

サウディ・アラビア王国  
海水淡水化技術協力計画調査  
報告書

付 属 資 料

平成 4 年 7 月

国 際 協 力 事 業 団

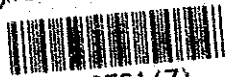
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マイクロ  
フィルム作成

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## 1. M1 添付資料 (スケール防止に関する室内実験)

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蒸発法による海水淡水化装置におけるスケールの生成と防止に関する既往の研究内容を調査した。

調査方法は、日本科学技術情報センターのデータベース「JOIS」の「科学技術文献ファイル」及び米国 DIALOG INFORMATION SERVICE社のオンライン情報検索システム「DIALOG」の各ファイルを利用して関係文献を検索した。

検索に使用したキーワード、論理式及びヒット件数は下記のとおりである。

JOIS 科学技術文献ファイル (1981.01 — 1991.11)

- ① 海水淡水化\*フラッシュ蒸発\* (原油+海水) ————— 9件
- ② 海水淡水化\*フラッシュ蒸発# ① ————— 19件
- ③ 海水淡水化\*蒸発\*付着 ————— 1件
- ④ 海水淡水化\*フラッシュ蒸発\*付着 ————— 1件
- ⑤ 海水淡水化\*蒸発 ————— 69件
- ⑥ 海水淡水化\* (スケール+スケーリング) ————— 16件

JOIS 科学技術文献ファイル (1975.01 — 1980.12)

- ① 海水淡水化\*フラッシュ蒸発 ————— 26件
- ② 海水淡水化\*蒸発 ————— 110件
- ③ 海水淡水化\* (スケール+スケーリング) ————— 76件

DIALOG

- ① FILE 44 AQUATIC SCIENCE ABSTRACTS (78-91/AUG)  
SEA WATER DESALINATION\*(SCALE+SCALING) ————— 49件
- ② FILE 245 WATERNET (71-91/SEP)  
(SEA WATER DESALINATION+SEA WATER\*DESALINATION)  
\*(SCALE+SCALING) ————— 15件

③ FILE 8 COMPENDEX

(SEA WATER DESALINATION+SEA WATER\*DESALINATION)

\*(SCALE+SCALING)\*(EVAPORATION+DISTILLATION) ——— 26件

④ FILE 28 OCEANIC ABSTRACTS (64-91/OCT)

(SEA WATER DESALINATION+SEA WATER\*DESALINATION)

\*(SCALE+SCALING)\*(EVAPORATION+DISTILLATION) ——— 23件

⑥ FILE 14 ISMEC (73-91/AUG)

(SEA WATER DESALINATION+SEA WATER\*DESALINATION)

\*(SCALE+SCALING)\*(EVAPORATION+DISTILLATION) ——— 3件

次に、この検索結果より文献のタイトル、要約等から判断して、多段フラッシュ型海水淡水化装置のスケールの生成と防止に関係すると思われる文献を抽出した。

以下、これらの文献について、雑誌別にタイトル、巻数、頁数、発行年、そして主要なものは、その要旨を示す。

## DESALINATION

### 1. スケール抑制剤の開発と応用に関する過去25年間の経過

3.0 243-255 (1979)

スケール抑制剤に要求される性質が、脱塩技術の進歩とともに変わってきた。初期のポリリン酸塩から最近の高温用添加剤までを概説。酸処理とそれ以外の添加剤処理の場合について処理法を比較し、抑制剤の効果を上げるための設計因子を記述。特に伝熱面の掃除のしやすさ、デミスタの保守の容易さなどの点から添加剤の長期的な適合性を検討。実験室内での模擬プラントを使った抑制剤の評価法を記述。将来の装置設計の傾向と、長期使用に耐えるための抑制剤のタイプについて説明。

### 2. 限界スケール防止現象

3.1 257-266 (1979)

### 3. スケール生成に及ぼす塩水滞留時間の影響

3.1 267-277 (1979)

スケール抑制剤を使用する海水脱塩プラントでの標記問題を検討。装置内での海水の移動を、かくはん流と静流とに分け、スケールの沈殿に及ぼす海水の滞留時間と過飽和度の影響を数学モデルを使って解析。かくはんはスケール生成を増加し、同じ滞留時間ではかくはん流の方がプラグ流れより沈殿がひどく、沈殿への誘導期間も短かった。得られた一般的傾向を、モデル化した脱塩装置で示した。滞留時間と添加剤濃度の影響を炭酸カルシウムについて暫定した沈殿速度定数を使って示した。

#### 4. 脱塩用スケール抑制剤の実験室での応用試験

3.1 279-288 (1979)

#### 5. 海水脱塩におけるスケール抑制剤フロコン247によるアルカリスケール防止の化学

3.1 289-298 (1979)

#### 6. 酸と添加剤の使用 多段フラッシュプラントにおける実際の経験

3.1 299-307 (1979)

#### 7. スケール防止法概説

3.1 309-320 (1979)

多段フラッシュ蒸発法による海水淡水化に関して、海水中のスケール成分を記述し、ポリリン酸ソーダ添加、酸添加及び有機高分子剤添加法を概説。各方法について適用温度範囲、スケール抑制性能と装置の腐食の問題を検討。塩水温度を上げると生産水コストは低下する。有機高分子剤は、コスト的に酸添加に匹敵するまでになった。硫酸イオン交換法、流動層蒸発器、活性剤の適用など、海水蒸発におけるスケール防止技術の最近の進歩を概説。

#### 8. 多段フラッシュ蒸発器において酸処理から添加剤処理に変えたことによる腐食の低下とその監視

3.9 339-350 (1981)

地中海地域での標記の海水淡水化設備において、スケール抑制剤として硫酸・塩酸に代えてポリカルボン酸系の添加剤を使用した場合の効果について記述。塩水側と

蒸溜水側について防食効果を監視した。塩水の電導度、溶解イオン、溶存酸素、か  
くはん度、温度の影響を、また蒸溜水については炭酸ガスと酸素の溶解量の影響を  
検討した。試験したいずれの金属材料についても腐食速度は著しく低下した。試験  
期間中、プラントの性能は変わらず、むしろ、腐食に伴う運転停止がなくなり、操  
業効率が上昇した。

## 9. スケール制御用添加剤—多段フラッシュプラントにおける実際の経験

5.4 301-305 (1985)

### 1.0. 水溶液中における $\text{HCO}_3^-$ と $\text{CO}_3^{2-}$ イオンの熱安定性

6.9 241-249 (1988)

### 1.1. アラビア湾水に関する研究からのアルカリスケール生成の問題

7.1 313-324 (1989)

塩水を $\text{N}_2$ で5時間バブリングしながら還流コンデンサを用いて加熱し、室温に冷  
却後 $\text{HCO}_3^-$ 、 $\text{CO}_3^{2-}$ 、 $\text{OH}^-$ と沈殿の $\text{CaCO}_3$ 、 $\text{Mg}(\text{OH})_2$ を定量し  
た。その結果、 $\text{CaCO}_3$ の溶解度積の限度を超えた $\text{CO}_3^{2-}$ の存在を認め、この  
研究に熱力学的方法は不適當であることが分かった。標題の問題の理解を深めるに  
は、 $\text{CaCO}_3$ の過飽和溶液の安定度、 $\text{CaCO}_3$ の晶出速度論、溶解 $\text{CaCO}_3$   
の安定性、添加剤の効果などの研究が必要である。

### 1.2. スケール制御の実際の経験

7.3 313-325 (1989)

### 1.3. ベルガードEV—スケール防止に15年の経験

7.3 341-357 (1989)

チバ・ガイギー社のスケール抑制剤ベルガードEVの初期の歴史及びその後の研究  
によりEV2000を開発した経過を説明。同剤の最新の経験より、スポンジボー  
ル洗浄との併用、添加量の最適化、添加技術の進歩、高分子剤の分析法の進歩及び  
現場でのプラントモニターの開発について示した。

1.4. 化学薬品によるスケール生成の防止効果

7.4 355-361 (1989)

1.5. スケール抑制剤による脱塩プラントにおけるアルカリスケール生成の限界

8.3 55-63 (1991)

1.6. ドバイのMSF脱塩プラントでの高温条件下における新スケール抑制剤"アクアクリン KC-550"の性能試験

8.3 65-75 (1991)

CORROSION

1.7. 硫酸カルシウムスケールの生成速度と機構-インヒビタの影響

3.5 304-308 (1979)

I&EC FUNDAMENTALS

1.8. 硫酸カルシウムスケールの伝熱表面での析出の機構

9 1-10 (1970)

日本海水学会誌

1.9. アルカリスケールの生成におよぼす添加物の影響

3.0 20-25 (1976)

小型蒸発装置を用いて、海水濃縮比2、温度101℃でアルカリスケールの生成に及ぼす、13種の各種添加剤の効果を検討した。スケール抑制効果のあるものは、縮合リン酸ナトリウムとポリアクリル酸ナトリウムであり、その効果は析出抑制作用によるものである。ポリアクリル酸ナトリウムの効果は重合度によって異なり、重合度の小さいもの (p.g.75, 100) が有効であった。

2.0. 海水濃縮におけるスケールの生成と防止に関する研究

3.2 3-33 (1978)

製塩と海水淡水化におけるスケールの生成と防止について検討した。生成については、海水の濃縮方法と海水中のスケール成分の挙動及び付着スケールの組成の相互関係、かん水中の硫酸カルシウムの転移における溶解、析出速度、さらに、防止については、アルカリ及び硫酸カルシウムスケールの生成に及ぼす各種添加物の影響、スケール防止のためのヘキサメタリン酸ナトリウム添加法の開発等について研究を行った。

## 2.1. 海水淡水化および製塩におけるスケール制御

3.6 14-21 (1982)

海水利用工業において、スケールの生成と防止は重要なテーマであり、この方面の最近の研究の進歩の特色を述べた。海水淡水化についてはスケール生成（アルカリスケール、硫酸カルシウムスケール）、スケール防止（pHコントロール法、限界処理法、スポンジボール法、種晶添加法等）に関し説明し、また製塩については海水の前処理及び濁質除去、スケール生成と防止等について解説。

## 2.2. 多段フラッシュ蒸発式海水淡水化プラントにおける循環ライン中の炭酸物質の挙

3.9 19-26 (1985)

標記プラントにおいてアルカリスケールの析出に関連する炭酸物質の循環ライン中での物質収支を計算し、造水量 $100\text{ m}^3/\text{日}$ 、10段フラッシュ蒸発プラントを用いたpHコントロール法による実験データと比較検討した。ライン中の炭酸物質の物質収支は炭酸物質の解離平衡の過渡状態を基本において計算できた。ラインは各蒸発段の凝縮器及びラインヒータの伝熱管内と一連のフラッシュ室を循環している。このライン中の炭酸物質の物質収支計算は伝熱管ではスケール析出をフラッシュ室では脱炭酸とスケール析出を考慮すればよい。フラッシュ室でのライン流れについて蒸発域と滞留域の二つの領域に分けるモデルを得た

### 化学工学論文集

## 2.3. 多段フラッシュ蒸発式海水淡水化装置におけるアルカリスケールの生成

6 248-254 (1980)

多段フラッシュ蒸発式海水淡水化装置における伝熱管表面でのスケール生成を造水量 $100\text{ m}^3/\text{日}$ 、10段フラッシュ蒸発器を用いて試験検討した。100時間連続



運転試験において、硫酸カルシウムスケールの析出はみられなかったが、蒸発器のブラインヒータと凝縮器に多量のアルカリスケールが生成した。アルカリスケールの主成分は低温域では炭酸カルシウム、高温域では水酸化マグネシウムであった。アルカリスケールの生成はブライン中の炭酸物質の解離平衡に関係している。浸管式蒸発管では伝熱管表面でブラインが蒸発するが、フラッシュ蒸発器では伝熱管内でブラインの蒸発は起きないので、フラッシュ蒸発器での炭酸物質の挙動は浸管式蒸発管での挙動と異なる。スケール析出速度データを用いてフラッシュ蒸発器における炭酸物質の物質収支を計算し、炭酸カルシウムと水酸化マグネシウムの表面晶析速度係数をブライン温度の関数として得た。

