	30.523	17024	2525	V		0.4354	
	44	30.614	17879	2442	V		0.4573	
	45	30.808	157350	51219	V		4.0245	
	46	31	49113	10120	V		1.2561	
	47	31.114	29692	3669	V		0.7594	
	48	31.264	25644	3103	V		0.6559	
	49	31.413	20509	2913	V		0.5245	
	50	31.539	23986	3937	V		0.6135	
	51	31.694	60595	7374	V		1.5498	
	52	31.842	19110	5913	V		0.4888	
	53	31.963	43566	7583	V		1.1143	
	54	32.064	50602	8821	V		1.2942	
	55	32.224	38629	8882	V		0.988	
	56	32.392	61886	4326	V		1.5828	
	57	32.57	17962	3972	V		0.4594	
	58	32.754	219721	66750	V		5.6197	
	59	33.007	131509	21103	V		3.3636	
	60	33.169	57865	5201	V		1.48	
	61	33.56	158320	8862	V		4.0493	
	62	33.941	96912	10697	V		2.4787	
	63	34.094	60312	10992	V		1.5426	
	64	34.265	90564	6170	V		2.3163	
	65	34.591	239258	67277	V		6.1194	
	66	34.85	160884	6893	V		4.1149	
	67	35.325	161946	9479	V		4.142	
	68	35.713	66009	8022	V		1.6883	
	69	35.87	88051	8361	V		2.252	
	70	36.115	54852	5715	V		1.4029	
	71	36.331	175373	43822	V		4.4854	
	72	36.54	78167	5375	V		1.9993	
	73	36.81	15070	3883	V		0.3854	
	74	36.87	14583	3799	V		0.373	
	75	37.013	67690	6180	V		1.7313	
	76	37.204	24814	5078	V		0.6347	
	77	37.315	58839	5410	V		1.5049	

Fig. 4 のピーク情報 その1

78	37.546	24120	5678	V	0.6169
79	37.662	29347	4058	V	0.7506
80	37.82	23206	3117	V	0.5935
81	37.983	78236	20982	V	2.001
82	38.239	52088	2882	V	1.3322
83	38.612	47229	3049	V	1.208
84	38.926	18204	2189	V	0.4656
85	39.159	18621	2340	V	0.4763
86	39.316	15521	1665	V	0.397
87	39.561	32941	9704	V	0.8425
88	39.744	15902	1028	V	0.4067
89	40.148	13189	985	V	0.3373
90	40.538	5232	662	V	0.1338
91	40.629	4488	418	V	0.1148
92	41.072	12439	3675	V	0.3182
93	41.217	1940	221	V	0.0496
94	41.417	1131	247	V	0.0289
95	41.67	2283	299		0.0584
96	41.896	19144	3864	V	0.4896
97	42.344	1878	522		0.048
98	42.52	8820	2037		0.2256
99	42.791	110492	19876		2.826
101	43.455	10432	1745		0.2668
102	43.903	2996	1051		0.0766
103	44.237	190334	66749		4.8681
104	44.75	812	140		0.0208
106	45.224	3132	1049		0.0801
107	45.367	459	101		0.0117
108	46.502	3789	613		0.0969
109	47.733	1844	-32		0.0472
110	48.113	2642	742		0.0676
111	48.833	353	94		0.009
112	49.122	2654	741		0.0679
113	49.685	820	47		0.021
114	49.925	600	52		0.0154
115	50.533	3437	151		0.0879
116	51.067	1543	77		0.0395
117	52.228	2983	611		0.0763
118	52.863	685	89		0.0175
121	54.095	3150	616		0.0806
122	56.253	2668	530		0.0682
123	57.167	705	39		0.018
124	58.782	2163	403		0.0553
125	61.757	1650	238		0.0422
126	65.271	1383	133		0.0354
127	65.921	594	34		0.0152
128	68.891	441	38		0.0113
129	69.458	960	91		0.0246
	TOTAL	3909825	681107		99.9999

3.9098×10^6

Fig. 4 のピーク情報

その2

911015 © Shimadzu

重油1000μg/mlの海水80℃蒸留水のn-ヘキサン抽出液 Fig. 5

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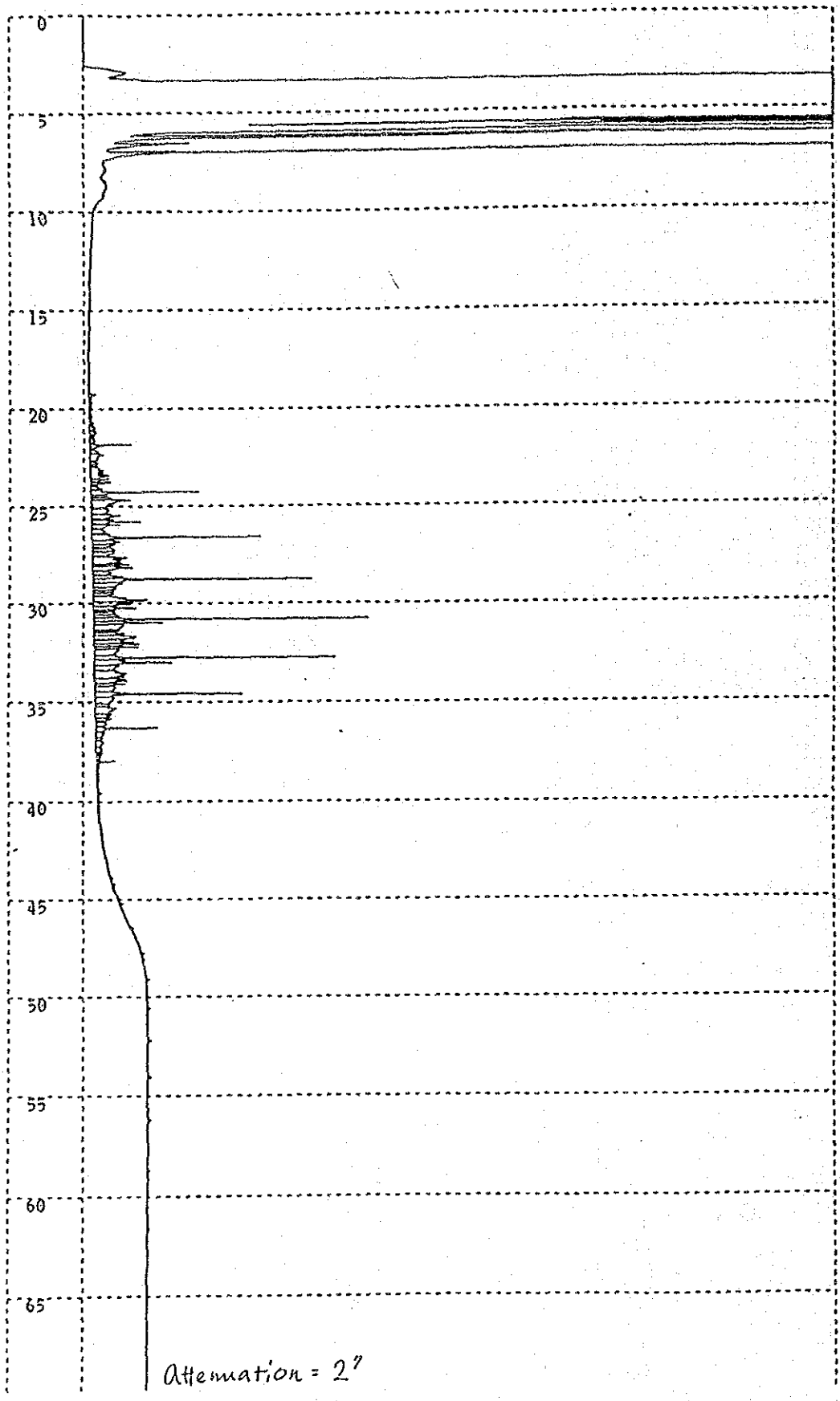


** 定量計算結果 **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1	12.083	687	50			0.0195	
	3	13.516	591	102			0.0167	
	4	16.767	607	60			0.0172	
	5	18.16	763	111			0.0216	
	6	18.383	1449	131	V		0.041	
	7	18.563	964	115	V		0.0273	
	8	18.979	1153	129			0.0326	
	9	19.276	10393	1206	V		0.2943	
	10	19.689	2546	426	V		0.0721	
	11	19.9	2076	265	V		0.0588	
	12	20.1	1335	170	V		0.0378	
	13	20.397	5459	338	V		0.1546	
	14	20.723	7021	660	V		0.1988	
	15	20.965	11433	1006	V		0.3237	
	16	21.142	10888	1156	V		0.3083	
	17	21.383	4966	655	V		0.1406	
	18	21.633	10079	666	V		0.2854	
	19	21.842	33000	6856	V		0.9344	
	20	22.2	12405	1353	V		0.3513	
	21	22.349	33687	2245	V		0.9539	
	22	22.771	10278	1327	V		0.291	
	23	22.883	7774	997	V		0.2201	
	24	23.136	57088	2008	V		1.6165	
	25	23.625	17803	2982	V		0.5041	
	26	23.797	17114	3259	V		0.4846	
	27	24.081	17077	1916	V		0.4835	
	28	24.29	75288	17361	V		2.1318	
	29	24.446	17547	2574	V		0.4968	
	30	24.583	24876	3534	V		0.7044	
	31	24.708	90679	6815	V		2.5676	
	32	25.096	7256	2452	V		0.2055	
	33	25.221	41009	3049	V		1.1612	
	34	25.49	50255	4841	V		1.423	
	35	25.797	55256	7953	V		1.5646	
	36	25.966	37308	4614	V		1.0564	
	37	26.247	31510	2226	V		0.8922	
	38	26.596	125116	27104	V		3.5427	
	39	26.845	45076	4652	V		1.2763	
	40	26.983	34611	3772	V		0.98	
	41	27.265	40625	3774	V		1.1503	
	42	27.344	45199	3629	V		1.2798	
	43	27.655	101307	5741	V		2.8686	
	44	28.011	30884	6252	V		0.8745	
	45	28.178	60400	6829	V		1.7103	
	46	28.452	36462	3259	V		1.0324	
	47	28.645	45390	5179	V		1.2852	
	48	28.771	139850	34773	V		3.9599	
	49	28.992	58484	5089	V		1.656	
	50	29.232	32377	3885	V		0.9168	
	51	29.365	26253	3318	V		0.7434	
	52	29.538	27628	3491	V		0.7823	
	53	29.855	92665	9199	V		2.6239	
	54	29.994	67049	6962	V		1.8985	
	55	30.263	46819	7174	V		1.3257	
	56	30.385	23545	3679	V		0.6667	
	57	30.538	26842	3725	V		0.7601	
	58	30.676	33408	4253	V		0.946	
	59	30.823	149234	44945	V		4.2256	
	60	31.013	63358	11430	V		1.794	
	61	31.147	79029	5184	V		2.2377	
	62	31.412	28092	4348	V		0.7954	
	63	31.552	40561	5129	V		1.1485	
	64	31.706	62620	7197	V		1.7731	
	65	31.856	19275	5487	V		0.5458	
	66	31.976	42209	6954	V		1.1952	
	67	32.078	52774	7655	V		1.4943	
	68	32.237	36132	7317	V		1.0231	
	69	32.355	73633	4027	V		2.085	
	70	32.76	135643	40333	V		3.8408	
	71	33.015	99109	12667	V		2.8063	
	72	33.179	53960	4232	V		1.5279	
	73	33.447	105189	4025	V		2.9785	
	74	33.949	58120	5509	V		1.6457	
	75	34.104	33060	3111	V		0.9361	
	76	34.278	53231	3554	V		1.5073	
	77	34.594	105808	24174	V		2.996	