#### 2.4. 多段フラッシュ蒸発式海水淡水化装置におけるスポンジボール洗浄法によるスケール防止

且 516-522 (1983)

造水量  $100 \text{ m}^3/\text{日}$ 、10段フラッシュ蒸発プラントを用いてスポンジボール洗浄法のスケール防止効果を調べた。ボール洗浄効果はスケール析出速度がスケール付着量に逆比例し、スケール除去はスケール析出速度が非常に大きいので充分でないことが分かった。補給海水に、酸を少量添加して50%脱炭酸するpHコントロール法にスポンジボール洗浄法を併用すれば、スケール生成と腐食を防止できる優れた効果を示した。

以上 海水淡水化装置におけるスケールの生成と防止に関する文献について内容を調査したが、本研究の目的であるスケール抑制剤が海水に混入された油によってどのような影響を受けるかについての報文は見当たらなかった。

しかし、既往の基礎的な研究報文については、本実験研究を進める際に大いに役立ち、今後、研究結果を定量的にまとめるときの参考資料になるものと思われる。



RESULTS OF CORROSION STUDIES ON MILD STEEL (MS) AND  
STAINLESS STEEL, TYPE 316 (SS) IN BRINE WITHOUT  
OIL AND WITH 100 PPM OIL AT 50°C & 80°C

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Saline Water Conversion Corporation*

### Introduction

The general corrosion rates were determined for the two abovementioned materials, MS and SS under the different conditions given above, i.e., in plain brine, containing no oil and in brine having 100 ppm of oil mixed in it. The experiments were conducted under these conditions at 50°C and 80°C.

### Experimental

Electrochemical methods were employed to determine the corrosion rates. The experiments were carried out on EG & G model 342-2 Soft Corr measurement system. The system consists of model 273 potentiostat/galvanostat, model 342 corrosion software and model 30 IBM PS-2. All the experiments were carried out using a corrosion cell with saturated calomel as reference electrode (SCE) and graphite as counter electrode (EG & G model K0047).

The test specimens (of MS & SS) were taken in the form of small circular disks, about 1.5-2 mm thick and 16 mm diameter. They were ground successively with different grades of emery paper down to 600 grit, then washed, rinsed, degreased, rinsed again in distilled water, dried and placed in the sample holder of the model K0047 test cell. The total exposed surface area of the specimen was  $1\text{cm}^2$ . The brine solution was first deaerated by purging with pure nitrogen for 1 hour, then the specimen was placed in solution and allowed to attain steady-state for 2 hours. Thereafter the experiments were run with the softcorr program. The polarization resistance (PR) technique was used for the corrosion rate measurements. Tafel experiments were also run

separately for each experimental conditions to obtain values of anodic and cathodic tafel slopes which are needed for calculating the corrosion rates by the PR technique. Throughout the measurements, the brine solution was kept stirred by a magnetic stirrer.

The experimental set up used is explained in Fig. 1, where the test cell arrangement and other instruments used are shown schematically with the help of sketches and block diagrams.

### Results and Discussion

As already stated, the corrosion rates were determined using polarization resistance (PR) technique. Figs. 2 & 3 show the polarization resistance plots obtained for mild steel at 80°C in brine containing no oil and 100 ppm oil respectively. Similar plots were obtained for all the other experimental conditions and corrosion rates were determined from each of them. The corrosion rates obtained for both MS and SS under all the experimental conditions are presented in table 1 below :

Table 1

Experimental Conditions	Corrosion rates, mpy	
	Mild Steel	SS 316
Plain brine, 80°C	12.81	0.2065
Brine + 100 ppm oil, 80°C	10.43	0.135
Plain brine, 50°C	16.82	0.2797
Brine + 100 ppm oil, 50°C	6.68	0.1498

The most noteworthy feature brought out by the experimental results is that mixing of 100 ppm of oil to the brine brings down the corrosion rates from those obtained in plain brine without oil addition. This is true for both MS and SS at both the experimental temperatures. This observation indicates that the oil might form some kind of film on the metal surfaces that causes

a reduction in the corrosion rates. The reduction in corrosion rates, however, is more pronounced at 50°C compared to that at 80°C, particularly for mild steel. Also, the corrosion rates at 80°C are lower than those at 50°C in plain brine for both MS and SS. These observations may suggest that there is perhaps more effective deaeration at 80°C as compared to the deaeration at 50°C, which in turn will lead to a lower oxygen reduction at the higher temperature. The net effect is that the corrosion rate is lower at the higher temperature. There is yet another possibility that the adsorption characteristic of the oil film on metal surface may be significantly affected by the temperature, such that at the lower temperature (50°C) the oil film is forming a more effective barrier than at the higher temperature (80°C). Thus there is a greater reduction in corrosion rate at 50°C than at 80°C between plain brine and oil mixed brine.

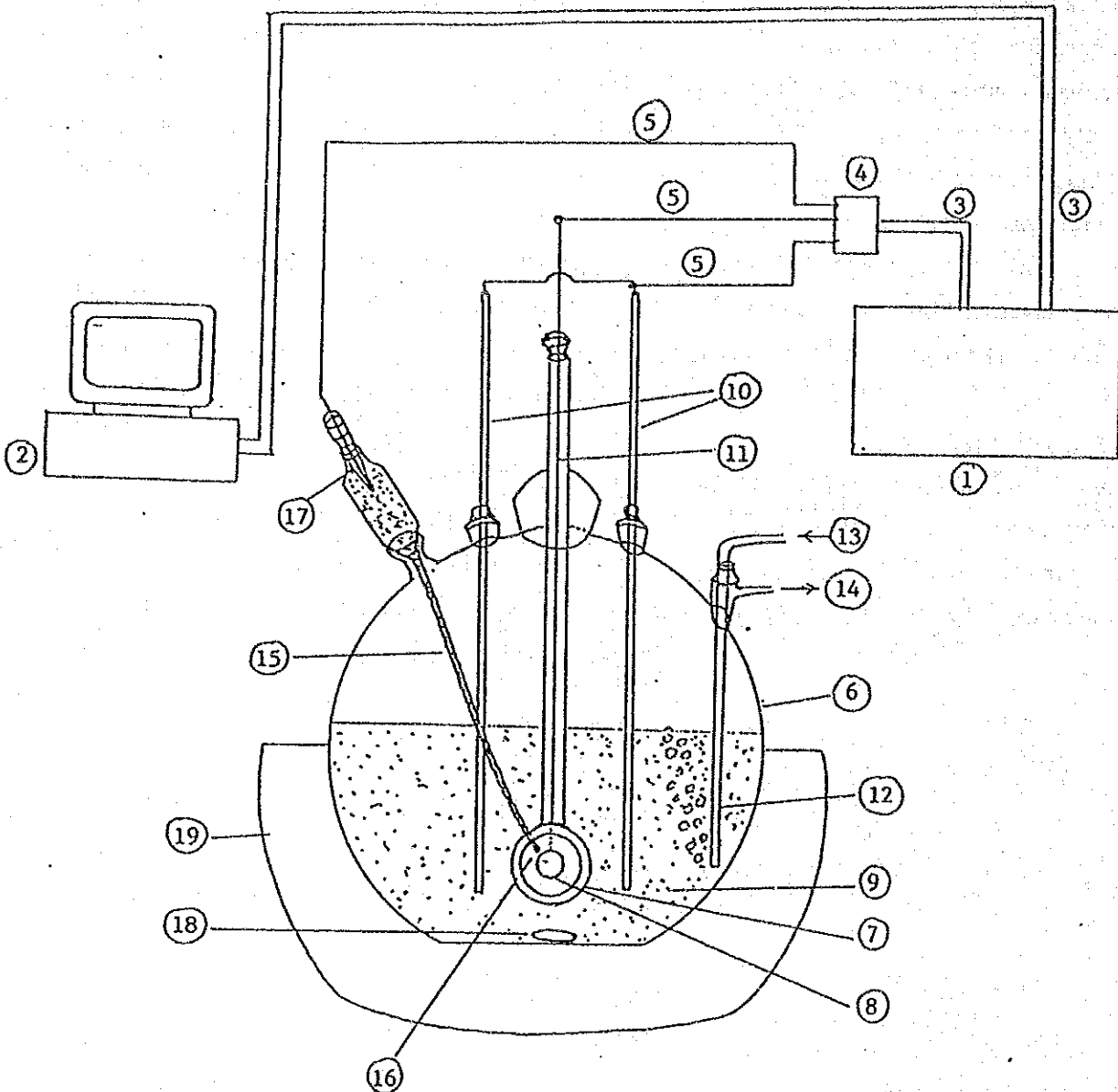


Fig. 1 Schematic presentation of experimental set up shown with the help of sketches and block diagrams (not drawn proportionately).

**Legends :**

(1) Potentiostat, EG &G model 273, (2) Computer, IBM Model 30 PS-2, (3) Cables connecting Potentiostat to differential electrometer & computer, (4) Differential electrometer, (5) Electrical leads from different electrodes to differential electrometer, (6) Test cell (EG & G model K0047), (7) Specimen holder (Teflon), (8) Specimen (exposed area = 1 cm<sup>2</sup>), (9) Test solution (brine), (10) Counter electrodes (graphite), (11) Electrical contact between specimen & electrical lead, (12) Gas-purging tube, (13) Purified nitrogen IN, (14) Nitrogen OUT, (15) Salt bridge capillary; (16) Membrane tip, (17) Reference electrode (saturated calomel), (18) Magnetic stirring bit, (19) Heating bath with magnetic stirrer & temperature controller.

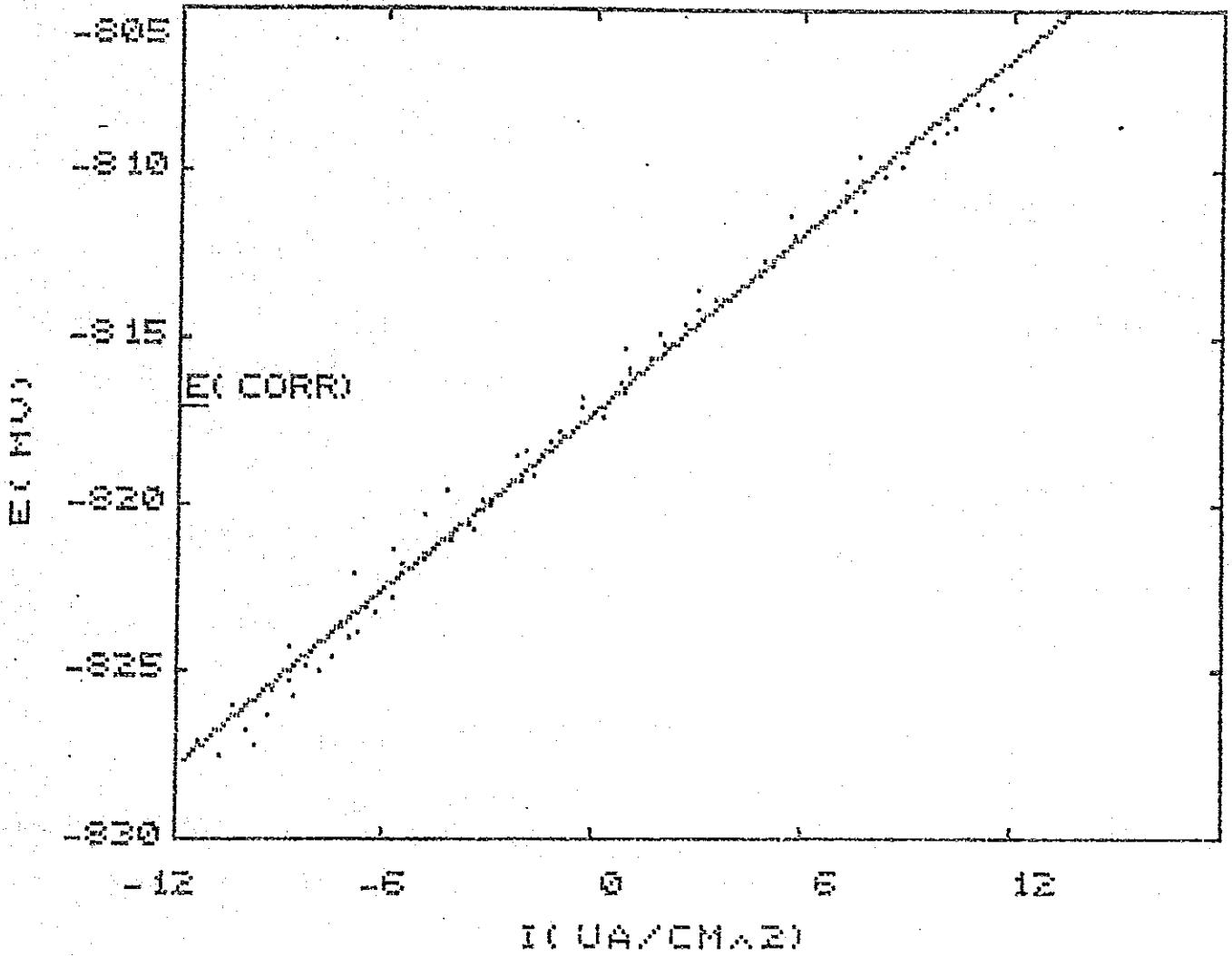


Fig. 2 Polarization-resistance plot for mild steel at 80°C in plain brine containing no oil.

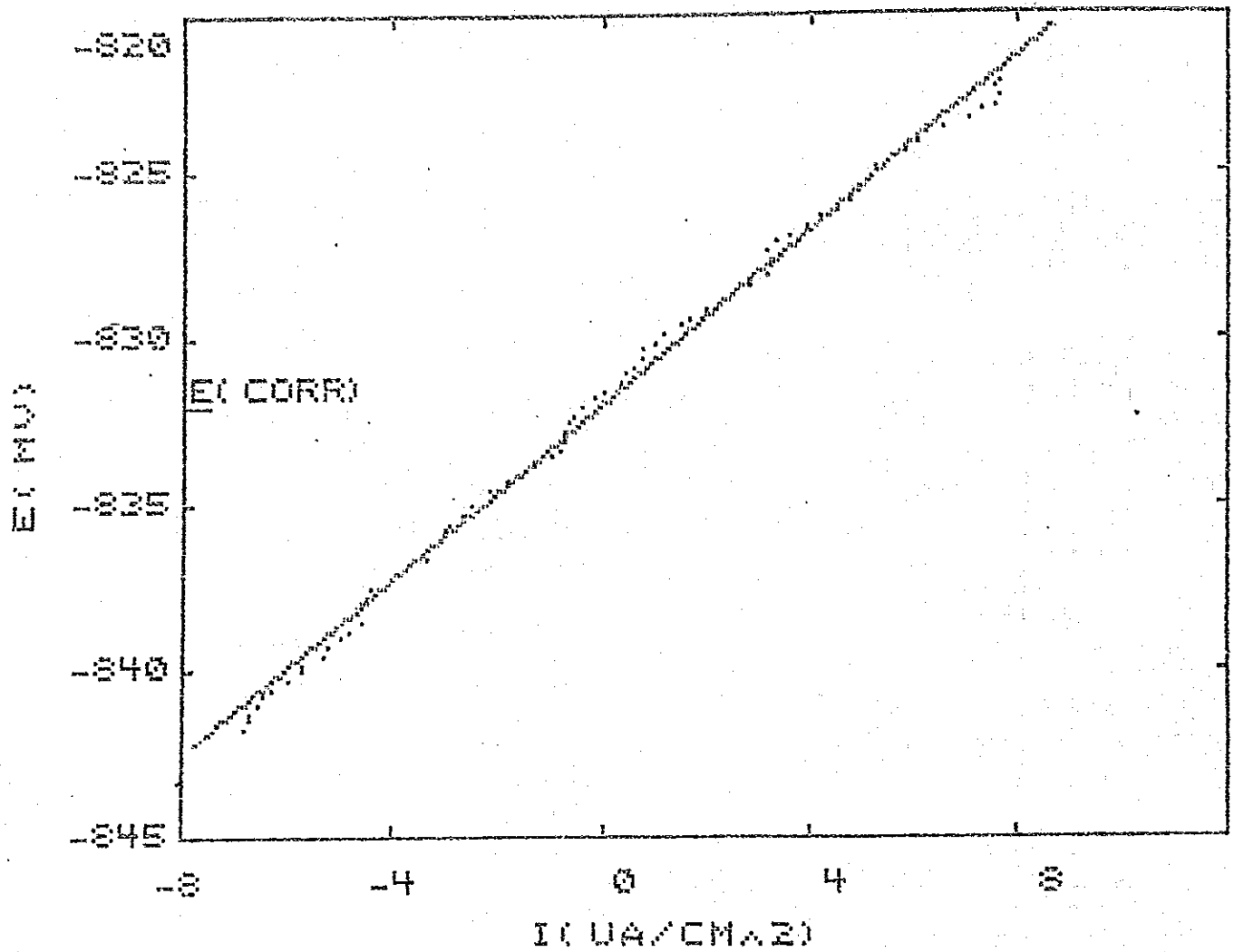


Fig. 3 Polarization resistance plot for mild steel at 80°C in brine mixed with 100 ppm oil.



## M 1 - 3 油分共存下における予備実験データ

## 1) 調査目的

試料油中の炭素数の分布および含量をガスクロマトグラフ法  
によって測定する。

## 2) 分析方法

ガスクロマトグラフ分析法

(A) 炭素数12以下については、島津GC-14Aを用いて分析を行った。

(B) 炭素数13以上については、島津GC-7Aを用いて分析を行った。

## 3) 結果

各炭素数の含量は、パラフィン炭化水素として計算した。

(A) については、n-ヘプタン換算により各炭素数の含量を求めた。

(B) については、n-セタン換算により各炭素数の含量を求めた。

※参考文献：舟阪渡、池川信夫 編著

最新ガスクロマトグラフィー、-基礎と応用-

II 応用編 (1969年)

チャート一覧表 (A : 島津GC-14A、B : 島津GC-7A)

(A) - 1、(B) - 1 …… 試料 (100%)

(A) - 2、(B) - 2 …… 試料 (7t)で20%に希釈したもの)

(A) - 3 …… 炭素数 6~10の標準物質

(A) - 4 …… 炭素数 11~14の標準物質

(B) - 3 …… 炭素数 11~14及び15~20の標準物質

(B) - 4 …… 炭素数 18~20、24、28の標準物質

試料名           A重油          

炭素数	試料	A重油	炭素数	試料	A重油
C-1		不検出	C-17 (%)		2.83
C-2		不検出	C-18 (%)		2.15
C-3		不検出	C-19 (%)		1.23
C-4		不検出	C-20 (ppm)		8700
C-5 (ppm)		11	C-21 (ppm)		3400
C-6 (ppm)		128	C-22 (ppm)		1850
C-7 (ppm)		416	C-23 (ppm)		708
C-8 (ppm)		647	C-24 (ppm)		458
C-9 (ppm)		3080	C-25 (ppm)		190
C-10 (ppm)		9620	C-26 (ppm)		130
C-11 (ppm)		9380	C-27 (ppm)		86
C-12 (%)		1.32	C-28 (ppm)		11
C-13 (%)		1.26			
C-14 (%)		2.49			
C-15 (%)		2.76			
C-16 (%)		1.85			

(単位 )

試料名

水質

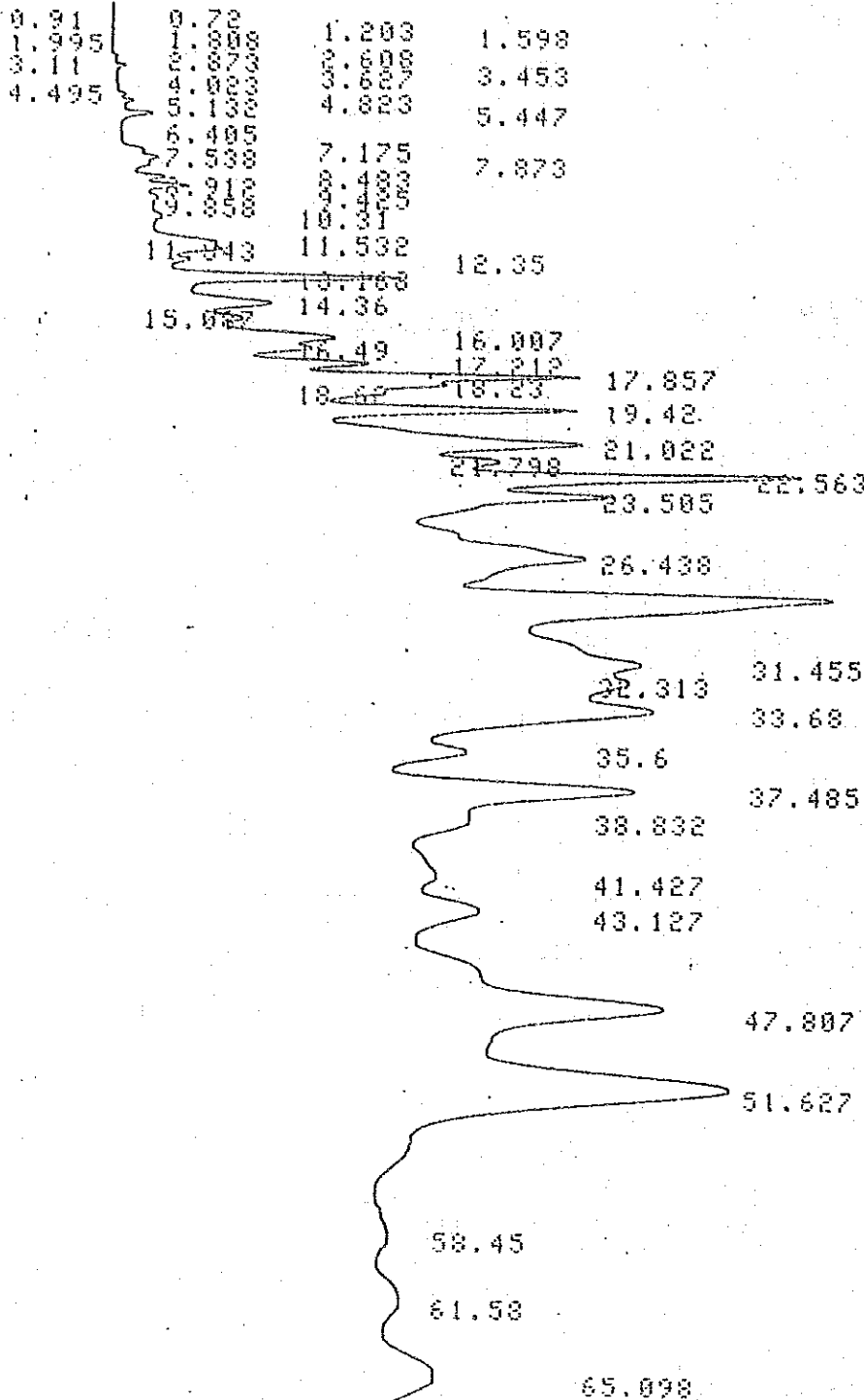
成分	試料 原 水	1.8 倍 濃縮ライノ	'91-10-17 ライノろ液	'91-10-17 ライノ+スケール 防止剤ろ液	'91-10-18 ライノ+ A重油+ スケール防止剤 ろ液
pH (°C)	8.0 (23)	8.3 (23)	8.5 (23)	8.8 (23)	7.9 (23)
導電率 (25°CmS/cm)	45.5	68.1	—	—	—
Mアルカリ度 (as CaCO <sub>3</sub> )	110	180	300	260	85
Na	9300	17600	—	—	—
K	390	—	—	—	—
Ca	380	670	—	—	—
Mg	1200	2200	—	—	—
Cl	18900	33500	42000	33000	32400
SO <sub>4</sub>	2500	4400	—	—	—