Fig. 5のピーク情報

その1

095

223-02000-01

911015

⊕ Shimadzu

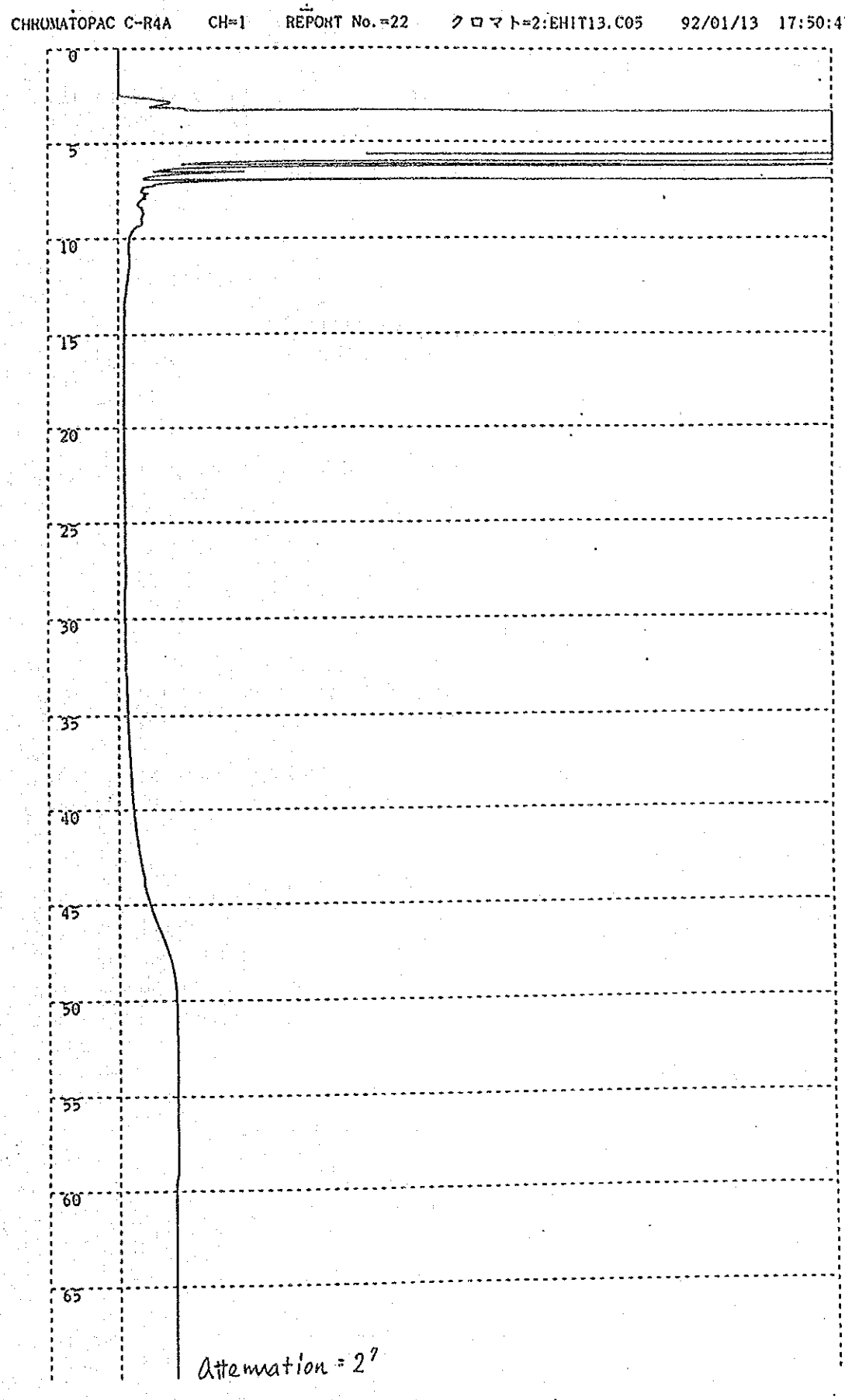
Fig. 5 のピーク情報 その2

78	34.855	57848	3180	V	1.638
79	35.093	18504	2397	V	0.5239
80	35.337	57779	3567	V	1.636
81	35.633	26778	2632	V	0.7582
82	35.881	22033	2599	V	0.6239
83	36	4910	1663	V	0.139
84	36.118	20125	1743	V	0.5699
85	36.333	44708	9433	V	1.2659
86	36.549	19783	1438	V	0.5602
87	36.829	8770	1045	V	0.2483
88	37.028	19139	1339	V	0.5419
89	37.35	13025	983	V	0.3688
90	37.567	4547	1056	V	0.1287
91	37.683	8327	689	V	0.2358
92	37.993	11303	3139	V	0.32
93	38.253	3815	290	V	0.108
94	38.629	3501	267	V	0.0991
95	38.917	1198	146	V	0.0339
96	39.033	518	117	V	0.0147
97	39.2	490	101	V	0.0139
98	39.576	2484	708	V	0.0703
100	39.867	405	43	V	0.0115
101	40.2	522	62		0.0148
102	40.483	417	52	V	0.0118
104	41.088	747	270		0.0212
105	42.522	1136	300		0.0322
106	43.867	1281	-1		0.0363
107	44.248	2174	517		0.0615
108	45.223	2211	555		0.0626
109	46.494	3158	567		0.0894
110	47.667	1717	-3		0.0486
111	48.033	1110	13		0.0314
112	49.133	2436	617		0.069
113	49.867	2386	70		0.0675
114	50.6	2633	543		0.0746
115	51.067	621	31		0.0176
116	51.533	313	18		0.0089
117	53.268	1052	33		0.0298
118	54.049	3221	682		0.0912
119	56.189	3593	606		0.1017
120	57.048	345	38		0.0098
121	57.749	1420	52		0.0402
122	58.694	2595	425		0.0735
123	60.433	442	26		0.0125
124	60.927	581	47		0.0165
125	61.647	2174	302		0.0616
126	64.233	354	24		0.01
127	65.143	1367	154		0.0387
128	69.291	1000	91		0.0283
	TOTAL	3531630	495282		99.9999

3.5316×10^6

n-ヘキサン (blank)

Fig. 6



M 4 － 4　実験研究推進のための配布資料

AGENDA ON NOV. 2

(M-4)

*1) ① Instrument apparatus and reagents needed for experiment

To use your Research Center equipment if necessary except specific instruments

② Collection of information

More emphasis on collection of materials for your practice or its own accumulated so far

③ Preparation for prelimirary report

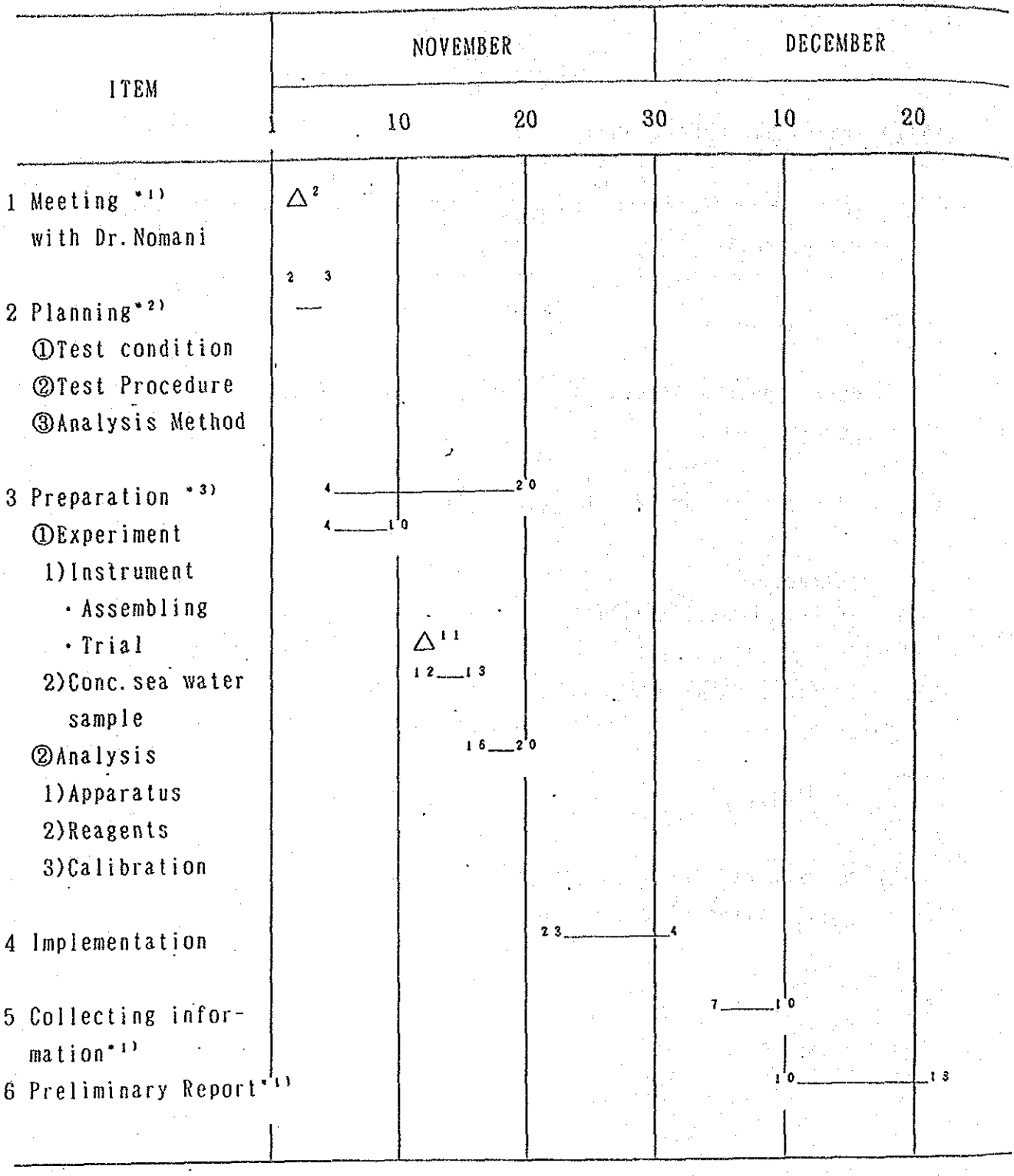
To recommend that you are to prepare a preliminary report in reference to the inception report

*2) ① To discuss influence on quality of product water
② Reconfirmation of test condition and analysis method for oil etc.

*3) Apparatus transported from YANBU and JAPAN

① Oil content determination apparatus etc.
② Rotary evaporater under Mr. Al-Satihi's care at present

DETAILED SCHEDULE ON RESEARCH THEMES(M-4)



M-4 Experiment

3, NOV. '91
Rev. 23. NOV. '91

1. Conditions

		Temperature °C 50	80	95	Test order
Conc. of oil mg/ℓ	0	0	0	0	①
	10	0	0	0	②
	50	0	0	0	③
	100	0	0	0	④
Vapor pressure	mmHg (approx.)	90	350	630	
Sampling order		end ←			begin with

2. Quantitative analysis for constituents in samples

(1) Distillate — Oil content and Cn distribution with GC/MS for every series of tests.
In comparison with data by GC/MS, oil content with IR/TOC for 50/100 mg/ℓ oil tests independent of GC/MS tests.

(2) Brine & Concentrated Sea Water — pH, EC & oil before and after 50 °C experiment has been done for every series of experiments.

M-4 Test procedure 5, NOV. '91
Rev, 23, NOV. '91

1. Trials

① Put 500ml of concentrated seawater (brine) into the evaporator flask.

② Heat the oil until the temperature of brine reached to 95°C, refluxing distillate with the trap stopper closed.

③ If reached, make negative pressure so that gentle flushing may be maintained. 630 mm Hg and open the trap stopper to begin with trap of distillate sample.

④ For 10 minutes, continue to trap distillate sample and break vacuum.

⑤ Then Take out ca. 20 ml of distillate sample and measure the amount of it accurately.

⑥ Cool the brine to 80°C and increase negative pressure to 350 mmHg, refluxing distillate.

⑦ According to the procedure as shown the above, proceed the test.

⑧ Cool the brine to 50°C and increase negative pressure to 90 mmHg.

⑨ Repeat the above procedure, and measure the amount of brine.

⑩ Conduct analysis of oil for each samples.

2. Main experiments

Continuous test from 95°C to 50°C for each condition of oil concentration is required to be completed within a day.

① Put 500 ml of brine and accurately weighed heavy oil into the flask and shake vigorously.

② Proceed the test as shown in trials.

3. Determination of oil concentration

In case of the analysis of oil by IR/TOC and GC/MS, total amount of each fraction and concentrated brine sea water should be used.

ca. 20 ml* of fraction —shake→ 1 ml → TOC

the rest —extract 2 times with 5 ml of CCl_4→ IR

ca. 20 ml* of fraction —extract 2 times with 10 ml of CH_2Cl_2→ GC/MS

* have to be measured accurately.

TEST RECORD

M-4 Nov. , 1991

Concentration of Oil in the Brine Sample: mg/L

* Brine Sample	Before Test	After Test
Amount (ml)		
pH		
E.C. (μs/cm)		
Oil (ppm)		

* Distillate Sample -- TOC	95°C	80°C	50°C
Amount (ml)			
TOC (mg)			
TOC (ppm)			

* Distillate Sample - IR	95°C	80°C	50°C
Amount (ml)			
Extraction : CCl_4 (ml)			
Oil (mg)			
Oil (ppm)			

Concentration of Oil in the Brine Sample: mg/L

* Distillate Sample -GC/MS	95°C		80°C		50°C	
Amount (ml)						
Extraction : CH_2Cl_2						
C 10 - Compound	mg	%	mg	%	mg	%
C11						
C12						
C13						
C14						
C15						
C16						
C17						
C18						
C19						
C20						
C21						
C22						
C23						
The rest						
Total oil (mg)						
Total oil (ppm)						

MATERIALS(2) FOR #4 M-GROUP MEETING 13 DEC. 1991

SOME TECHNOLOGICAL POINTS ON DISCUSSIONS OF THE RESULTS

1. Calculated value of oil concentration in distillates by Raoult's law *

In dilute solution, vapor pressure(p_1) of solvent is proportional to its molar fraction(N_1)

$$p_1 = p_1^\circ N_1$$

Where, p_1° :Vapor pressure(N_1=1) of pure solvent

2. Factors influenced on recovery of oil

3. Changes in constituents of oil by distillation

Changes in C_{16}, C_{17}, C_{18}

NOTE: * Legend in Fig.

—— · —— :Calculated for each hydrocarbon in the order of increasing number of carbons, assuming two liquid phase of water-pure hydrocarbons

—— ● —— :Calculated assuming two liquid phase of water-oil in the ideal solution where the Raoult's law is applicable to any regions

—— ○ —— :The measured value

~~6/17~~

Fig. Calculation of Oil in Distillate

1. Introduction

Reference to the inception report

2. Theory

(1) Behavior of volatile components during sea water desalination process

(2) Evaluation of literatures by information retrieval

3. Experimental Procedures

(1) Experiment apparatus (e.g. schematic diagram)

(2) Experiment conditions

(3) Procedure

- Distillation
- Distillate samples
- Analytical methods for oil (e.g. Principle etc.)

4. Results

(1) Data

(2) Observation (oil particulate, smell, degradation etc)

5. Discussions

(1) Transfer of Oil

- Mass Balance (e.g. Recovery, escaped volatile oil etc
- Transfer rate
- Composition change by distillation (e.g. volatile etc)
- Comparison between theory & experiment data etc (Mass balance, Transfer rate)

(2) Evaluation of Analytical method for oil

- TOC for water-soluble organic substances
- IR for oily matters
- GC/MS for organic substances, especially composition of oily matters

6 Conclusions

(1) Behavior of oil components

(2) Evaluation of Analysis

7 Recommendation for Future Work

In order to get data to be able to quantitatively evaluate oil transfer into product water, highly simulated test plant will be needed.