試料名 水質

成分	試料				
pH (°C)	'91-10-18 フイン+油+ スケール防止剤 る液				
Mアルカリ度 (as CaCO <sub>3</sub> )	8.7 (23)				
Cl	260				
	32900				

試料名                      A重油及びスケール

試料	成分	A重油	スケール1	スケール2	スケール3	スケール4
比重	(15/4°C)	0.8679	—	—	—	—
粘度	50°C (cSt)	2.848	—	—	—	—
S	(wt%)	0.64	—	—	—	—
Ca	(wt%)	—	13.32	34.79	17.96	32.92
Mg	(wt%)	—	5.64	0.64	4.22	1.17
試料名 スケール1 : ブライン+Na <sub>2</sub> CO <sub>3</sub> 1991-10-17						
スケール2 : ブライン+ A重油+ Na <sub>2</sub> CO <sub>3</sub> 1991-10-17						
スケール3 : ブライン+ BELCLEN 200+ Na <sub>2</sub> CO <sub>3</sub> 1991-10-17						
スケール4 : ブライン+ A重油+ BELCLEN 200+ Na <sub>2</sub> CO <sub>3</sub> 1991-10-17						
備考 : 各スケールは n-ヘキサンで洗浄後上記分析を行った。						



(A) - 1

056

Shimadzu  
atomic

20.46 CHROMATOGRAM

Date	H3. 11. 5	No.	
Room Temp.	21.75, 20G	Carrier Gas	N <sub>2</sub>
Model	22" GC-14A	Flow Rate	ml/min. atm.
Sample	A 5th	Inlet Press.	4.4 Kg/cm <sup>2</sup>
%L.G.	2 ml. l. l. g.	H <sub>2</sub> Flow Rate	ml/min.
% ppm Solvent		Air Flow Rate	l/min.
Column	%G. L. m. ID 2.0mm.	Detector	FID
Temp.	100 °C → 180 °C	Bridge Curr.	mA
Temp. Rate	4 °C/min.	Applied Voltage	V
Packing	Apiezon - L <sub>1</sub>	Rad. Source	
Wt. %	25	Range	12P mV. *
Support	Shimalite W (NAW)	Sensitivity	
Mesh	60 ~ 80	Detector Temp.	200 °C
Treatment		Injection Temp.	200 °C
Ref. Column		Chart Speed	3 mm/min.
		Operator	

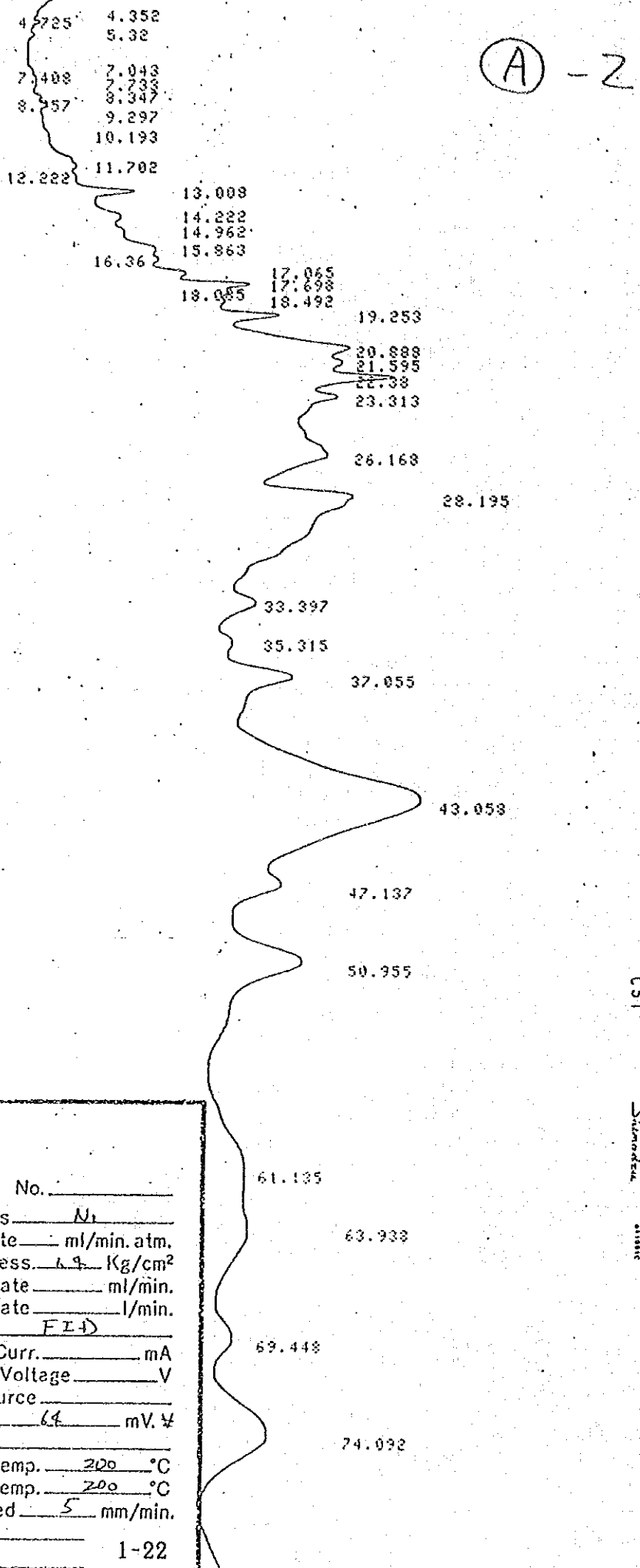
1	1.808	2000		0.0056	C-5
2	2.608	6318		0.0177	
3	2.873	4227		0.0118	
4	3.11	15140		0.0424	C-6
5	4.023	6577		0.0184	
6	4.495	12171		0.0341	
7	4.823	19194		0.0538	
8	5.132	2332		0.0065	
9	5.447	58644		0.1643	C-7
10	6.405	7430		0.0208	
11	7.175	5549		0.0155	
12	7.538	25688		0.072	
13	7.873	11354		0.0318	
14	8.483	41377		0.1159	
15	8.912	82519		0.2312	C-8
16	9.425	24100		0.0675	
17	9.858	3194		0.009	
18	10.31	31906		0.0894	
19	11.532	147411		0.413	
20	11.843	129955	V	0.3641	
21	12.35	33383		0.0935	
22	13.168	583696		1.6355	C-9
23	14.36	249071		0.6979	
24	15.087	67230		0.1884	
25	16.007	400506		1.1222	
26	16.49	184973	V	0.5183	
27	17.212	309380		0.8669	
28	17.857	841531	V	2.358	
29	18.23	341340	V	0.9564	
30	18.62	163377	V	0.4578	C-10
31	19.42	685493		1.9208	
32	21.022	1380078		3.867	
33	21.798	580900	V	1.6277	
34	22.563	1797889	V	5.0377	C-11
35	23.505	1191699	V	3.3392	
36	26.438	1393062		3.9034	
37	28.44	2507902		7.0246	C-12
38	31.455	705274		1.9762	
39	32.313	315480	V	0.884	
40	33.68	818100		2.2923	
41	35.6	287066		0.8044	
42	37.485	1390775		3.897	
43	38.832	118172		0.3311	(C-13)
44	41.427	193494		0.5422	
45	43.127	528707		1.4814	
46	47.807	2649582		7.4242	
47	51.627	3597298		10.0797	(C-14)
48	58.45	214783		0.6018	
49	61.58	302389		0.8473	
50	65.098	1421771		3.9838	
51	70.46	1292753		3.6223	
52	75.205	5269773		14.766	(C-15)
53	80.91	843376		2.3631	
54	86.823	213908		0.5994	

(A)-1a  
DATA

55 90.995 2178269 6.1035

TOTAL 35689656

1.365



(A) -2

### CHROMATOGRAM

No. \_\_\_\_\_

Date Hz. 11.5  
 Room Temp. 21 °C  
 Model 322" GC-4A  
 Sample Atch  
 Wt. % 2 ml. pl. mg.  
20% ppm Solvent EtZ  
 Column J.G. L. 2 m. ID. 3.0 mm.  
 Temp. 100 °C → 180 °C  
 Temp. Rate 4 °C/min.  
 Packing Apieson-L  
 Wt. % 25  
 Support Shimalite W (NAW)  
 Mesh 60-80  
 Treatment \_\_\_\_\_  
 Ref. Column \_\_\_\_\_

Carrier Gas N<sub>2</sub>  
 Flow Rate \_\_\_\_\_ ml/min. atm.  
 Inlet Press. 1.9 Kg/cm<sup>2</sup>  
 H<sub>2</sub> Flow Rate \_\_\_\_\_ ml/min.  
 Air Flow Rate \_\_\_\_\_ l/min.  
 Detector FID  
 Bridge Curr. \_\_\_\_\_ mA  
 Applied Voltage \_\_\_\_\_ V  
 Rad. Source \_\_\_\_\_  
 Range 64 mV.  $\mu$   
 Sensitivity \_\_\_\_\_  
 Detector Temp. 200 °C  
 Injection Temp. 200 °C  
 Chart Speed 5 mm/min.  
 Operator \_\_\_\_\_