8 References

9 Acknowledgements

RESUME OF REPORT FOR RESEARCH THEMES: M-1 & M-4
(contents) (JICA Team's Idea)

No.	M − 1	M − 4
I	Main title	
	Scale prevention for MSF plant supplied with oil-contaminated sea water	Quality of product water in MSF plant supplied with oil-contaminated sea water
II	Sub-title (from Inception Report)	
	Laboratory experiments on scale prevention	Analysis of oil dispersed in raw sea water at the heat rejection section of MSF plant
III	Author	
	• Dr. Essam E. F. El-Sayed • Saad Al Sulami • Tadatsugu Hamada	• Dr. A. B. Nomani • Yoshio Hamao • Tadatsugu Hamada
1.	Introduction	
	• On basis of outline of Introduction in inception report • In addition to above, refer to it that scale prevention has something close to do with dicision for site in design.	• On basis of outline of Introduction in inception report • In addition to above, refer to application of suitable method for oil analysis.
2.	Theory (This title could be changed to more appropriate one. Result of litereture survey shall be included.)	
	• Technological explanation concering acid and chemical dosing method (To emphasise importance of alkalinity for water conditioning. To explain threshold effect and crystal distortion in chemical dosing method) • Fouling factor peculiar to methods above. (On basis of measurment in opereting MSF plant in Saudi-Arabia)	• Behavior of volatile components MSF process (To explain that it is possible to analyse this on basis of the same way as steam-distillation. To present results of simulated calculation on given conditions) • To present analytical methods for oil in water. To explain advantage or disadvantage for each. To suggest effectiveness of GC/MS.
3.	Planning and procedure of experiments	
	• Experiment conditions • Measurement items • Experiment apparatus (e.g. configuration) • Experimental procedure	
4.	Results	

INCOMPLETION

SCHEDULE ON MAKING THE REPORTS FOR M-1, M-4 (REV-02)

DATE	MON.	YEAR	Dr. Essam	Dr. Nomani	Hamada	Hamao	Saad
16	DEC.	1991	Discuss contents of report with Hamada (Goto EGYPT)	(Goto INDIA)	Discuss contents of report with Dr. Essam	Check report	Test for brushing up of report
17	DEC.	1991					
18	DEC.	1991					
19●	DEC.	1991			(Holiday)	(Holiday)	(Holiday)
20●	DEC.	1991	Make report *Introduction *Theory *Discussion *Conclusion *Reference *Acknowledge.	Brush up report	(Holiday)	(Holiday)	(Holiday)
21	DEC.	1991			Check report of M-4 and discuss contents of M-1 report with Saad	Check report	Make report *Planning *Exp. method *Exp. result
22	DEC.	1991				Preparation of transportation	
23	DEC.	1991					
24	DEC.	1991					
25	DEC.	1991					
26●	DEC.	1991			(Holiday)	(Holiday)	(Holiday)
27●	DEC.	1991			(Holiday)	(Goto YANBU)	(Holiday)
28	DEC.	1991				SWCC YANBU FACILITY	
29	DEC.	1991					
30	DEC.	1991					
31	DEC.	1991			Receive report from Dr. Basu	(Go back to JUBAIL)	
1	JAN.	1992					
2●	JAN.	1992			(Holiday)	(Holiday)	(Holiday)
3●	JAN.	1992			(Holiday)	(Holiday)	(Holiday)
4	JAN.	1992	(Go back to SWCC)		Brush up report with Dr. Essam and Dr. Basu	Check report	
5	JAN.	1992	Brush up report with Hamada				
6	JAN.	1992					
7	JAN.	1992					
8	JAN.	1992		(Go back to SWCC)			
9●	JAN.	1992	(Holiday)	(Holiday)	(Holiday)	(Holiday)	(Holiday)
10●	JAN.	1992	(Holiday)	(Holiday)	(Holiday)	(Holiday)	(Holiday)
11	JAN.	1992		Brush up report with Hamada and Hamao	Check corrosion research instrument	Brush up report with Dr. Nomani and Hamada	
12	JAN.	1992					
13	JAN.	1992	Complete the tentative report:M-1	Complete the tentative report:M-4			
14	JAN.	1992			Receive the tentative report:M-1	Receive the tentative report:M-4	
15	JAN.	1992					
16●	JAN.	1992			(Goto JAPAN)		

3. R1 添付資料（逆浸透における殺菌法に関する研究）

R 1 － 1 Sterilization of Seawater ---------- 3- 1

APPENDIX R1-1

STERILIZATION OF SEAWATER

PROJECT #R1

ONE OF JOINT PROJECTS BETWEEN

SWCC AND JICA

JANUARY 1922

BY

HASSAN MUNSHI

SWCC RDTC, JUBAIL

SAUDI ARABIA

Introduction

Disinfection, literally, means free from injection. A disinfectant destroys disease for other harmful microorganisms, but not necessarily unharmful ones or ordinary bacterial spores.

Disinfection techniques can be roughly classified into two types chemical and physical. Popular chemical disinfectants for water include chlorine and other halogens, chlorine dioxide, chloramine, hydrogen peroxide and ozone.

Physical disinfection methods includes ultraviolet light, ultrasonic and ionizing radiation. The most widely used methods will be briefly described and compared.

Project RJ
B-1
Hassan A. Munshi
SWCC RDTC
Jubail, 31751
Saudi Arabia

A: Chemical Disinfectant

(1) Chlorine and Chloramines

The use of reduced primary chlorination dosages and chlonamine be considered for disinfection if trihalomethanes (THM) reduction by alternative disinfection practices is desired.

The application of minimal dosages of chlorine and chloramines for primary disinfection should be evaluated on a long-term basis to guarantee adequate pretreatment disinfection and THM removal. Reduced chlorine dosages may be the only acceptable disinfection technique to reduce THMs since the Environmental Protection Agency is currently investigating toxicity of chloramines, chlorite and chlorate.

(2) Chlorine Dioxide

Further testing with chlorine dioxide be considered since chlorine dioxide has been shown to be unreactive to cellulose acetate membranes at concentration up to 1.2 g/m^3, while minimizing THM formation. Membrane element disinfection efficacity of chlorine dioxide should be closely examined because in adequate disinfection of spiral wound membrane elements has been reported despite application of high chlorine dioxide dosages to contaminated element feed water.

Chlorine dioxide (ClO_2) is a gas at normal temperature and pressure and exists as molecular ClO_2 in pure aqueous solution. Pure chlorine dioxide is unstable and can be an explosive mixture in air at approximately eleven percent concentration (4). For that reason, it is production-site at antility. This usually is

by reaction of sodium chlorite with excess chlorine in acid solution.

$$HOCl + H^+ + 2\ ClO_2^- \text{ --- } 2ClO_2 + H_2O + Cl^-$$

Although disproportionation of ClO_2 to for ClO_2^- and ClO_3 takes place. This reaction is much slower and, therefore, is less important from a chemical disinfectant stand point than the nearly immediate hydrolysis of molecular chlorine to hydrochlorite.

Chlorine is the most widely used disinfectant. Although it is not known exactly how chlorine destroys microorganisms, it is assumed that chlorine inhibits essential cell enzyme systems through oxidation.

Chlorine highly reactive and poisonous gas, is heavier than air and water-soluble. Dissolved in water, chlorine forms hypochloerous acid (HOCl), which in turn dissociated into only a hydrogen ion (H^+) and a hypochlorine ion (OCl^-).

The amount of Cl_2 plus $HOCl^-$ plus OCl^- in the water is known as increases its mass transfer rate from the gas to the liquid phase.

Chlorine dioxide does not react with the ammonia, but ammonia free waters do exhibit a demand for ClO_2 similar in magnitude to that for free chlorine.

Disinfection of Feed to Seawater RO Plants by Chloramine

Chlorination is a process where by chlorine in liquid for gaseous form (or in combination with other materials) is added to the system. The denaturing effect of chlorine on (animal or plant) microorganisms tissues forms the basis for its use as an effective water or waste water disinfectants. The disinfectant capabilities of chlorine depends on its chemical form in water which in turn is dependent on pH, temperature, organic contents of the water and other quality factors. When chlorine dissolves in water it hydrolyzes according to the reaction:

$$Cl_2 + H_2O = HOCl + H^+ + Cl^-$$

In dilute solution and at pH above 4, all chlorine will present in the form of HOCl or dissociated ions $H^+ + Cl^-$.

The hypochlorous acid HOCl is a weak acid and is dissociated to hydrogen and hypochlorite ions as follows:

$HOCl^- ==== H^+ + OCl^-$

This ratio between HOCl and OCl^- is the function of the pH, with 96 % HOCl remaining at pH 6, 75 % at pH 7, 22 % at pH 8 and 3 % at pH 9. The relationship of HOCl to pH is significant as the undissociated form appears to be the bactericidal agent in the use of chlorine for disinfection.

Sodium Bisulphate
SBS Method
$NaHSO_3$ Shock Treatment

The efficacy of $NaHSO_3$ as a biocide against marine microorganisms depends on exposure time, concentration of the $NaHSO_3$ and the types of microorganisms present. In general, the higher the concentration and the longer the exposure time, the greater will be the degree of disinfection. Using 30-minute exposure time, the effect of $NaHSO_3$ concentration of the degree of disinfection for sea water is given in Table 1. As can be seen from Table 1, a concentration of 50 mg/liter $NaHSO_3$ gives a very high degree of disinfection with a 30-minute exposure time.

Concentration of $NaHSO_3$ (mg/l)	%Kill[a,b]
200	96
500	99
750	99.9
1,000	99.9

[a]: Exposure time = 30 minutes

[b]: Serial dilution plating technique with Marine Agar

Shock treatment for seawater RO plants should be performed for at least 30 minutes at a $NaHSO_3$ concentration of at least 500m mg/l. However, the optimum dosage of exposure time must be determined for each site.

Since biological activity is site dependent, no absolute upper limit for the biological activity can be set with respect to the need for $NaHSO_3$ shock treatment. However, as a general guides if the RO feed and brine have less than 10^3 - 10^4 CFU/ml, significant biofouling would not be anticipated. Between 10^4 and about 10^6 CFU/ml, the degree of biofouling may effect the RO performance.

The shock treatment injection point should be located slightly upstream of the $NaHSO_3$ dechlorination point. Thus, when shock treatment is applied, all areas which have carried dechlorinated water will be disinfected by the shock treatment.

anticipated. At each site the biological activity must be monitored and correlated with the overall RO performance. This will establish a historical baseline at each site with respect to biological activity. Using the baseline data, day-to-day judgments can then be made concerning significant increase (100-fold)in biological activity and the need to shock treat with $NaHSO_3$.

UV Radiation Method

In this treatment system, water is disinfected by exposure to UV light at a specific intensity (dose rate) and wave length. In practice, water flows through a cylindrical stainless irradiation chamber known as a photoreactor.

In order to automate the UV disinfection process so that it operates effectively without supervision, a primary requirement is to continuously monitor the dose rate received by the feed water.

Copper Sulfate

The suitability and effectiveness of copper sulfate as a method of controlling plankton and algae for surface seawater RO systems are demonstrated by the successful operation of actual plants.

Four plants in addition to the Umm Lujj II plant are employing copper sulfate with Fluid Systems TFC elements. These are the 12,000 m^3/d plant in Jeddah, the 500 m^3/d Jeddah ship repair yard plant (1980), the 1,200 m^3/d Rosarito Beach, Mexico plant (1986), and the 10,000 m^3/d plant in Ras Tajura, Libya (1948). For these plants, there has been no appreciable biological fouling or growth of algae downstream of the point of copper sulfate injection.

Evaluate the Effectiveness of $NaHSO_3$

Samples of the RO feed and brine for microbiological analyses should be collected prior to and after the treatment. The results from the microbiological analyses will show the effectiveness of the $NaHSO_3$ shock treatment.

If the microscopic technique is used, the effectiveness of the shock treatment will be obtained within a couple of hours of sample collection. Culturing techniques will require several days before the effectiveness of the shock treatment can be determined.

Anaerobic and sulfate reducing bacteria are more resistant to $NaHSO_3$ than aerobic bacteria. RO plants are designed to avoid dead endsand stagnant area where anaerobic bacteria can thrive. If anaerobic bacteria become a problem, off-line disinfection and cleaning can be used to kill and remove them from the RO system.

Home Effective ISW Disinfection

No naturally occurring microorganisms or mutants have been found which can resist inactivation by UV. Bacteria, viruses, bacterilphages, molds, protozoa, yeasts, and algae are all susceptible and can be killed or inactivated if irradiated with UV at a sufficient intensity and suitable wave length for effective disinfection. The dose rate is vitally important. Unlike chemical disinfection, however, there are no drawbacks to overdosing with UV except that it wastes energy.

Effect of Ultraviolet Radiation on Various Microorganisms

Different microorganisms are affected differently by ultraviolet radiation. To demonstrate this, it is beneficial to review some data. Tobin, et. have documented the resistance of various microorganisms as shown in the Table.

Resistance of Different Microorganisms to UV Light

	40% Destruction				100% Destruction			
Bacteria								
legionella pueumephila	380 Micro watts-sec/cm^2				2,760 Micro watts-sec/cm^2			
E. Coli	3,000	-	-	-	6,600	-	-	-
P. aeruginesa	5,500	-	-	-	10,500	-	-	-
Spores								
Bacillus subtilis	12,000	-	-	-	22,000	-	-	-
Viruses								
Influenza	3,600	-	-	-	6,600	-	-	-
Tobaccomosic	240,000	-	-	-	440,000	-	-	-

When UV light impinge on a microorganism, it rearranges the DNA and RNA molecules of the microorganism. This blocks the microorganism's ability to replicate itself, and consequently, it ability to breed colonies. The peak region of germicidal effectiveness is the 240-260 nm UV wave length range. This is much shorter wave length than the UV light wave curve from the sun, which cuts off at about 290 nm.

Evaluation of the Efficiency of UV Disinfection System

* One problem that has been cited as serious disadvantage in the use of UV disinfection is the difficulty of measuring the UV dose. Unlike chlorination and ozonation, there is no measurable chemical residual. This makes the immediate control of the process difficult.

* Engineers choosing UV disinfection systems must usually rely on estimated of average dose based on insubstantiated estimates of intensity theoretical residence time.

* Most current UV disinfection systems employ tubular germicidal lamps enclosed in a second quartz tube submerged in a chamber through which the fluid flows.

Currently there are two major types of UV equipment. Low-pressure lamps and medium-pressure lamps being produced for the industrial market place. Due to the energy output differences of the lamps, the equipment designs are different. The low-pressure lamps system is designed as a multiple lamp system in the form of a tube bundle. The medium-pressure lamp system is designed using a single lamp in a probe form.