CSI Shimadzu



STOP 1

C-REAX  
CHANNEL 1  
SAMPLE NO 0  
REPORT NO 591

FILE 3  
METHOD 41

Ⓐ-Z の DATA

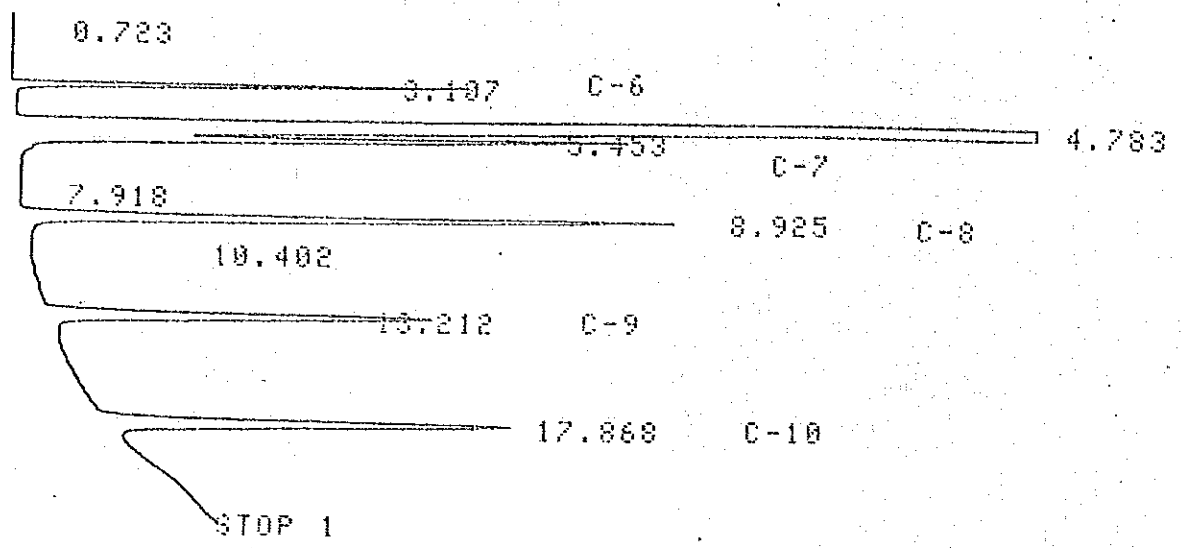
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	1.365	28751954	E		72.4012	
2	4.352	1452			0.0037	
3	4.725	12143			0.0306	
4	5.32	10669			0.0269	
5	7.043	3154			0.0079	
6	7.408	4753			0.012	
7	7.733	2233			0.0056	
8	8.347	9860			0.0223	
9	8.757	15523			0.0391	
10	9.297	4062			0.0102	
11	10.193	3477			0.0088	
12	11.702	37870			0.0954	
13	12.222	5906			0.0151	
14	13.008	104571			0.2633	
15	14.222	40752			0.1026	
16	14.962	11433			0.0288	
17	15.863	31955			0.0805	
18	16.36	14658			0.0369	
19	17.065	39567			0.0996	
20	17.698	117746			0.2965	
21	18.085	33231	V		0.0837	
22	18.492	8148			0.0205	
23	19.253	126405			0.3183	
24	20.888	333132			0.8389	
25	21.595	89076	V		0.2243	
26	22.38	183421			0.4619	
27	23.313	91885			0.2314	
28	26.168	485926			1.2236	
29	28.195	1566030			3.9435	
30	33.397	175005			0.4407	
31	35.315	50315			0.1267	
32	37.055	408930			1.2312	
33	43.058	3756336			9.4589	
34	47.137	172782			0.4351	
35	50.955	1141706			2.875	
36	61.135	125004			0.3148	
37	63.938	260282			0.6554	
38	69.448	205784			0.5182	
39	74.092	1195816			3.9112	
TOTAL		39711976			99.9999	

700

START 1

91/11/85

17:03:11



C-R2AX  
 CHANNEL 1  
 SAMPLE NO 8  
 REPORT NO 595

FILE 3  
 METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.107	463185			1.5281	C-6
2	4.783	25838134	E		85.2433	L
3	5.453	1038794	V		3.4271	C-7
4	7.918	5688			0.0188	
5	8.925	1295350			4.2735	C-8
6	10.402	3274			0.0108	
7	13.212	853282			2.8151	C-9
8	17.868	813344			2.6833	C-10
TOTAL		30311042			100	

(A) - 4

4.678

1  
C11

5.868

15.152

17.792 17.512 18.09

19.588 19.118

20.262

22.302

(C11)

23.965

26.41

28.327

(C12)

30.873

32.108

33.6 33.275

34.4 34.043

37.273

(C13)

39.752

40.498

42.5

51.5

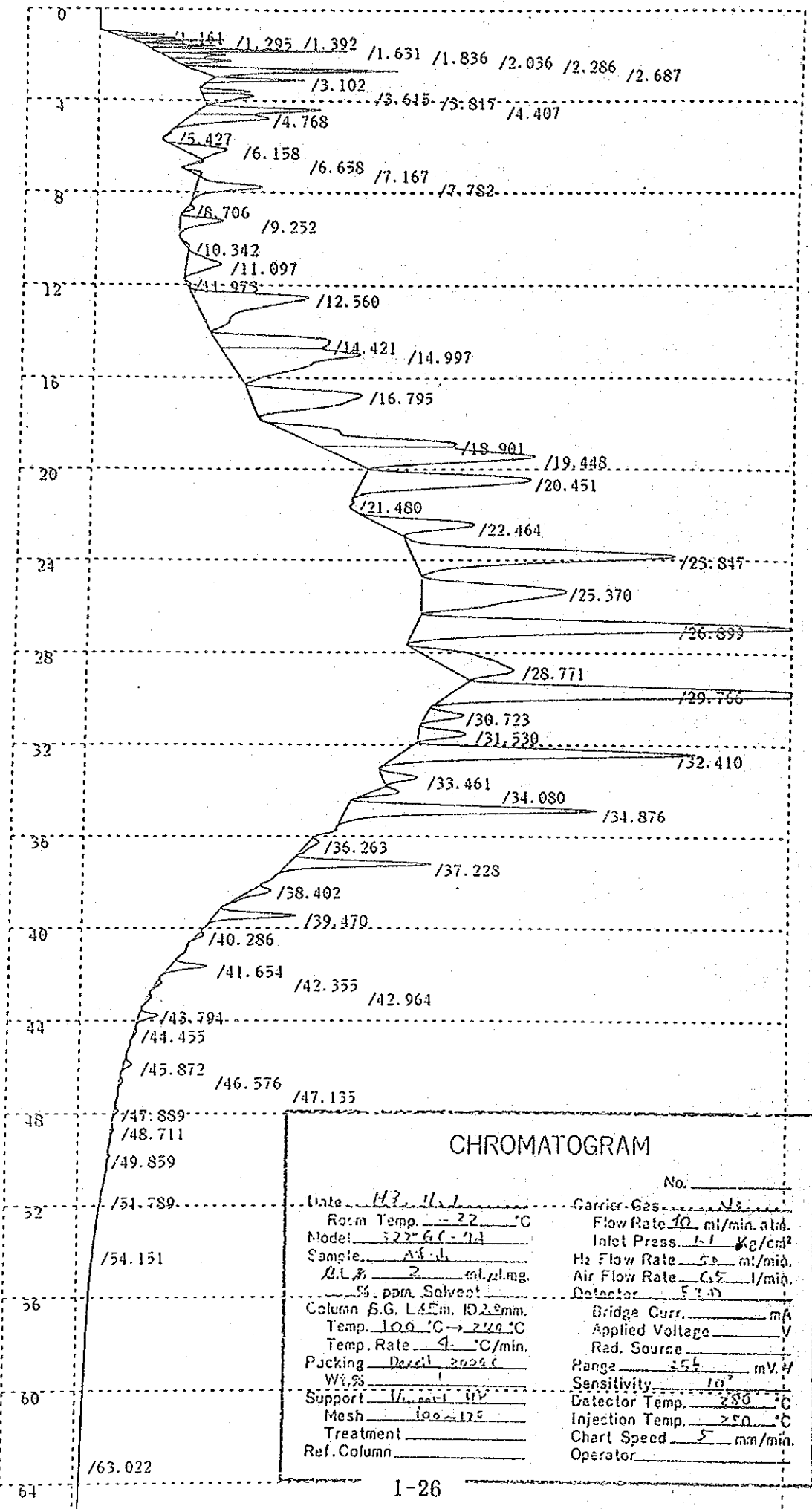
(C14)

58.438

62.482

STOP 1-25

(B) - 1



**CHROMATOGRAM**

No. \_\_\_\_\_

Date <u>11/2/91</u>	Carrier Gas <u>N<sub>2</sub></u>
Room Temp. <u>-22</u> °C	Flow Rate <u>10</u> ml/min. air
Model <u>522 GC-7A</u>	Inlet Press. <u>1.1</u> kg/cm <sup>2</sup>
Sample <u>Asst.</u>	H <sub>2</sub> Flow Rate <u>5</u> ml/min.
<u>2</u> ml. pt. mg.	Air Flow Rate <u>0.5</u> l/min.
<u>5</u> ppm. Solvent	Detector <u>FID</u>
Column <u>S.G. L. 10m. ID 2mm.</u>	Bridge Curr. _____ mA
Temp. <u>100</u> °C → <u>220</u> °C	Applied Voltage _____ V
Temp. Rate <u>4</u> °C/min.	Rad. Source _____
Packing <u>Porasil Q 200/100</u>	Range <u>25</u> mV/V
Wt. % _____	Sensitivity <u>10<sup>9</sup></u>
Support <u>100/120</u>	Detector Temp. <u>280</u> °C
Mesh <u>100-120</u>	Injection Temp. <u>250</u> °C
Treatment _____	Chart Speed <u>5</u> mm/min.
Ref. Column _____	Operator _____

Shimadzu  
 073  
 223-02000-11  
 910115  
 Shimadzu

\*\* 流量計算結果 \*\*

CH	PKNO	TIME	AREA	HEIGHT	NK	IDNO	CONC	NAME
1	2	1.161	120888	18043				
	3	1.295	102592	18195	V			
	4	1.392	169544	33515	V			
	5	1.631	155451	25256				
	6	1.836	450195	69589				
	7	2.036	224317	23179	V			
	8	2.286	196310	20956	V			
	9	2.687	856388	73273				
	10	3.102	263311	32690				
	11	3.615	167460	18000				
	12	3.817	321303	18332	V			
	13	4.407	654600	43231				
	14	4.768	581295	29030	V			
	15	5.427	10025	995				
	16	6.158	405350	16394				
	17	6.658	25815	1786				
	18	7.167	37455	3115				
	19	7.782	549204	23931				
C-12	20	8.706	41425	2732				
	21	9.252	336767	15830				
	22	10.342	17470	1019				
	23	11.097	456619	12837				
	24	11.973	16013	1342				
C-13	25	12.56	1918664	41050				
	26	14.421	1266421	40452				
	27	14.997	2322311	48874	V			
C-14	28	16.795	1972882	40202				
	29	18.901	1706937	52561				
	30	19.448	2641127	71027	V			
C-15	31	20.451	2317603	61455				
	32	21.48	11351	1058				
	33	22.464	1036011	34083				
C-16	34	23.847	3611472	96449				
	35	25.37	2724592	53006				