The low-pressure designs is used where absolute sterilization is required in such industries as pharmaceutical, food and beverage. Medium-pressure is well suited for all types of wastewater and right-turbidity applications.

Consideration into UV Materials:

Various factors affecting UV disinfection equipment must be taken into consideration when selecting or sizing a particular unit. Sizing application--as well as site--specific.

These factors must be considered for each application:

* Flow rate, both normal and peak demand

* Absorption coefficient (transmissionability) of the fluid being treated

* The type of organisms to be treated, and the associated energy levels required for the organisms

* Fluid composition, such as temperature, pressure, organic, inorganic, seasonal variation, scaling tendency, etc.

* Installation location (indoor, outdoor) and environment (wet, dry, fumes)

* Electrical power source available

* Type of operation (batch or continuous)

* Maintenance capability of on-site operators

Ultraviolet equipment is a tool that may be used to greatly enhance microbial control of a water treat. Size it for the task that it is to accomplish.

A Comparison of Chlorine, Ozone and UV

Yipand Korosewich compared UV with chlorine and ozone. Their Results are in the Figure

UV disinfection destroys common bacterial and viral pathogens more effectively than either chlorine or ozone. In figure, the dose required to destroy common pathogens is compared to E. Coli, which is the common indicate organism. It shows that UV destroys the other pathogens with essentially the same dose are required for E. Coli. Chlorine and ozone require much higher dose to destroy most of the other pathogens. A low indicator organism count using UV ensures destruction when using chlorine and ozone. The destruction of spores and cysts require much higher UV dosages.

Oxidation of organic is important in addition to disinfection. Here too, ozone excels with 52 percent higher oxidation power than chlorine and 38 percent higher than HOCl chlorine oxidation byproducts sometimes present water treatment problems, which are reduced by using ozone. Ultra violet light alone has no oxidation power. But it can catalyze ozone or hydrogen peroxide oxidation to form hydorxyl radicals, which have 105 percent higher oxidation power than chlorine.

References

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DISINFECTION

A comparison of chlorine, ozone and UV.

By James E. Cruver, Ph.D.

Disinfection, literally, means free from infection. A disinfectant destroys disease or other harmful microorganisms, but not necessarily unharmful ones or ordinary bacterial spores. The Environmental Protection Agency and World Health regulations define water disinfection by the absence of an indicator coliform bacteria group. On the other hand, sterilization implies complete destruction of all forms of life, which includes microorganisms. Most water we use is disinfected; medical and pharmaceutical applications require sterile water.

Disinfection techniques can be roughly classified into two types: chemical and physical. Popular chemical disinfectants for water include chlorine and other halogens, chlorine dioxide, chloramine, hydrogen peroxide and ozone. Physical disinfection methods include ultraviolet light, fine filtration, heat, ultrasound and ionizing radiation. The most widely used methods are chlorination (including chloramines), ozonation and ultraviolet disinfection. In this article, these three methods will be briefly described and compared.

Chlorine and ozone are both chemical disinfectants. In general, chemical disinfection is a compromise between the benefits of destroying microorganisms and the detriments of chemical toxicity and undesirable byproduct formation. Chemicals require time for reaction, and the degree and rate of reaction are strongly influenced by temperature and sometimes pH. The chemicals not only react with microorganisms, but also with other suspended and dissolved materials in water. Whereas some of these chemical reactions are desirable, such as oxidation of iron, other reactions yield undesirable byproducts.

Ultraviolet disinfection, a rapid physical process that causes molecular rearrangements, produces no undesirable byproducts. However neither does it provide residual killing power. Whether this limitation is a disadvantage depends on the particular application. Important process variables that affect UV performance include the concentration of suspended matter and the UV light absorbance of the fluid.

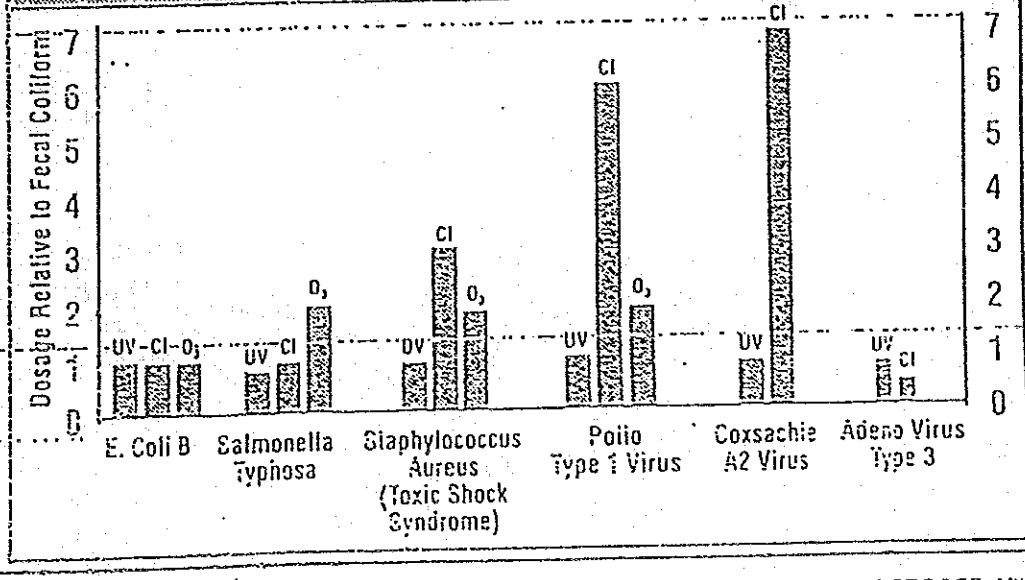
Chlorine

Chlorine is the most widely used water disinfectant. Although it is not known exactly how chlorine destroys microorganisms, it is assumed that chlorine inhibits essential cell enzyme systems through oxidation.

Chlorine, a highly reactive and poisonous gas, is heavier than air and water-soluble. Dissolved in water, chlorine forms hypochlorous acid (HOCl), which in turn dissociates into a hydrogen ion (H^+) and a hypochlorite ion (OCl^-).

The amount of Cl_2 plus HOCl plus OCl^- in the water is known as

Figure 1
Disinfection Dosage Relative to Fecal Coliform for Other Bacteria and Viruses



4. R2 添付資料（海水の前処理実験）

R２－１　海水淡水化装置の前処理装置 ---------- 4- 1

R２－３　油分濃度計操作方法
（堀場製作所　非分散型赤外線分析ＯＣＭＡ－２２０型
取扱説明書） ---------- 4- 11

R２－３　ジャーテスター操作方法
ＡＳＴＭ Designation : D 2035-80
Standard Practice for Coagulation-Flocculation
Jar Test of Water ---------- 4- 53

R２－４　データベースを用いた情報検索方法テキスト
Information Retrieval Using Databases ---------- 4- 57

R２－５　データベースを用いた情報検索実施結果 ---------- 4- 75

R２－６　凝集濾過補足実験 ---------- 4-115

R 2 － 1　海水淡水化装置の前処理装置

前処理設備の一般的な方式選定及び留意点等について、基本的な考え方を下記に記述する。

1.　前処理設備の設置目的

(ｱ) 海水中には溶解性塩類とは別に、微生物、微細砂、コロイド状物質等が存在これらが膜面に沈着したり、目詰まりを起すこと（Fouling）によって逆浸透モジュールの圧力損失の上昇、原水の偏流あるいは膜性能自体の低下をもたらす。

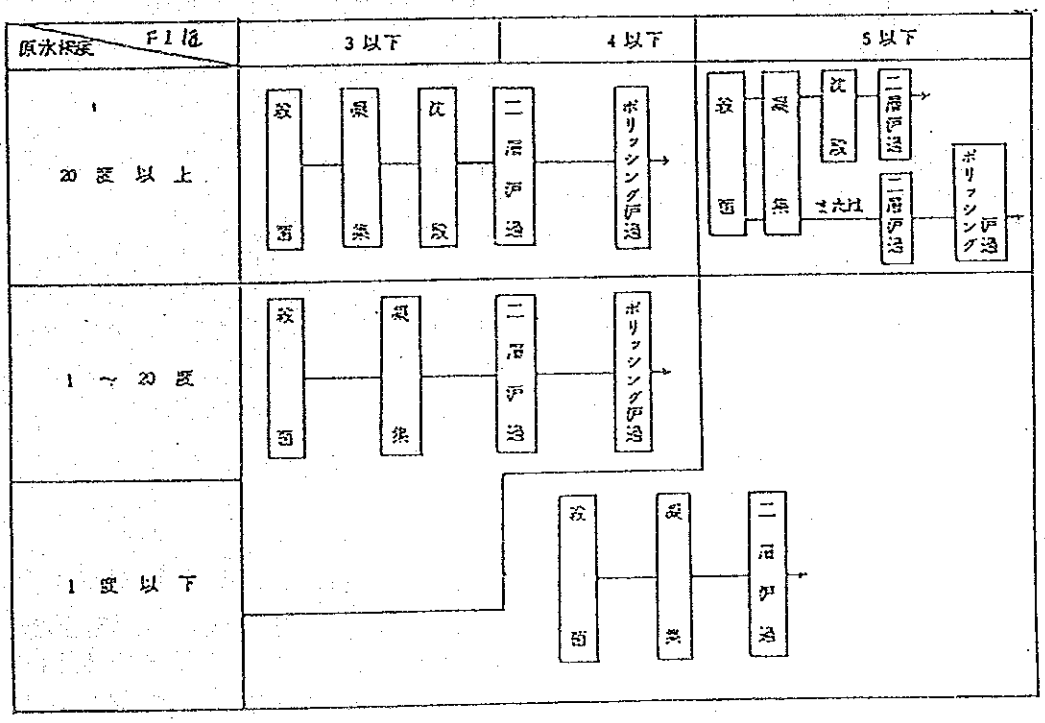
(ｲ) 海水はカルシウム及びマグネシウム硬度が高く、モジュール内で海水が濃縮されるとゝもにこれらが膜面に析出する　（Scaling）

等から膜面でのファウリング、スケーリング防止のため、前処理での濁質、硬度成分の除去を行う事を目的とする。

2.　前処理プロセス

(ｱ) 前処理プロセスの選定目安

表１．原海水及びFI値による選定目安の一例



・原海水の濁度劣化の原因としては
1)微生物の増殖（特に春季～夏季）
2)台風の接近や海のうねりによる海底の濁質のまきあげ（秋季）

3. 前処理設備の機能

（ア）懸濁物質の除去

・濁質が細かい粒子でコロイド状になっている場合、通常の砂ろ過だけでは捕集するのは難しいため

a)凝集沈殿法 ……・フロック形成池で原水に凝集剤を添加し粒径の大きいフロックを作り沈殿池で沈降スラッジとして引き抜く方法。

・大きな沈殿池が必要となり、経済的な問題で濁度の高い原海水の処理に採用。

b)凝集ろ過法 ……・配管中に凝集剤を注入、フロックを作り２層ろ過器で除去する方法。

・濁度が１～20度の範囲の原海水の処理に採用。

（イ）膜の化学的劣化の防止

a)酢酸セルローズ膜 ……・加水分解によって脱塩性能が低下、特にPH.7以上で加水分解の速度が大きくなる。

・硫酸注入によるPHコントロール

b)合成高分子膜 …… ・残留塩素による酸化分解による性能低下
（ポリアミド、ポリエーテル系）

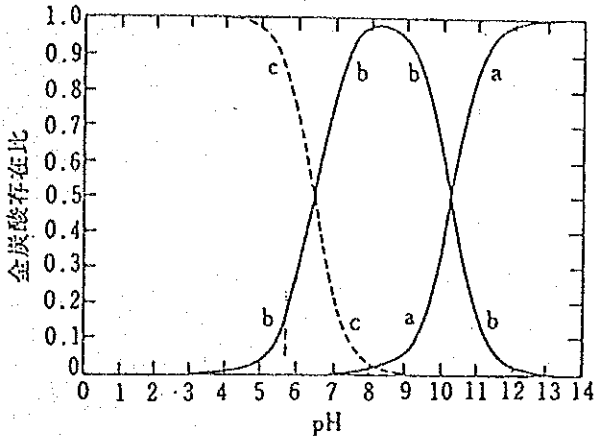
・脱塩素処理として、重亜硫酸ソーダ（SBS, NaHSO3), 亜硫酸ソーダ（Na2SO3）等の還元剤の注入
（NaHSO3+C12+H2O→NaHSO4+2HCL)

（ウ）スケーリング防止

a)海水中にはカルシウム、マグネシウム、鉄、マンガン等が含有されて居るが特にカルシウム、マグネシウムは約 400 mg/l, 1350 mg/l程度溶解され、逆浸透膜モジュール内での濃縮に伴って、析出が始まる。（特に、炭酸カルシウムは海水中の溶解度が低く約２倍程度の濃縮によって析出）

b)海水中では炭酸イオン（CO_3^{2-}），重炭酸イオン（HCO_3^-）で存在、
重炭酸カルシウムの溶解度は比較的大きいため膜面での析出は
起こり難い。

表．2　PHの変化と炭酸イオンの関係



曲線 a：CO_3^{2-}，b：HCO_3^-，c：H^2CO_3

c)硫酸等で原海水のＰＨ値を７．０以下にコントロールする事により
膜面でのスケール防止

（エ）スライムの防止

a)海水中に微生物が存在すると、膜面にフライムとして付着したり
酢酸セルローズ膜の場合、膜自体を侵食するとも言われ、
前処理での除去、滅菌が必要

b)酢酸セルローズ膜：　膜が塩素に対し比較的耐久性があるため、
モジュール内を適当な残留塩素濃度（0.2～0.5 mg/l）に保持して、
微生物の繁殖を抑える方法が一般的

c)高分子膜：　塩素による酸化分解を受けやすいため
滅菌のため注入した塩素をモジュールの直前で還え剤である
重亜硫酸ソーダ（SBS $NaHSO_4$ ）注入が必要