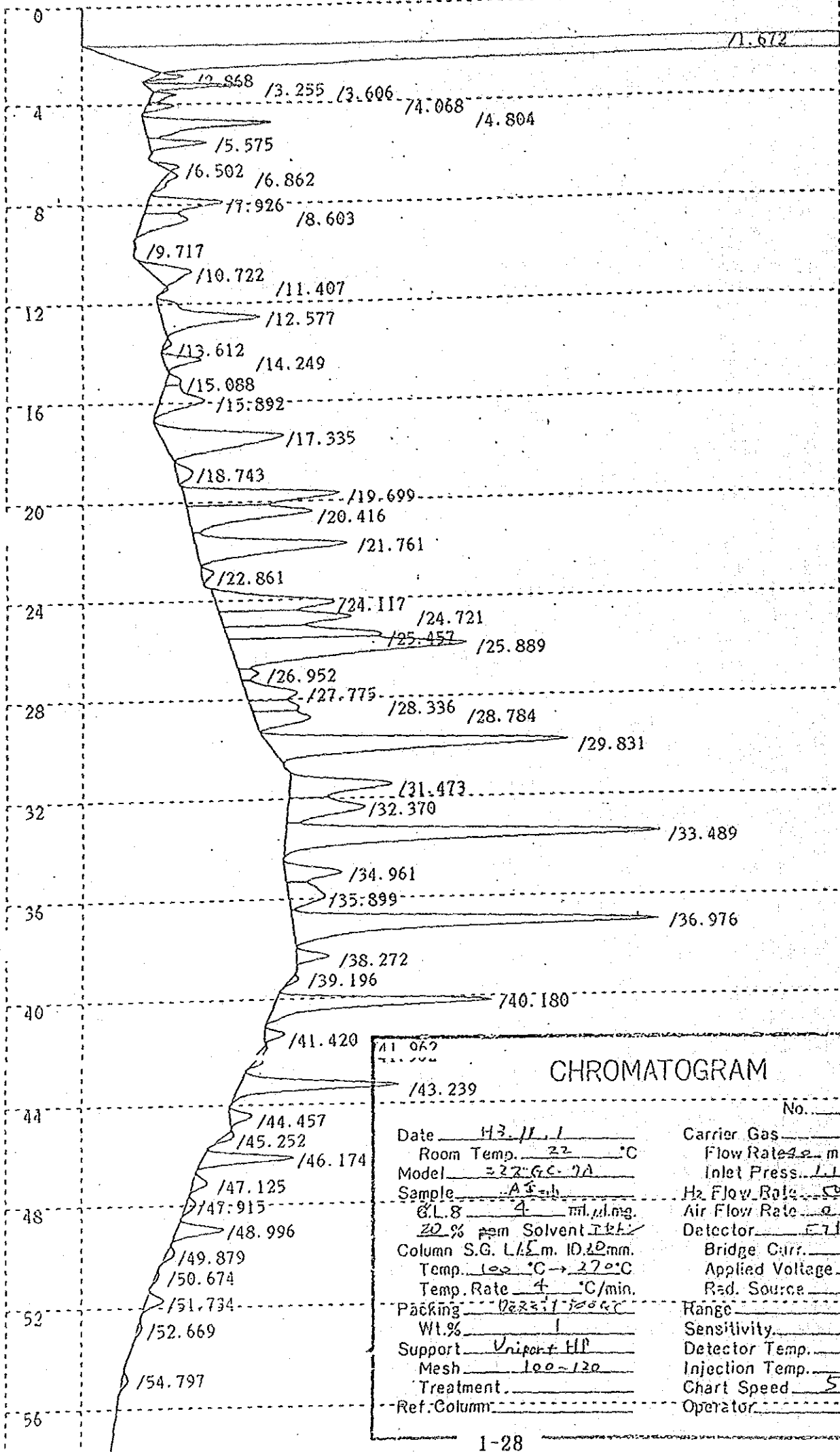
(B) - 1 a DATA

C-17	36	26.899	4700377	139496				
	37	28.771	1432288	22258				
C-18	38	29.766	4438173	150187				
	39	30.723	317430	13837				
	40	31.53	404091	17076				
C-19	41	32.41	2840897	107747				
	42	33.461	267674	12351				
	43	34.08	198783	9857				
C-20	44	34.876	2125784	91946				
	45	36.263	104802	3759				
C-21	46	37.228	921103	52074				
	47	38.402	168122	6608				
C-22	48	39.47	462224	28759				
	49	40.286	34469	2224				
C-23	50	41.654	212497	12935				
	51	42.355	29830	1661				
	52	42.964	25637	1229				
C-24	53	43.794	113509	6750				
	54	44.455	18145	1001				
C-25	55	45.872	55662	3070				
	56	46.576	20825	1160				
	57	47.135	9230	430				
C-26	58	47.889	22161	1439				
	59	48.711	24274	802				
C-27	60	49.859	10110	706				
C-28	61	51.789	5145	363				
	62	54.151	4986	247				
	63	63.022	5502	148				

TOTAL 46660848 1733604

0

(B) - 2



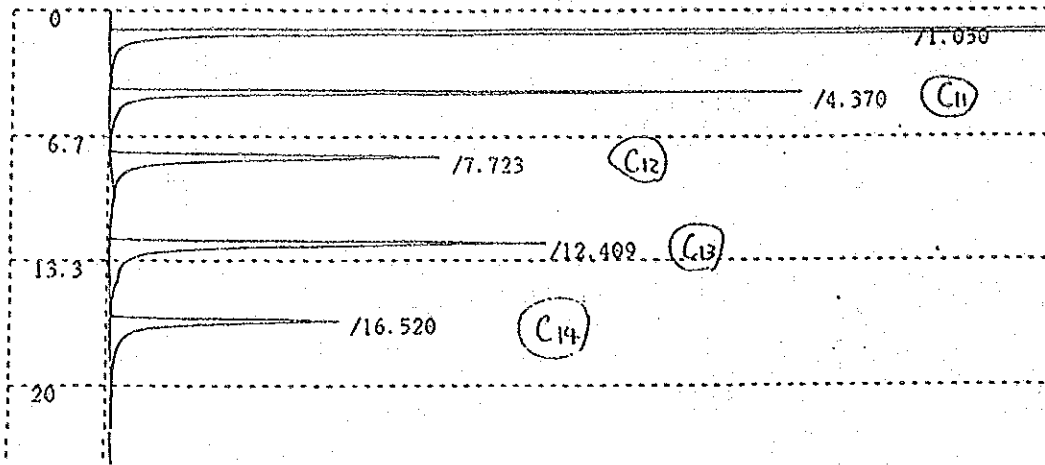
CHROMATOGRAM		No. _____
Date <u>H3. 11. 1</u>	Carrier Gas <u>N<sub>2</sub></u>	
Room Temp. <u>22</u> °C	Flow Rate <u>2.2</u> ml/min. atm.	
Model <u>322 G.C. 7A</u>	Inlet Press. <u>1.1</u> Kg/cm <sup>2</sup>	
Sample <u>A.S. 1</u>	H <sub>2</sub> Flow Rate <u>50</u> ml/min.	
G.L. <u>4</u> ml. / ml. mg.	Air Flow Rate <u>0.5</u> l/min.	
<u>20 %</u> ppm Solvent <u>100</u>	Detector <u>FID</u>	
Column S.G. <u>L.I.E.m. 10.20mm.</u>	Bridge Curr. _____ mA	
Temp. <u>100</u> °C → <u>270</u> °C	Applied Voltage _____ V	
Temp. Rate <u>4</u> °C/min.	Rad. Source _____	
Packing <u>Porapak Q</u>	Range _____ mV, V	
Wt. % <u>1</u>	Sensitivity <u>10<sup>2</sup></u>	
Support <u>Unipor HP</u>	Detector Temp. <u>250</u> °C	
Mesh <u>100-120</u>	Injection Temp. <u>250</u> °C	
Treatment _____	Chart Speed <u>5</u> mm/min.	
Ref. Column _____	Operator _____	

\*\* 定数計算結果 \*\*

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	6	1.672	32345432	1389335	E			
	7	2.868	29559	2589				
	8	3.255	89712	8126				
	9	3.606	33430	2088				
	10	4.068	38252	2354	V			
	11	4.804	242366	10842				
	12	5.575	79395	4896	V			
	13	6.502	19232	1482				
	14	6.862	29638	928				
	15	7.926	174184	6490				
	16	8.603	136395	3967	V			
	17	9.717	2755	183				
	18	10.722	119770	3529				
	19	11.407	3980	285				
	20	12.577	281920	8311				
	21	13.612	10409	612				
	22	14.249	70419	3217				
	23	15.088	27685	1240				
	24	15.892	162691	3902	V			
	25	17.335	441857	10460				
	26	18.743	45045	1377				
	27	19.699	415462	13109				
	28	20.416	369498	10440	V			
	29	21.761	434388	12700	V			
	30	22.861	22179	997				
	31	24.117	337789	9934				
	32	24.721	331966	10930	V			
	33	25.457	319038	13003	V			
	34	25.889	775591	19947	V			
	35	26.952	32147	1511	V			
	36	27.775	143421	4250	V			
	37	28.336	95166	3969	V			
	38	28.784	154590	4633	V			
	39	29.831	768236	25129				
	40	31.473	280460	8560				
	41	32.37	248159	6488	V			
	42	33.489	912637	31733	V			
	43	34.961	134493	4629				
	44	35.899	136920	3011	V			
	45	36.976	872350	30877	V			
	46	38.272	58182	2650				
	48	39.196	13548	654				
	49	40.18	463839	18261				
	50	41.42	33629	1633				
	51	41.962	18675	401				
	52	43.239	331054	13318				
	53	44.457	43669	1751				
	54	45.252	20685	855				
	55	46.174	169491	7665				
	56	47.125	33420	1248				
	57	47.915	25047	785				
	58	48.996	99157	4083				
	59	49.879	17703	621				
	60	50.674	6785	184				
	61	51.734	37253	1487				
	62	52.669	11649	270				
	63	54.797	14700	456				
TOTAL			42567048	1738384			0	

ⓑ -2 の DATA

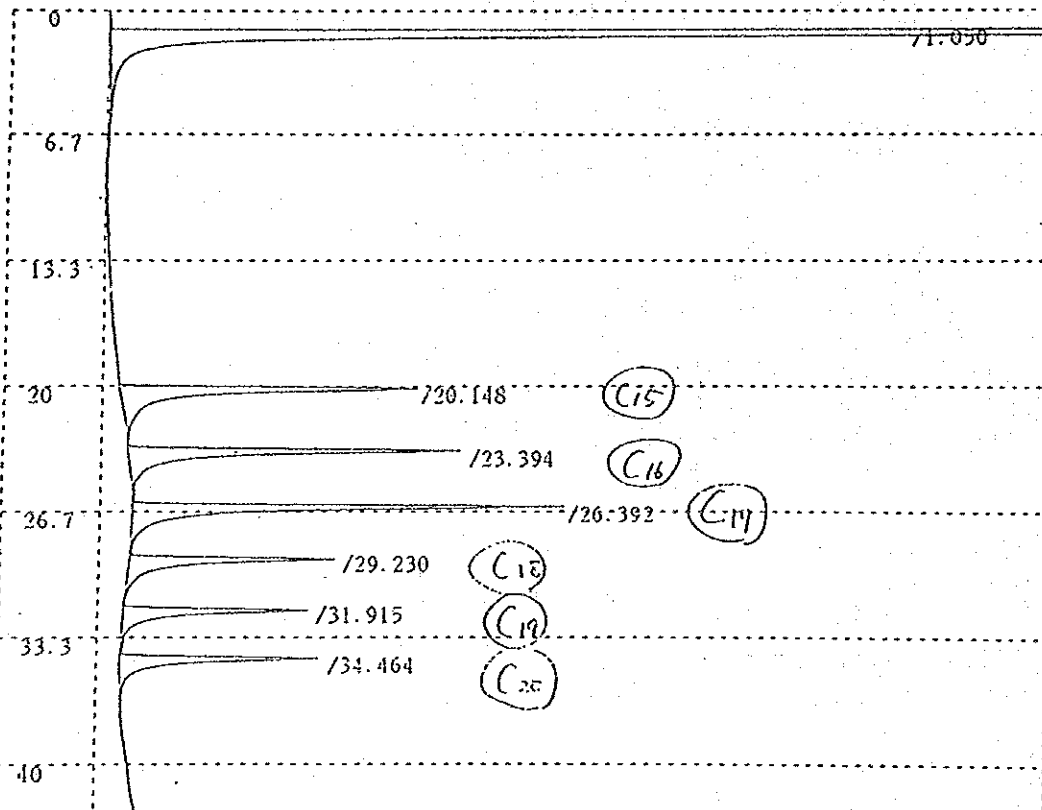
(B) - 3



91015  
Skimmed

\*\* 定量計算結果 \*\*

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	2	1.05	10706914	1386623	E			
	3	4.37	303332	23408				
	4	7.723	248211	11032				
	5	12.409	341481	14734				
	7	16.52	182673	7636				
TOTAL			11782609	1443432			0	