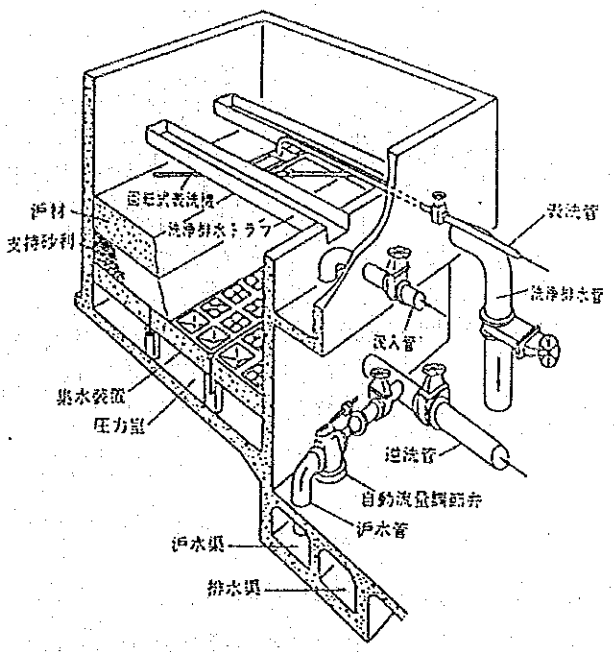
4.　ろ過器形式

（ア）ろ過器の選定目安

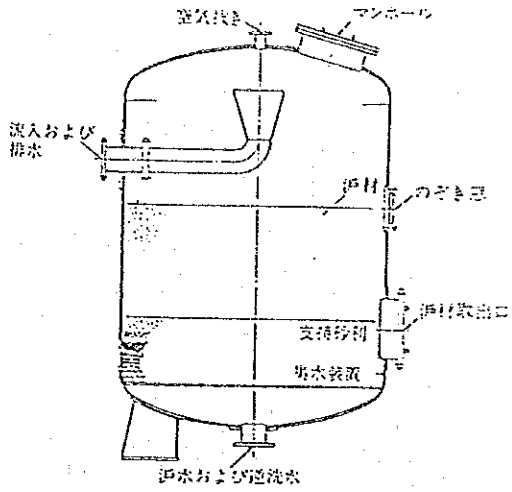
・プラントの全体スペース及びろ過器の性能等を考慮して選定
（表３．圧力式及び重力式ろ過器の比較参照）

表3　圧力式及び重力式ろ過器の比較

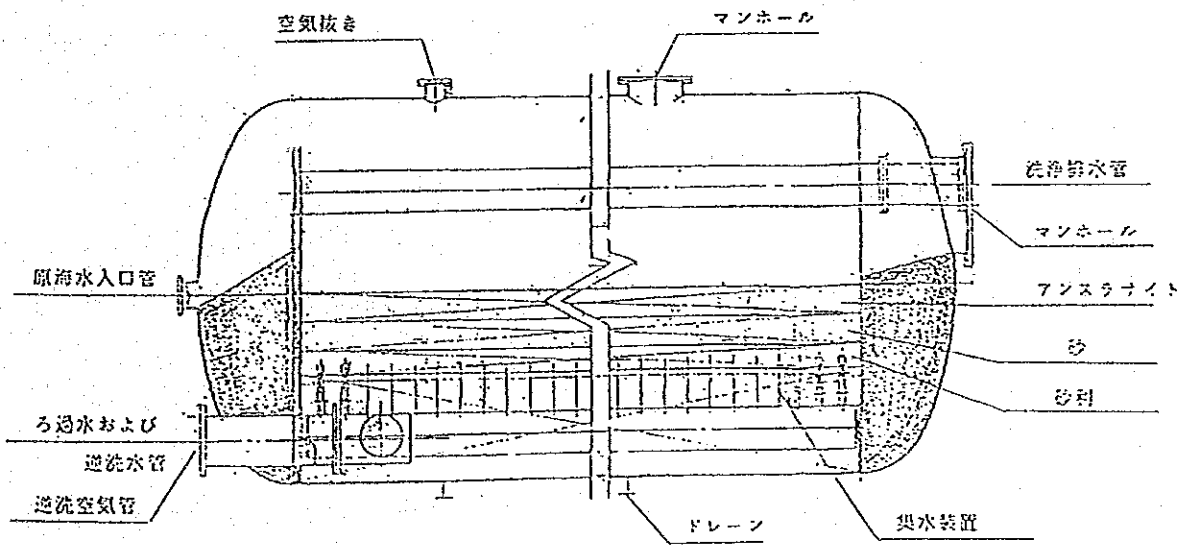
	圧力式二層ろ過器	重力式二層ろ過器
構造	横型，　円筒式	横型，　半地下式
材質	鋼製，ラバーライニング	コンクリート製
ろ過材	砂　＋　アンスラサイト	砂　＋　アンスラサイト
ろ過速度	8　～　15 m／時	5　～　8 m／時
圧力損失	8　～　10 m	2　～　3 m
ろ過面積	小	大
ろ過性能	やゝ良	良
逆洗回数	1回／日	1回／日
逆洗方式	空気　＋　水　＋　リンス	空気　＋　水　＋　リンス
逆洗速度	30　～　36 m／時	30　～　36 m／時
逆洗用水	ろ過水	ろ過水
メインテナンス性	やゝ複雑	簡単
特徴	1. ろ過速度が大きく据付面積が小さい 2. 逆洗用の所要空気及びろ過水量が少ない 3. 大容量のプラントでは圧力式ろ過器の寸法制限から基数が多くなる 4. 建設期間による分割納入が可能	1. ろ過速度が小さく据付面積が大きい 2. 逆洗用の所要空気及びろ過水量が多い 3. 消費動力が少ない 4. ろ過水の水質が良い 5. 一体構造物であり建設期間の分割が不可



重力式ろ過器の構造



竪型圧力式ろ過器の構造



横型圧力式二層ろ過器の構造

5. 薬品注入設備

(ア) 塩素滅菌

目的：塩素の殺菌効果により、原海水中の微生物、貝類（ムラサキイガイ、フジツボ）等の繁殖防止及び滅菌。

薬品種別：1)塩素ガス（液体塩素）

2)薬品（カルキ、次亜塩素酸ナトリウム（NaClO）、次亜塩素酸カルシウム（$Ca(ClO)_2$））

3)海水電気分解による次亜塩素酸ナトリウム

評価：

a) 1)塩素ガス注入及び2)薬品注入は設備コストは安いが、薬品の投入、ガスボンベの取り替え等の人手が掛かると共に、ガス又は薬品の購入コストも高い。

b) 3)海水電気分解による次亜塩素酸ナトリウムの使用には塩素発生装置を設置する必要があり、初期の設備コストは高いが、ガス又は薬品の注入に比較して、保守管理が容易で、運転コストが安い。

特に、大型プラントの場合には塩素発生装置を採用するのが一般的である。

(イ) 凝集剤

目的：海水中に含まれるコロイド状の極微少な汚濁物を凝集剤を注入することにより、凝集させ、ろ過器で除去。

薬品種別：1)アルミニウム塩系

2)鉄塩系

評価：

海水処理の凝集剤としては、鉄塩系（塩化第二鉄（$FeCl_3$）等）が有利である。

（6．　凝集剤の特徴比較　参照）

(ウ) pHコントロール

目的：原海水のpH値は約8.2 程度であり、膜の加水分解の防止及び炭酸カルシウム等の膜面へのスケール付着防止のため、pH6.0～6.8に調整する。

薬品種別：1)硫酸（H_2SO_4）

2)塩酸（HCl）

評価：機能的には硫酸又は塩酸のどちらを使っても問題ないが、取扱易さ、コストの面から硫酸が一般に使用されている。

6. 凝集剤の特徴比較

1 凝集効果

水処理に使用される凝集剤として代表的な鉄塩系とアルミニウム塩との一般的な特徴比較を下表に示す。
海水のＰＨ値（ＰＨ＝８．２程度）より考慮してＰＨ範囲の大きな鉄塩系の凝集剤が海水の凝集剤としては有利である。

処理原水のＰＨ値の大小による鉄塩，アルミニウム塩の凝集効果の詳しい比較を次図に示すが鉄塩系の凝集剤が，ＰＨ値８近辺ではアルミニウム塩と比較して，良好な凝集効果をしめす。

表－１　Fe塩・Al塩・Ca塩の特性比較

優位性	特性事項＼凝集剤の種類	硫酸第二鉄	硫酸アルミニウム
○	加水分解生成物溶解度(g／100ml)	4.8×10^{-9}(18℃)	1.5×10^{-4}(20℃)
○	加水分解定数	2.5×10^{-3}	1.05×10^{-5}
○	溶解度積（MPO_4）	1.3×10^{-22}	3.9×10^{-11}
○	水酸化物比重	3.3～3.9	2.42
○	pH範囲	4.0～11	6.0～8.5
	最適pH範囲	6.1～7.4	6.0
○	薬剤注入率	小	中
○	スラッジ圧縮性	良い	悪い
○	〃　脱水性	良好	普通
○	底泥からのリンの溶脱性	最も少ない	－
○	リンとの親和性	最も大きい	－
○	処理コスト	少ない	普通

鉄系凝集剤の長所

イ）フロック（水酸化物）の生成が早く，重いので沈降速度が大きい。
ロ）フロック（水酸化物）の圧縮性が大きい。
ハ）適正ＰＨ範囲がひろい。
ニ）スラッジの脱水性がよい。
ホ）一度沈殿した水酸化物は再溶解しにくい。
ヘ）ランニングコストが比較的廉価。

河川水におけるＡｌ塩，Ｆｅ塩の凝集ＰＨ範囲の比較例

テスト方法

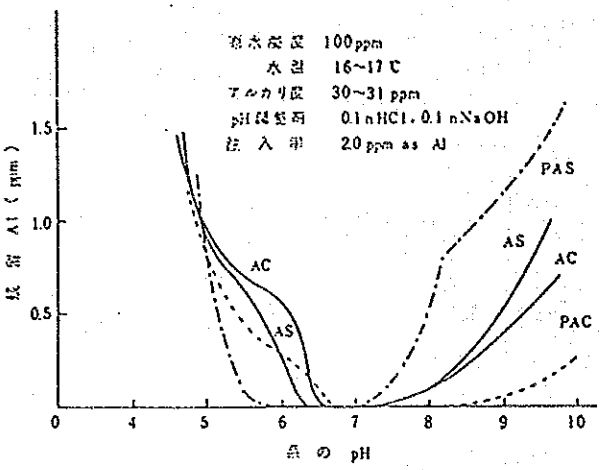
ジャーテスト

急速攪拌	１００ｒｐｍ	１分
緩速攪拌	６０ｒｐｍ	１０分
静置時間		５分

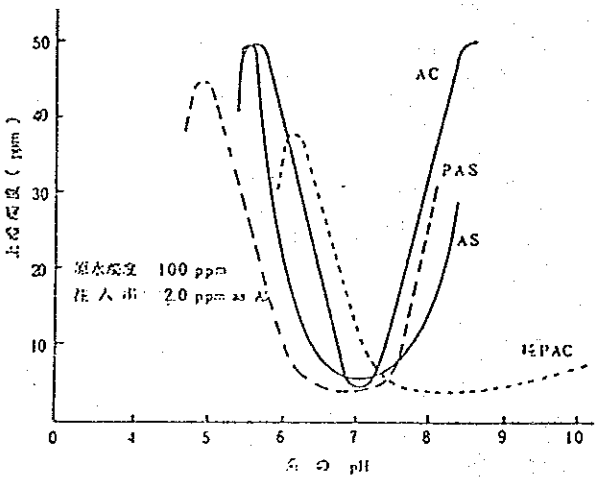
静置後の上澄濁度，残留成分を定量した結果をしめす。

Ａｌ塩の凝集ＰＨ範囲はＰＡＳ（ポリ硫酸アルミニウム）でＰＨ６－７，ＰＡＣでＰＨ７－８付近となる。　一方Ｆｅ塩の凝集ＰＨ範囲は塩種にかかわりなくＰＨ６－８付近であり，Ｆｅ塩はＡｌ塩と比較して広いＰＨ範囲を有する。

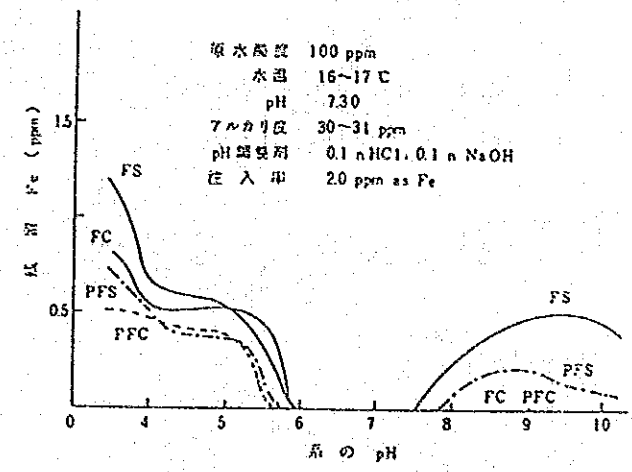
除濁効果も一般にＡｌ塩が中性域をはずれると極端に悪化するが，Ｆｅ塩は広いＰＨ範囲において良好な除濁効果を有する。



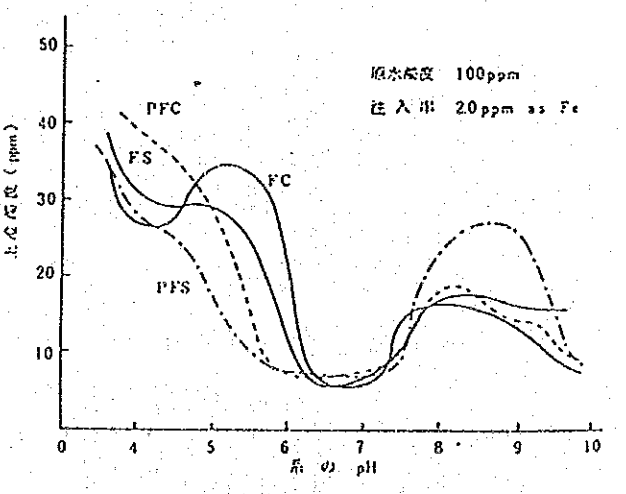
図－５　河川水におけるAl塩の凝集pH範囲（1）



図－６　河川水におけるAl塩の凝集pH範囲（2）



図－７　河川水におけるFe(Ⅲ)塩の凝集pH範囲（1）

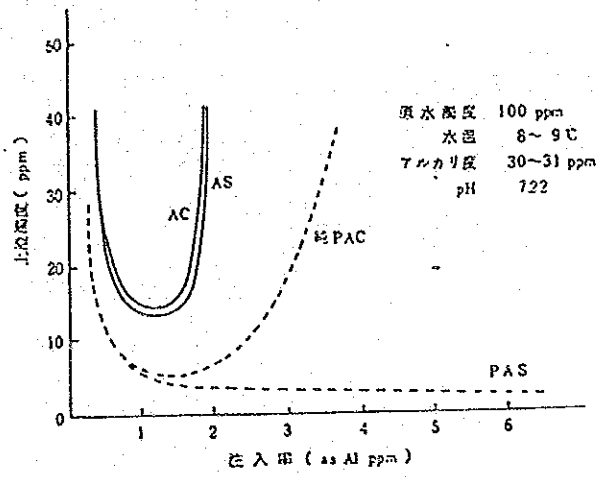


図－８　河川水におけるFe(Ⅲ)塩の凝集pH範囲（2）

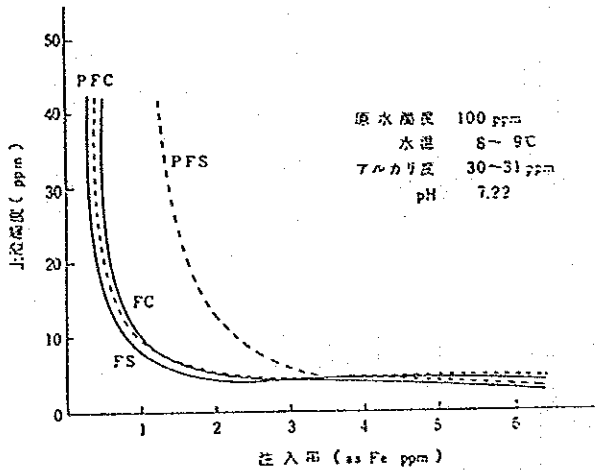
2　実用性

凝集剤としての評価判定は，凝集効果のみならず適正注入率の許容幅のひろさ凝集処理作業の安全上重要となる。

次図にアルミニウム塩と鉄塩の注入率の大小による凝集効果の比較例を示すが鉄塩系の凝集剤は広い注入範囲において安定した凝集効果をしめしている。



図－1　Al正塩と塩基性塩の凝集効果の比較



図－2　Fe(Ⅲ)正塩と塩基性塩の凝集効果の比較

3　鉄塩凝集剤の使用上の注意

1）酸性が強く，腐食性が強いため，機械，装置の材質に注意が必要。

2）凝集・逆洗水等の廃水処理が不可欠。

3）凝集剤原液の取り扱いに注意が必要。

4　凝集剤の注入率の目安

凝集剤として塩化第２鉄（ＦｅＣｌ３）を使用し，添加濃度を変化させた場合の処理水ＦＩ値　，濾過器圧力損失の計測例を次図に示す。

Ｆｅ濃度０．５ｍｇ／ｌ程度の添加で処理水ＦＩ値は４以下となり，注入濃度を高めることにより，さらに除濁効果はよくなるが除濁とともに濾過器圧力損失が増加する。ＲＯ設備の前処理としては，１．０ｍｇ／ｌ程度の添加濃度が最適と思われる。

凝集剤の注入による効果の一例

凝集剤注入なし	処理水質	処理水のＦＩ値４．５－６．２であり，原海水のそれとほとんど同じでろ過されていない。
	ろ過器差圧	０．０１ｋｇ／ｃｍ２　／日　以下
０．５　ｍｇ／ｌ（ａｓＦｅ）凝集剤注入	処理水質	処理水のＦＩ値は通水初期の４．８－３．５より最終的に２．５以下に安定する。通水後５－６時間を除けばＦＩ値で２．５－３となっている。
	ろ過器差圧	０．３５ｋｇ／ｃｍ２　／日　以下
１．４　ｍｇ／ｌ（ａｓＦｅ）凝集剤注入	処理水質	処理水のＦＩ値は通水初期の４．０－３．０より最終的に２．０以下に安定する。通水後５－６時間を除けばＦＩ値で２．０－２．５となっている。
	ろ過器差圧	０．６ｋｇ／ｃｍ２　／日　以下

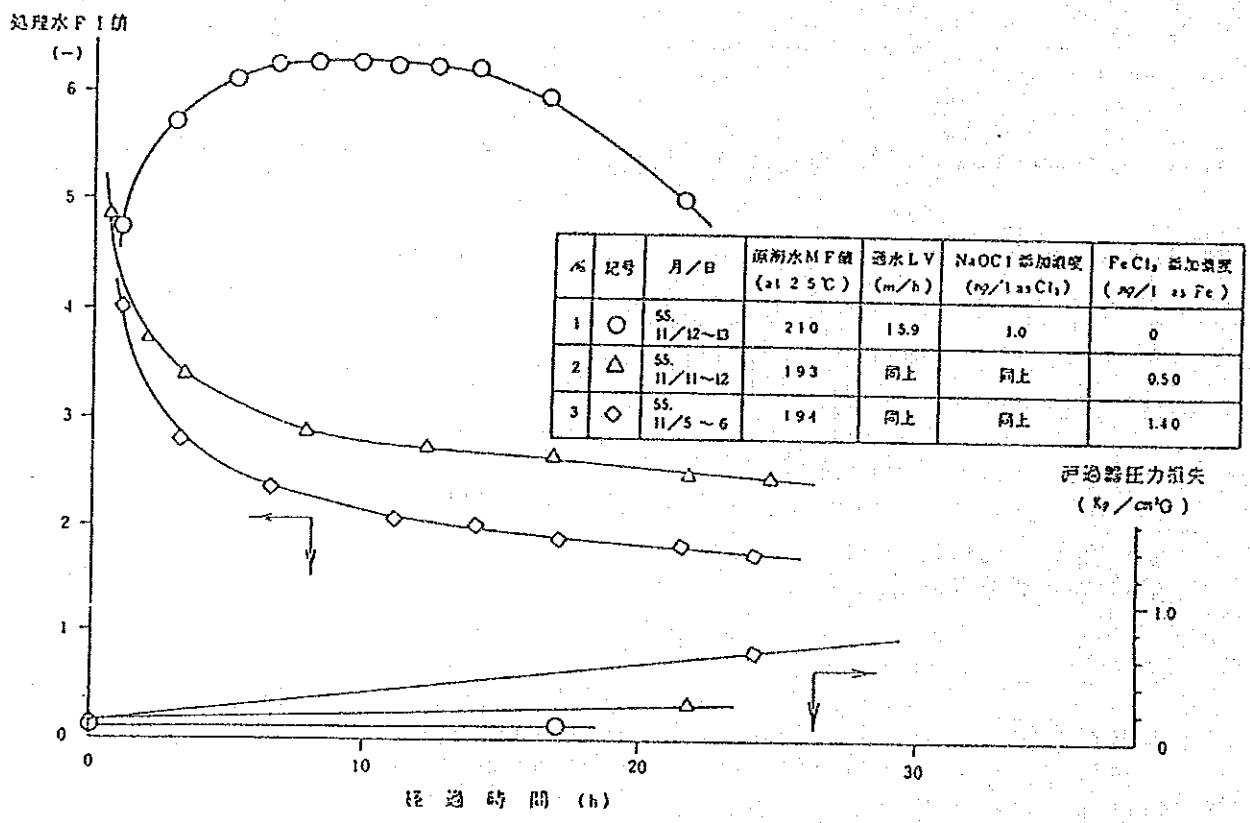


図 3.2.4　凝集剤（$FeCl_3$）添加濃度変化による処理水ＦＩ値の経時変化

R２－２　油分濃度計操作方

掘場製作所　非分散型赤外線分析ＯＣＭ－２２０型

取扱説明書

Instruction Manual

OCMA-220

Oil Content Analyzer

Horiba, Ltd.

Kyoto, Japan

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1. Description

The OCMA-220 is designed to measure quickly and precisely, the organic hydrocarbon contamination in fresh or salt water samples. The hydrocarbons may be present in the sample water in the form of oils, fats, greases or waxes.
A nondispersive infrared (NDIR) analyzer is used to measure the concentration of hydrocarbons in the solvent solution. This technique closely approximates several standard methods in which the oil is extracted with a solvent, which is then analyzed by infrared spectrometry or weighed.
The OCMA-220 is a portable, precise instrument ideally suited for quickly analyzing the oil content of ballast and bilge water and the hydrocarbon contamination in industrial plant waste water effluent or in rivers, lakes and oceans.

The instrument consists of two sections; a sampling handling section, and an analyzer section. Both sections are contained in one portable housing. The oil content is read directly in ppm oil on a digital panel meter.
All accessories necessary for the operation of the OCMA-220 are included in the shipping kit except the extraction solvent which must be obtained by the operator. The ship kit includes:

20mℓ syringe for sample	1
20mℓ syringe for solvent	1
25μℓ microsyringe for calibration	1
Heavy oil for calibration (10mℓ bottle)	1
Water/oil filters	5
200mℓ beakers	4
Fuse	1
Power cord (2.4m)	1
Philips screw driver	1
Instruction manual	1
Instruction label	1

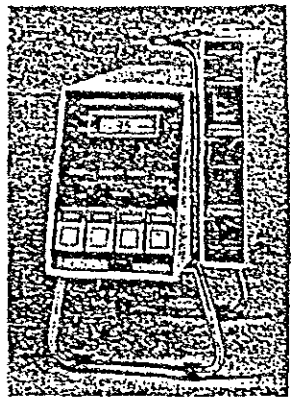
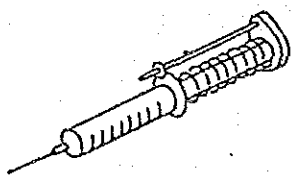
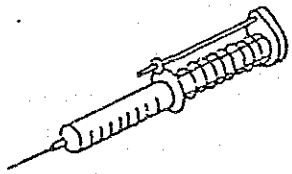
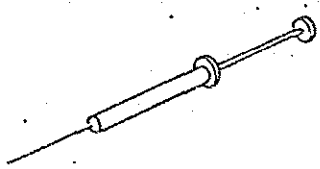
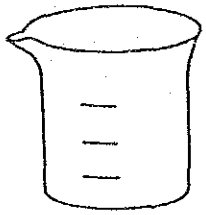
2. Specifications

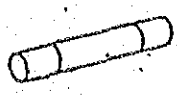
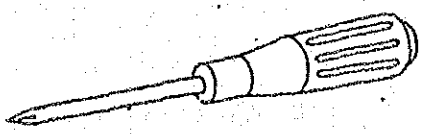
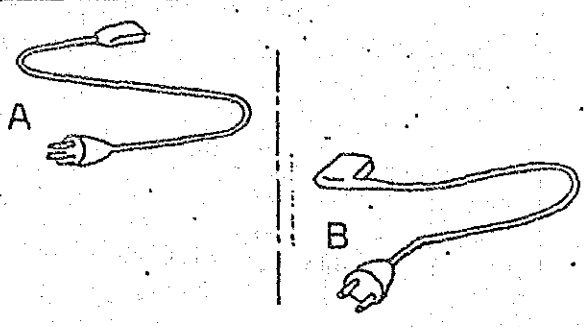
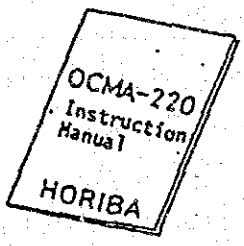
2.1 Specifications

Principle	:	Solvent extraction, NDIR analysis
Ranges	:	0∿5, 0∿20 ppm (dual ranges)
Measuring object	:	Organic hydrocarbon contamination in fresh or salt water sample
Data display	:	Liquid crystals, 3-digit panel meter
Repeatability	:	0∿20 ppm: ±2% of full scale ±1 digit 0∿5 ppm : ±4% of full scale ±1 digit
Calibration	:	Known oil/solvent mixture for span calibration; pure solvent for zero
Sample volume	:	(A) 0∿20 ppm: 15mℓ per measurement (B) 0∿5 ppm : 20mℓ per measurement
Solvent	:	Flon S-316 (Fluorochlorocarbon)**
Solvent volume	:	(A) 0∿20 ppm: 15mℓ per sample (B) 0∿5 ppm : 10mℓ per sample
Extraction	:	By built-in extractor * Externally extracted samples may also be measured.
Ambient temperature	:	0∿40°C
Output	:	0∿100mV DC
Power requirement	:	AC line voltage, 50 or 60Hz to be specified, approx. 50VA
Dimensions	:	220(W) × 362(D) × 375(H)mm [8.7(W) × 14.3(D) × 14.8(H) inch]
Weight	:	Approx. 10kg (22 lb)

** Other kind of solvent such as carbon tetrachloride may be used, but the analyzer must be adjusted (including optical alignment) for proper operation.

2.2 Contents of standard package

	Item	Q'ty	Appearance
1	OCMA-220	1	
2	Syringe for solvent (20mℓ)	1	
3	Syringe for sample (20mℓ)	1	
4	Microsyringe for calibration (25μℓ)	1	
5	Glass beaker (200mℓ)	4	
6	Heavy oil calibration (10mℓ)	1	
7	Water/oil filter	5	

	Item	Q'ty	Appearance
8	Fuse For 100~115V AC area: 1A For 220~240V AC area: 0.5A	1	
9	Phillips screw driver	1	
10	Power cord For North America: A For Europe : B	1	
11	Instruction manual	1	
12	Instruction label	1	

3. Principle of Operation

3.1 General

Oil is generally a mixture of many different hydrocarbon compounds which exhibit widely diverse chemical properties. Because any two oil samples may contain different concentrations of these hydrocarbon compounds, the results of an analysis of water samples for oil content may depend upon the method of analysis used.