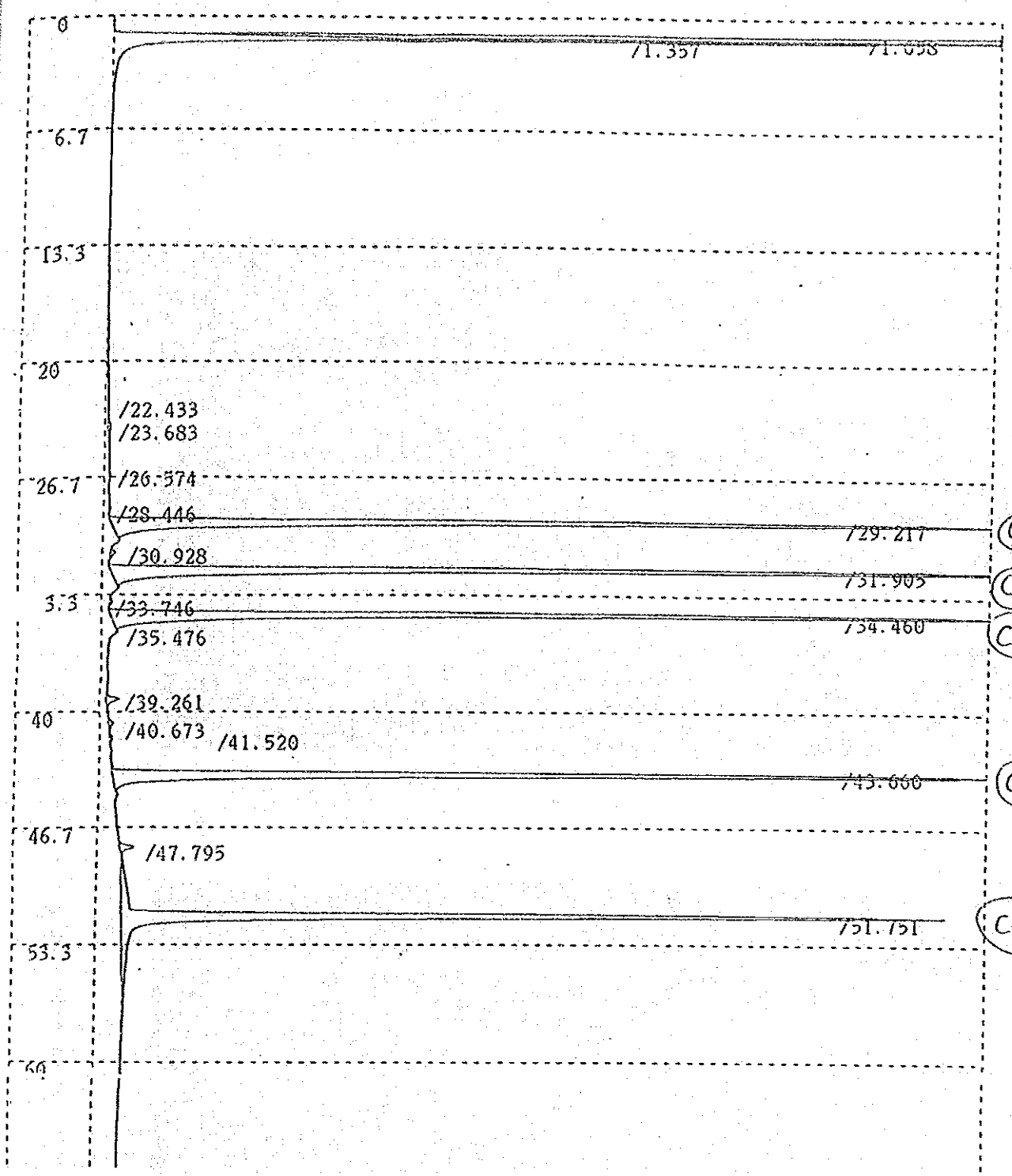
069  
223-02000-11  
91015

\*\* 定量計算結果 \*\*

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	5	1.05	11422233	1386705	E			
	55	20.148	63924	2498				
	56	23.394	65727	2787				
	57	26.392	82878	3536				
	58	29.23	40875	1710				
	59	31.915	35796	1549				
	60	34.464	37549	1650				
TOTAL			11748980	1400534			0	



(B) - 4



定量計算結果 \*\*

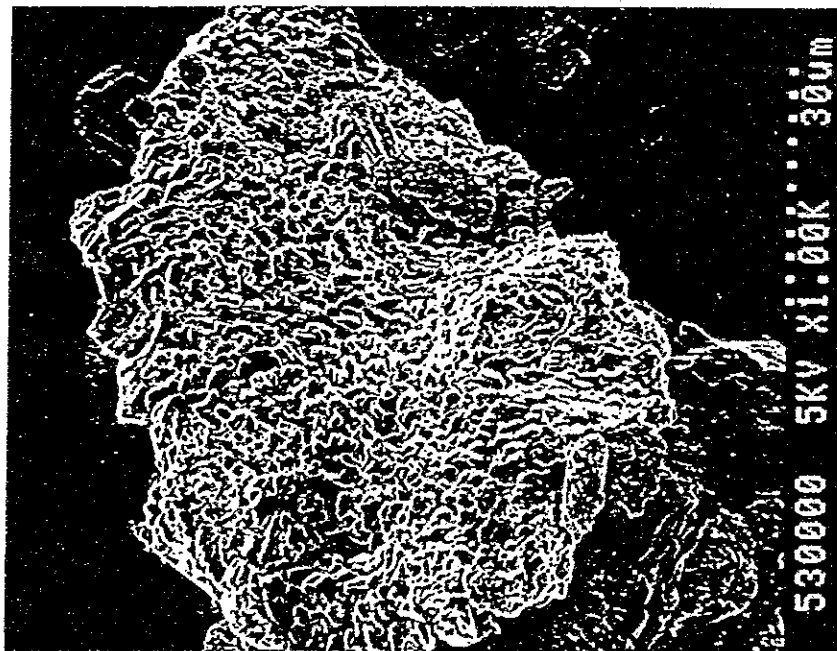
PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
2	1.058	10896319	1385126	E			
3	1.357	475112	144738				
28	22.433	1518	1				
30	23.683	2538	62				
32	26.574	1477	53				
34	28.446	1362	54				
35	29.217	546548	33312				
37	30.928	3249	183				
38	31.905	588972	36568				
39	33.746	1742	111				
40	34.46	572831	34495				
41	35.476	1118	96				
43	39.261	7501	419				
44	40.673	3198	159				
45	41.52	1648	85				
47	43.66	550944	32603				
49	47.795	8611	512				
50	51.751	597310	29018				

070

223-02000-11

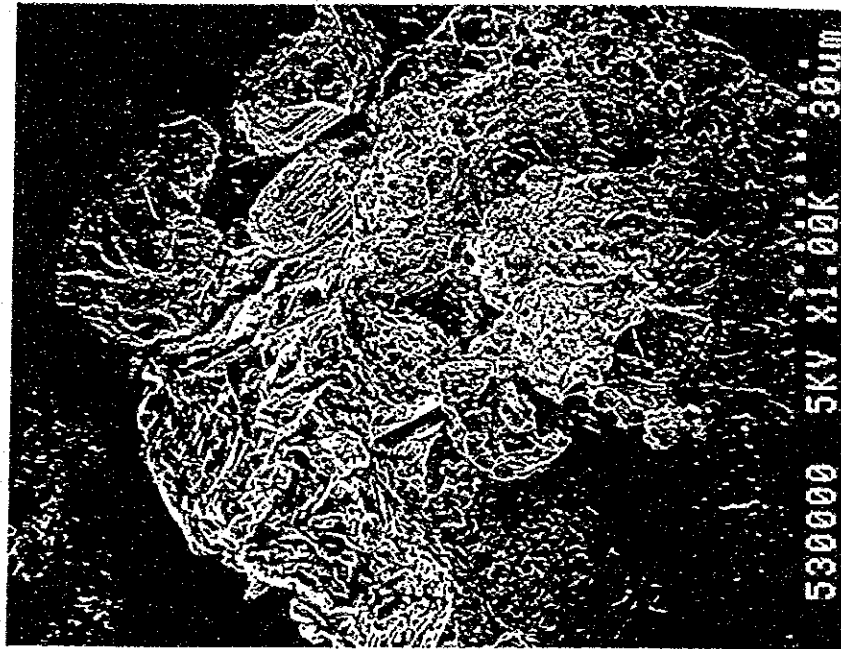
910:15

Shimadzu



a)

X1000

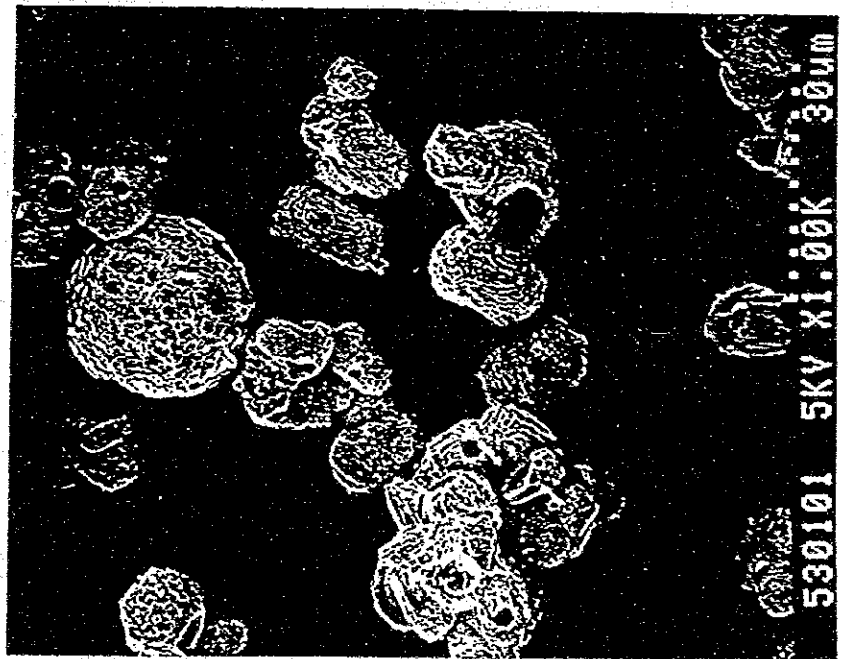


b)

X1000

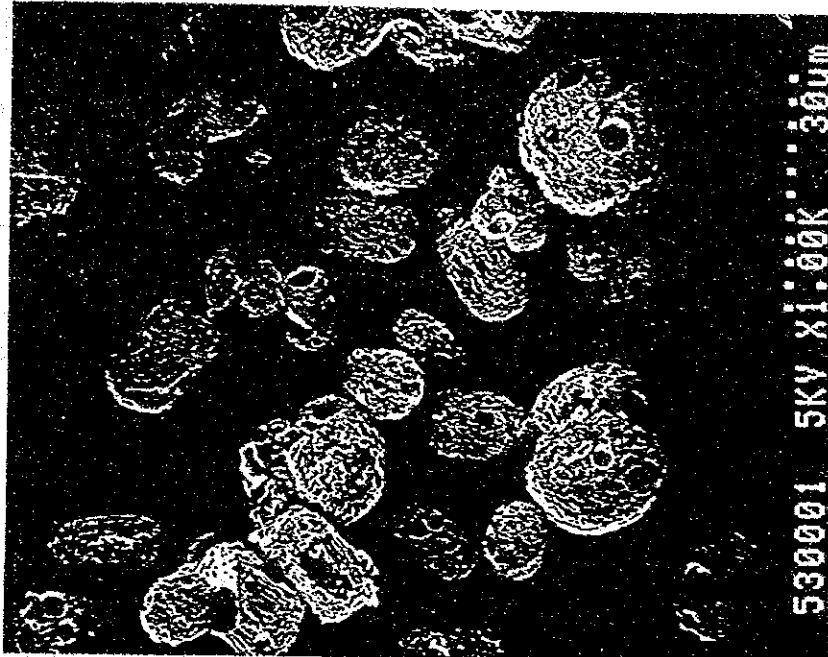
写真1

試料1のSEM観察結果 (ブライイン+Na<sub>2</sub>CO<sub>3</sub>, 1991-10-17)



a)