Two distinct characteristics common to most oils are: (1) they are composed of hydrocarbons, and (2) they are insoluble in water. The OCMA-220 utilizes these two characteristics to allow an accurate measurement of oil in water regardless of the type of composition of the oil in the sample.

The hydrocarbon compounds contain CH radicals. Each of these radicals exhibits a very distinct energy absorption band in the range of 3.4 to 3.5 microns in the infrared spectrum (refer to Fig. 3-1). This absorption band is almost identical for any type of oil. Consequently, when the infrared absorption of an oil sample is measured between 3.4 and 3.5 microns, the absorptivity varies in direct proportion to the concentration of oil in the sample.

Because water also absorbs energy in the infrared ban between 3.4 and 3.5 microns, it is virtually impossible to measure low concentrations of oil in a water sample. Therefore, the oil dispersed in the water must be separated from the water before it can be accurately measured.

Halogenated solvents may be used to separate the oil from the water for subsequent infrared analysis because: (1) these solvents are almost insoluble in water, (2) they have a specific gravity heavier than water, (3) they readily dissolve all volatile or nonvolatile organic compounds, and (4) they do not absorb infrared energy in the 2 to 4.5 micron band (refer to Fig. 3-1).

This method provides a number of advantages over other methods used for the analysis of oil and grease in water. These include preservation of volatile components in the sample, specificity to hydrocarbons, lack of interference from suspended solids and colored substances, and good precision.

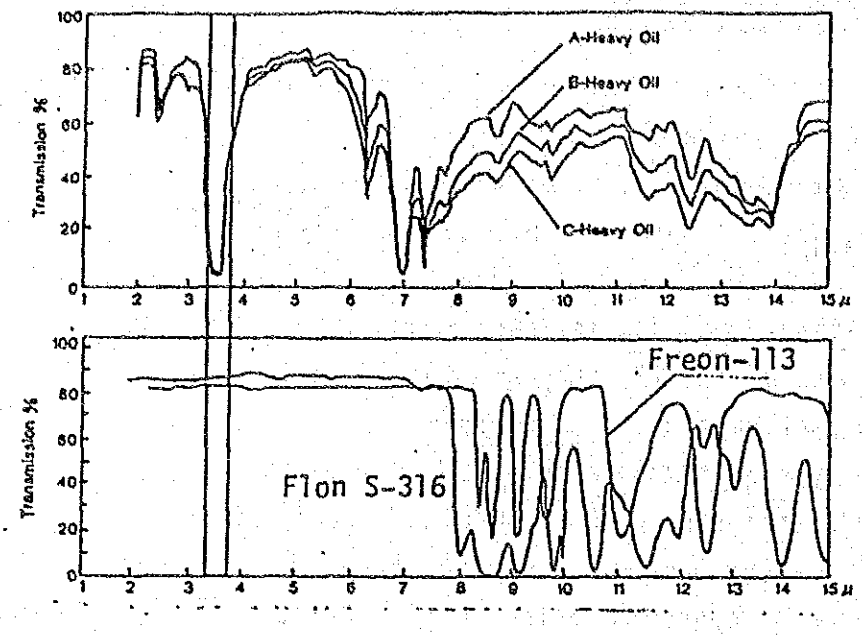


Fig. 3.1 Infrared Ray Spectrum of Heavy Oils and Flon S-316

3.2 Sample handling section

The sample handling section of the OCMA-220 is a compact, efficient solvent extraction system which separates the oil from the water in the sample to be analyzed. The basic steps in the extraction process are as follows.

The operator measures xmℓ of hydrocarbon-contaminated water with a syringe and injects the sample into the extractor chamber of the OCMA-220. The operator then adds ymℓ of solvent.

The sample and solvent are mixed together for about one minute by the built-in agitator in the extractor chamber. After the sample is mixed, it is allowed to set for approximately one minute to allow the solvent, which now contains the oil, to settle to the lower portion of the chamber.

After the solvent has separated from the water, a valve is opened manually to allow the solvent and oil to flow into the analyzer sample cell through a filter membrane. This filter prevents water the solids from entering the sample cell and causing an error in the analysis. After the sample cell has been filled with the solvent/oil mixture, the measure switch is depressed and the reading in ppm oil is taken from the digital panel meter directly. After the meter has been read the measure button is turned off and the discharge valve on the extractor is opened to allow the system to drain.

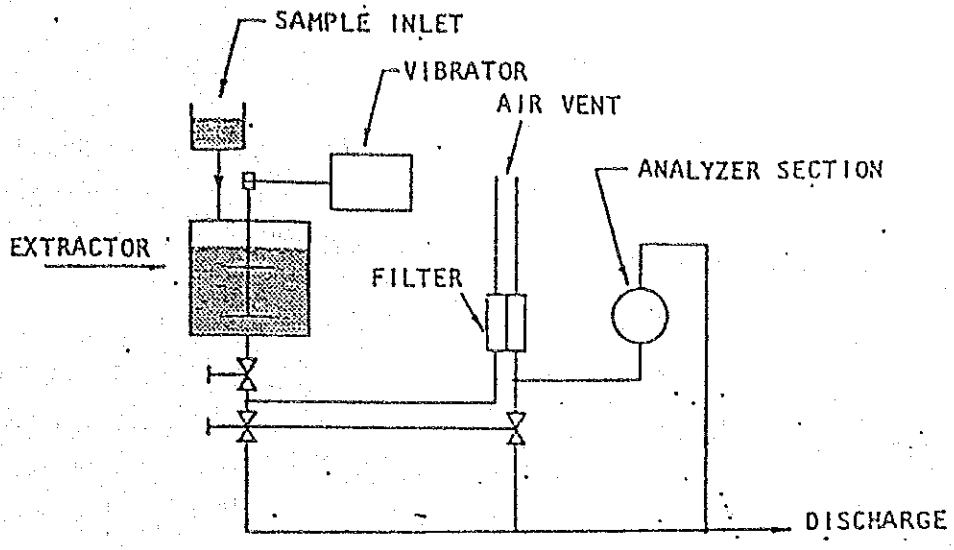


Fig. 3-2 Flow Diagram; OCMA-220

3.3 Analyzer section

The analyzer section of the OCMA-220 provides the stability and accuracy inherent in a differential measurement based on the principle of infrared energy absorption. The instrument utilizes a double-beam optical system which measures the amount of oils in a sample cell and compares that value to the response from a cell filled with reference gas. A simplified functional diagram is shown in Fig. 3-3.

Two infrared sources are used, one for the sample energy-beam, the other for the reference energy-beam. The beams are blocked ten times per second by the light chopper, a two-segmented blade rotating at five revolutions per second. In the unblocked condition, each beam passes through the associated cell and into the detector.

The sample cell is a flow-through tube that receives the liquid sample to be measured from the sample handling section of the instrument. The reference cell is a sealed tube filled with a reference gas. This gas is selected for minimal absorption of infrared energy of those wavelengths absorbed by the sample component of interest.

The detector consists of two sealed compartments separated by a flexible metal diaphragm. Each compartment has a window of a synthetic crystal to permit entry of the corresponding energy beam. Both chambers are filled to the same subatmospheric pressure with HC vapor. Use of this substance as the gas charge in the detector causes the instrument to respond only to that portion of net difference in energy due to the presence of the measured component.

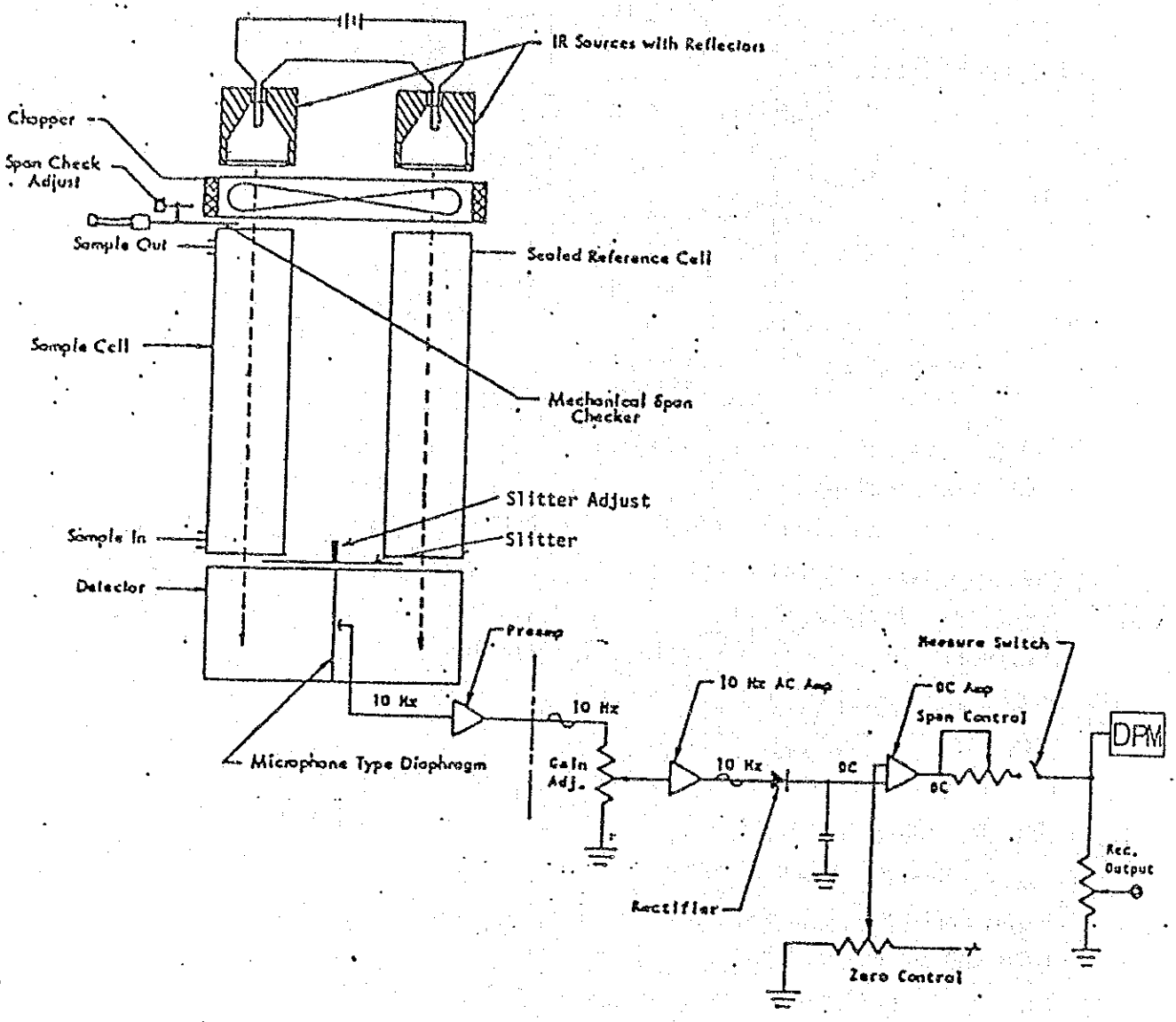


Fig. 3-3 Analyzer and Amplifier Diagram

In operation, the presence of the infrared-absorbing HC in the sample stream causes a difference in energy levels between the sample and reference sides of the system.
This differential energy increment undergoes the following sequence of transformations:

1. Radiant energy: In the sample cell, part of the original energy of the sample beam is absorbed by the HC. In the reference cell, however, absorption of energy from the reference beam is negligible.
2. Temperature: Inside the detector, each beam heats the gas in the corresponding chamber. Gas in reference chamber is heated more, however, since energy of the reference beam is greater.
3. Pressure: Higher temperature of gas in the reference chamber raises pressure of this compartment above that of the sample chamber.
4. Mechanical energy: Gas pressure in reference chamber distends diaphragm toward sample chamber. The energy increment is thus expended in flexing the diaphragm.
5. Capacitance: The diaphragm and the adjacent stationary metal button constitutes a two-plate variable capacitor. Distention of the diaphragm away from the button decreases the capacitance.

When the chopper blocks the beams, pressures in the two chambers equalize and the diaphragm returns to its undistended position. As the chopper alternately blocks and unblocks the beams, the diaphragm pulsates thus changing detector capacitance cyclically. This changing of the detector capacitance at a 10Hz rate is converted to an electrical signal and is routed to the preamplifier which is mounted on the detector. (Refer to Fig. 3-3.)
The preamplifier converts the signal which is very small and of a high impedance into a larger signal of low impedance. The signal is then routed to the first stage of the amplifier through the Gain Control Potentiometer. This potentiometer adjusts the gain of the overall system by attenuating amplifier input signal appropriately. The resultant attenuated signal is fed into the input of the AC operational amplifier where it is amplified. The output from the AC amplifier is then rectified and filtered prior to being sent into the DC amplifier. The DC amplifier then drives the output meter an amount directly proportional to the concentration of oils in the sample so that the concentration of oils is read directly from the front panel meter in ppm oil.
A metal "slitter" is provided in the optical system to allow the IR radiation in either the sample or the reference cell to be partially blocked by a manually adjustable amount. With the slitter centered so as to block no IR radiation

from either IR source element and with a zero standard in the sample cell, the IR intensities in each cell would ideally be equal. Due to small variations in IR sources, cell wall reflectivity and optical window transmissive properties, these intensities will likely be slightly in equal resulting in an output signal from the detector and an upscale reading at the meter. In adjusting the slitter, the sample and reference intensities are first balanced, resulting in a minimum output signal. The slitter is then moved slightly so as to block slightly more sample cell radiation (or slightly less reference side radiation). This imbalance results in an upscale signal which is compensated for electrically. Electrical zero biasing is provided by the zero adjust network shown in Fig. 3-3. It applies an adjustable zero-biasing signal to the input of the DC amplifier.

A recorder output is provided by means of an adjusting potentiometer to set the output at 0∿100mV for a full scale reading.

4. Installation

4.1 Location

To insure proper performance of the instrument, choose a location that will meet the following conditions as closely as possible.

1. Area should be free from excessive dust or moisture exceeding 80% R.H.
2. Area should be free from corrosive gases.
3. Location should not be subject to shock or vibration.
4. The instrument should not be subjected to direct sunlight or radiant heat, and rapid ambient temperature fluctuations should be avoided. Environmental temperature range should be maintained between 32°F (0°C) and 104°F (40°C).
5. Allow for adequate air circulation around the analyzer.
6. Since the analyzer section and the electronics cannot be air-purged, keep the instrument away from combustible gases.
7. Sufficient ventilation must be provided to prevent breathing of carbon tetrachloride vapor (or excessive breathing of Flon S-316 vapor). A fume hood is the desired location for the analyzer.
8. DO NOT install the instrument near electrical equipment which causes power source disturbances (radio frequency, furnaces, electric welders, etc.), and DO NOT connect the instrument's power line to the same power source used by such equipment.
9. Make the proper grounding. Be sure to locate the ground away from any potential fluctuation which could be caused by an electric heavy device.
10. Solvent may vary depending on production lot in its indication level (zero point). Hence solvents which were produced in different lots are to be used, pour expected amount of solvents to be used into a clean glass container for equalization of zero point level. Apply same zero point solvents both for span and measurement.

4.2 Power source

4.2.1 Power requirements

The power source required for the instrument is 115V (105V-125V) AC with a frequency of 60 or 50Hz. The actual frequency must be specified. (240V AC power is optional.)

4.2.2 Power cord

A 2.4 meters (eight-foot) power cord, equipped with a three-prong plug (for 115V version) or European plug (for 240V version) is provided with the OCMA-220. Before connecting power cord to the power outlet make sure all power switches are in the "OFF" position.

4.3 Output

The analysis can be recorded on a 0∿100mV potentiometric recorder by means of the recorder output jacks located on the back of the analyzer.

4.4 Grounding

When 3P power outlet with a grounding terminal is available as power source, the grounding terminal on the back panel need not be connected. With 2P power outlet (without grounding terminal), however, the grounding terminal must be connected to the earth through grounding wire. Grounding work must be provided in compliance with a required standard. If such grounding work is impracticable, connect the grounding terminal to a properly grounded water pipe or a steel frame

Caution
- ° Water piping containing vinyl chloride pipe cannot be used for grounding electrode.
- ° Do not use gas piping for grounding electrode.
- ° Use AWG20 or thicker wire for grounding wire.

5. Operating Controls and Adjustments

① Digital display

Digitally displays oil concentration. The display flickers for the concentration above full scale. The selected range is indicated by the mark ◄.

② Check button

The check button controls a preset mechanical device which partially blocks the sample beam and allows the operator to make a quick check on the overall sensitivity of the analyzer without having to use standard samples.

③ Span adjust

The span adjust is used to set the upscale standardization point. With a standard sample in the sample cell the span adjust is set for correct meter reading.

④ Zero adjust

The zero adjust is used to set the zero point (downscale standardization point). With a blank sample (pure solvent) in the sample cell, the zero adjust is set for a zero reading on the meter.

⑤ Power button

The power button controls the electronic circuitry. The OFF (down) position removes all power from the analyzer and the sample handling.

⑥ Range selector

Selects measuring range (5 ppm or 20 ppm). (Full scale output and range lamp are switched.)

⑦ Measure button

The measure button controls the output and meter circuits to prevent damage to the meter when changing samples or filling the sample cell.
After the cell has been filled with zero or standard solution or sample, the measure switch is switched to the ON position to take the reading. After completion of the reading the measure button is turned OFF.

⑧ Sample inlet

Sample and extraction solvent are injected through this inlet.

⑨ Range lamp (5 ppm)

Lights when 5 ppm range is selected. Confirm sample volume (15mℓ) and extraction solvent volume (10mℓ) before injection.

⑩ Range lamp (20 ppm)
Lights when 20 ppm range is selected. Confirm sample volume (15mℓ) and extraction solvent volume (15mℓ) before injection.

⑪ Extractor chamber
Oil is extracted from sample into the solvent here.

⑫ Extractor valve
The extractor valve controls the flow of sample from the extraction chamber to the analysis cell; the valve is left in the closed position until the extraction process has been completed. The valve is then placed in the open position for the balance of the analysis and the draining of the system.

⑬ Extraction timer
This is for setting extraction time.

⑭ Discharge valve
The discharge valve is used to drain the sample from the analyzer after completion of analysis.

⑮ Discharge outlet
When solution overflows or when discharge valve is set to open it drains through this outlet.

⑯ Extractor button
This button activates the agitator in the extractor chamber (visible through the Monitor Window). When the extractor switch is turned ON, the agitator is activated and the extractor lamp is lit. The agitator is controlled by a timer and will continue to operate for 40 seconds, 1, 2, 3, 4 or 5 minutes after which time the indicating lamp will go out and the agitator will stop.

⑰ Recorder output adjust control
This is for controlling full scale output of instrument.

⑱ Recorder output terminal
DC voltage output (full scale 0.1V)

⑲ Grounding terminal
Terminal for grounding

⑳ Fuse
Power fuse.

㉑ Power inlet

Connect supplied power cord to this inlet.

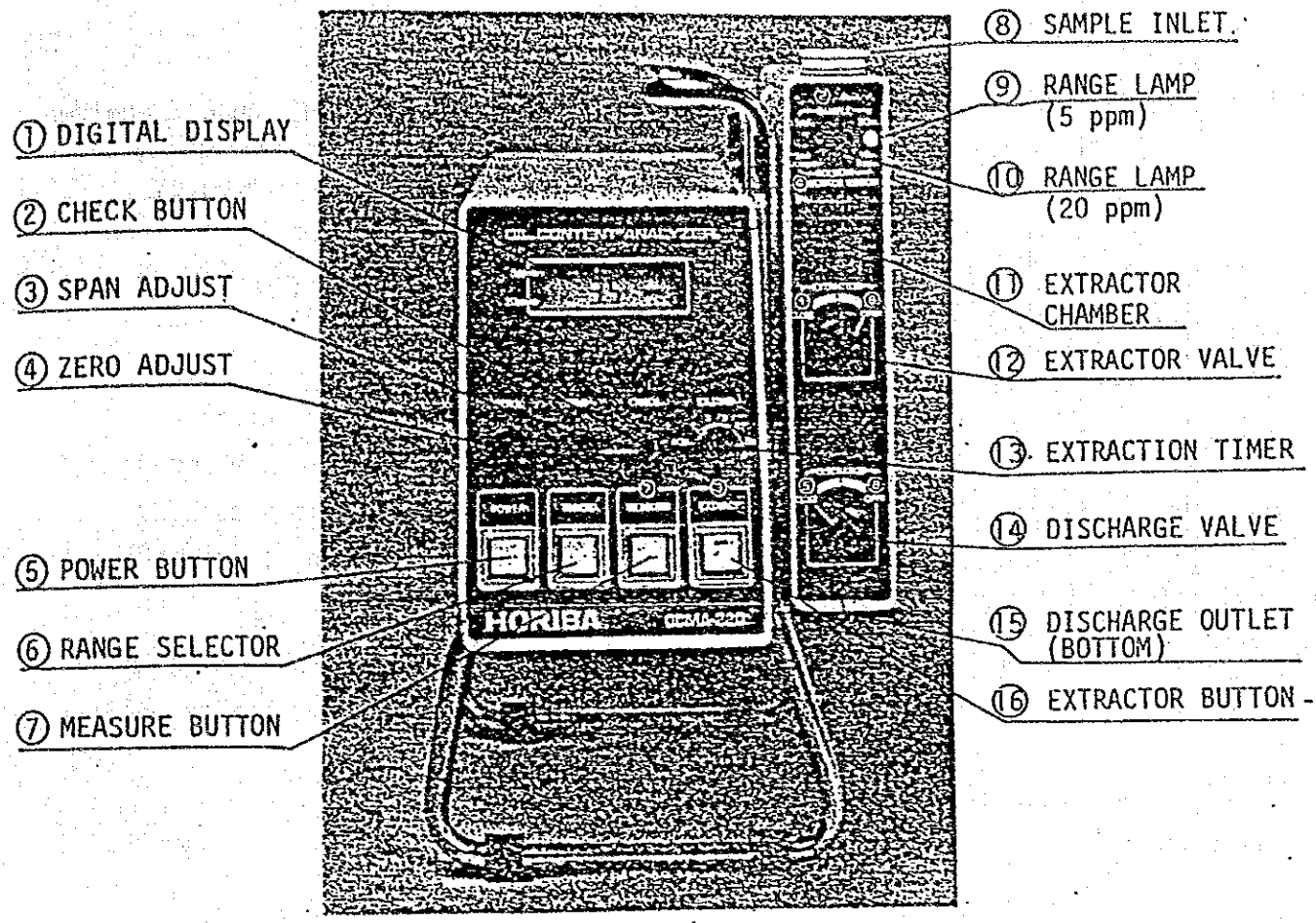


Fig. 5-1 Location of Operating Controls

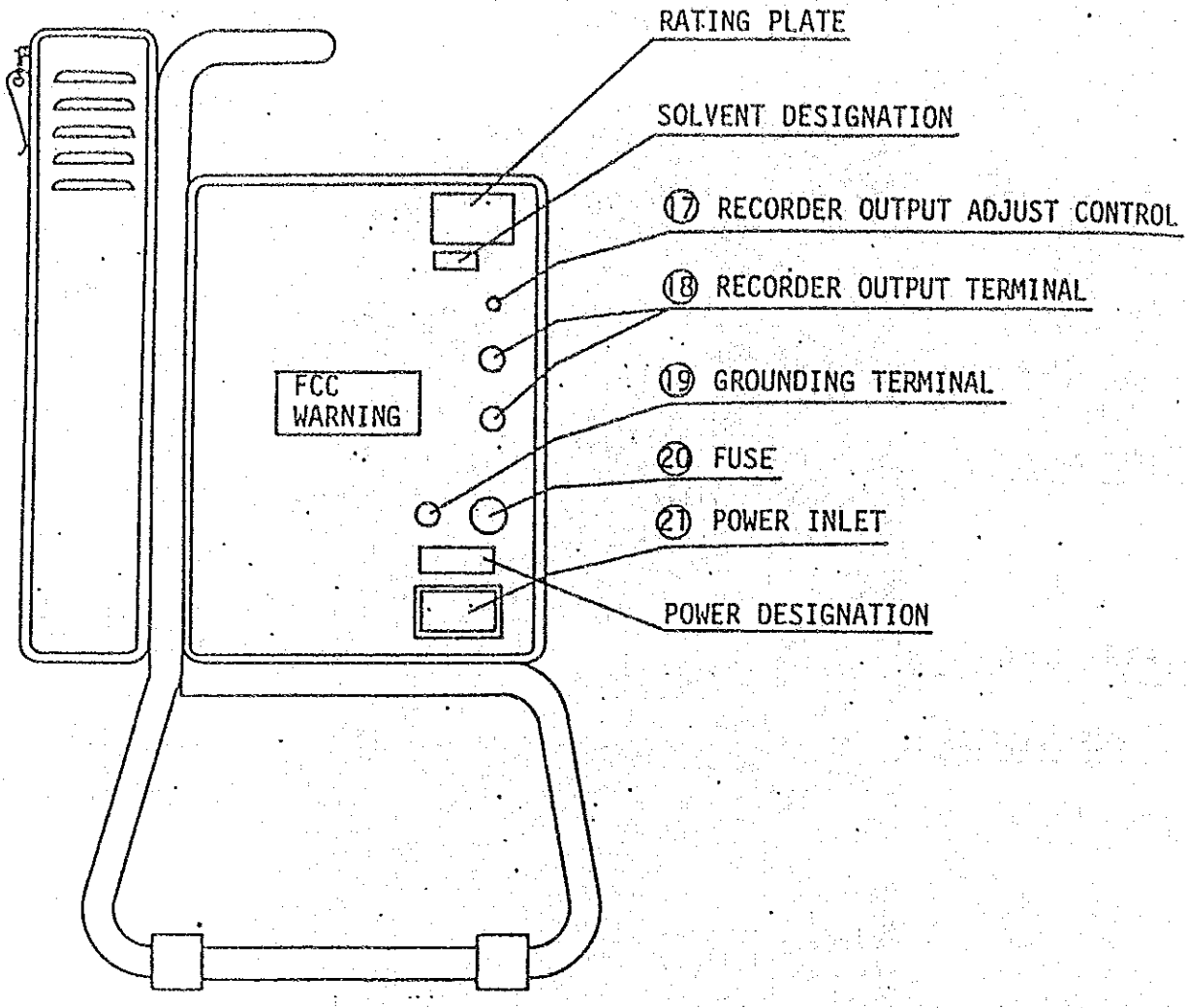


Fig. 5-2 Rear View

6. Preparation for Use

(1) Installation

Select proper location for installation with reference to requirements for location in Section 4.

(2) Check of grounding

Check that grounding is properly provided, with reference to the requirements for grounding in Section 4.

(3) Preparation of apparatus and solvent

Place beaker under discharge outlet to receive waste solution. (To minimize evaporation of solvent in the waste solution, pour tapping water in the beaker to the level of about 1cm from the bottom.)

Prepare syringes (for solvent and sample) and extraction solvent which meets the instrument specifications (carbon tetrachloride or Flon S-316).

Prepare span solution for upscale standard calibration.

Refer to the description below for the process of preparing span solution.

Caution Carbon tetrachloride is a toxic solvent. Users are cautioned to avoid skin contact or breathing of the vapors. Flon S-316 has relatively low toxicity. However, precautions must be taken to ensure the working area is properly ventilated when using the solvent. In all cases, the use of a laboratory venting hood is recommended. Proper care must be used in the disposal of waste solutions. The Horiba reclaimer can serve as a safe deposit for the spend solvent.

(4) Cleaning of apparatus

Rinse syringes two or three times in clean extraction solvent before use.

(5) Power ON

Insert plug into the specified plug socket after confirming that "POWER button" and "MEASURE button" on the instrument front panel are in OFF positions (buttons protrude).

[Preparation of Span Solution (for reference)]

° Introduction

HORIBA OCMA-220 measures oil content on the basis of the conversion to the concentration of "OCB standard solution".