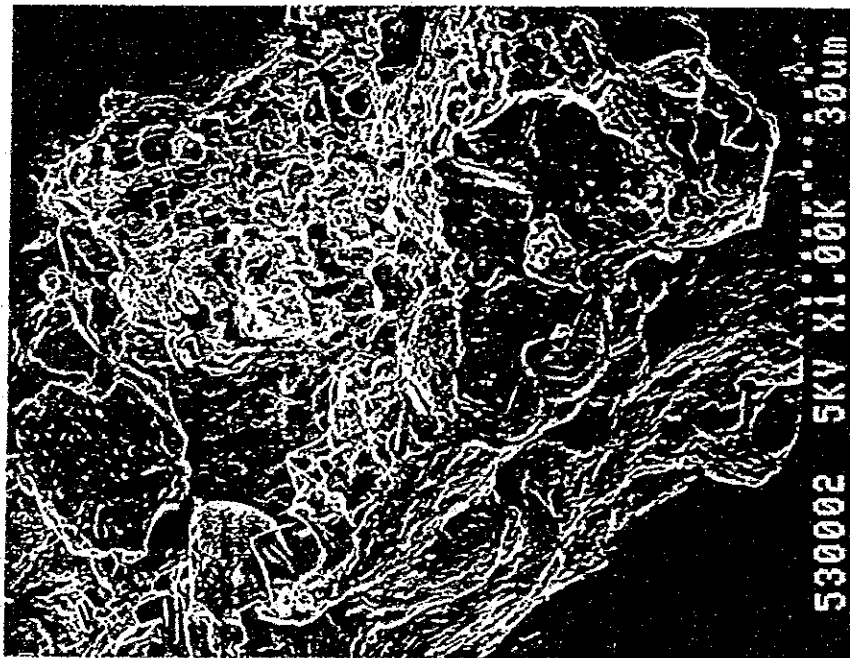
X1000



b)

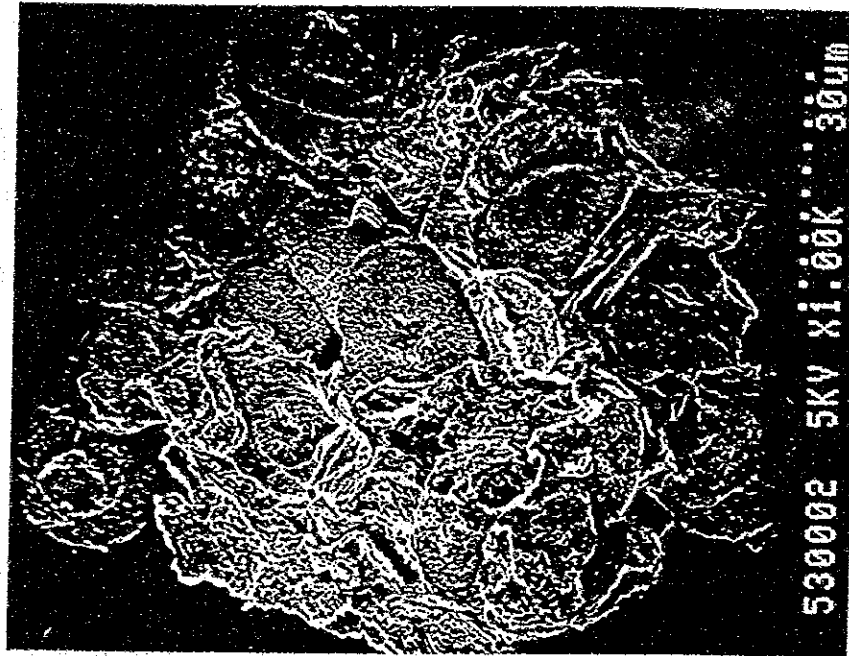
X1000

写真 2 試料 2 の SEM 観察結果 (フライン + A 重油 + Na<sub>2</sub>CO<sub>3</sub>, 1991-10-17)



a)

×1000

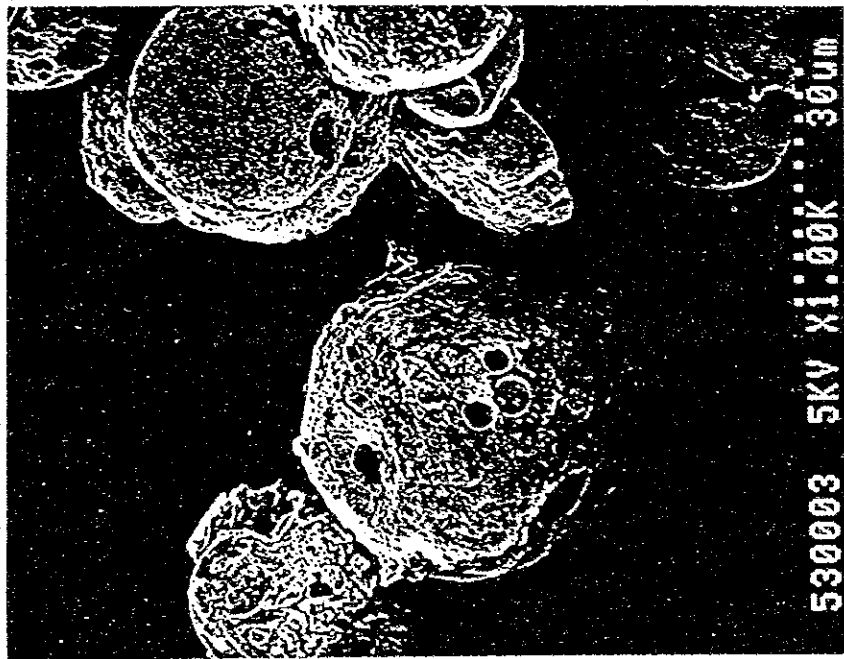


b)

×1000

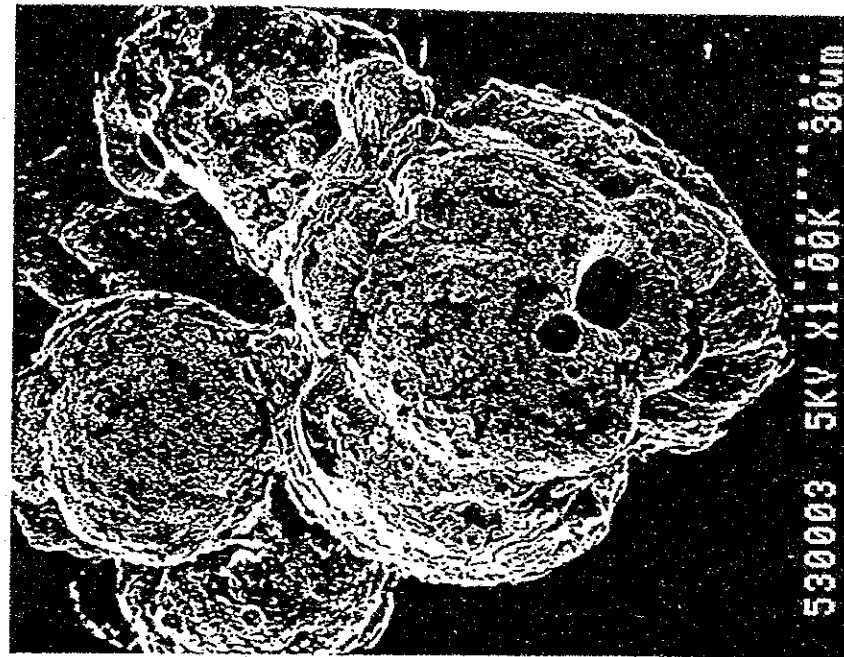
写真 3

試料 3 の SEM 観察結果 (ブライン + BESSIEE-200 + Na<sub>2</sub>CO<sub>3</sub>, 1991-10-17)



a)

x1000



b)

x1000

写真4 試料4のSEM観察結果 (ブライン+A重油+BEHGENE-200+Na<sub>2</sub>CO<sub>3</sub>, 1991-10-17)

写真4



## M 1 - 4 実験研究推進のための配布資料

M 1 - 4 - 1 実験スケジュール

M 1 - 4 - 2 実験方法

M 1 - 4 - 3 実験データ記録用紙

M 1 - 4 - 4 ミーティング資料

M 1 - 4 - 5 報告作成スケジュール

M 1 - 4 - 6 報告書作成時における技術的ポイント

M 1 - 4 - 7 報告書目次





DETAILED SCHEDULE ON RESEARCH THEMES(M-1)

ITEM	NOVEMBER			DECEMBER		
	1	10	20	30	10	20
1 Meeting *1) with Dr. El-Sayed	△ <sup>2</sup>					
2 Planning *2) ① Test condition ② Test Procedure ③ Analysis Method	2 — 3					
3 Preparation *3) ① Experiment 1) Instrument • Assembling • Trial 2) Conc. sea water sample ② Analysis *4) 1) Apparatus 2) Reagents 3) Calibration	4 — 13 4 — 10 5 — 10 △ <sup>11</sup>					
4 Implementation *5)		16 — 4				
5 To prepare SEM- photograph				7 — 10		
6 Preliminary Report *1)				7 — 18		
7 Collecting/Reading information *1)						(To do at January 1992)

(NOTE)

- Dr. El-Sayed will participate in the conference during 25-27 November.
- Mr. Monazir Imam will go back own country during 7-30 November.

AGENDA ON NOV. 2

(M-1)

- \*1) ① Instrument apparatus and reagents needed for experiment  
To use your Research Center equipment if necessary except specific instruments
- ② Collection of information and reading  
More emphasis on collection of materials for your practice or its own accumulated so far
- ③ Preparation for preliminary report  
To recommend that you are to prepare a preliminary report in reference to the inception report
- \*2) ① To discuss phenomena concerning precipitation & prevention of alkaline scale from the sea water in dynamic/static field
- ② Reconfirmation of test condition and analysis method for m-Alkalinity, pH etc.
- \*3) Apparatus transported from YANBU  
Rotary evaporator etc.
- \*4) Analytical item: EC, m-Alk, pH, Cl, Ca, Mg in the brine
- \*5) Test condition

Temperature(°C)	95		80	50
Additives dosing rate(ppm)	0	5	5	5
Oil concentration(ppm)	0	0.1, 10, 100	0.1, 10, 100	1, 10, 100

(Total 12 condition)

## TEST PROCEDURE FOR ITEM M-1

## 1. Preparation of brine

- ① Put 800ml of the raw sea water into the rotary evaporator( $S_R$ ).
- ② Concentrated\*1) it up to 571ml, evaporating by flashing under less than  $60^\circ\text{C}$  ( $S_{RB}$ ).
- ③ Prepare 11l of the brine in total and keep it in the polyethylene bottle with faucet.

2. Preparation of  $\text{Na}_2\text{CO}_3$  solution for m-alkalinity control.

Prepare 1/2N  $\text{Na}_2\text{CO}_3$  solution (Dissolve 25.15g of  $\text{Na}_2\text{CO}_3$  with pure water and make its volume to 1 liter)

## 3. Precipitation test

## 3.1 Test conditions (12 conditions)

Test NO.	Sb*2	S1-1~S1-4	S2-1~S2-4	S3-2~S3-4
Temperature( $^\circ\text{C}$ )		95	80	50
Additives dosing rate(ppm)	0	5	5	5
Oil concentration(ppm)	0	0, 1, 10, 100	0, 1, 10, 100	1, 10, 100

## 3.2 Apparatus

Refer to Fig 3.1 (hereinafter referred to as "FLASK").

## 3.3 Procedure

## 3.3.1 Blank test

- ① Put 800ml of the test sample into the FLASK.
- ② Raise the temperature of the test sample up to  $95^\circ\text{C}$  in the oil bath heated in advance.
- ③ Keep refluxing for 30 min and observe if some precipitation occurs.

\*1) Concentration factor= $S_R/S_{RB}=1.4$

\*2) Blank test

- ④ If not, add the specified amount\*<sup>1</sup> of Na<sub>2</sub>CO<sub>3</sub> solution and keeping refluxing for 1 hour.
- ⑤ Immediately\*<sup>2</sup>, filter it with filtering paper(No.5C), then take the filtrate and the precipitate (B<sub>b</sub>, S<sub>b</sub>).

### 3.3.2 Main test

#### (1) S1-1, S2-1

- ① Put 800ml of the test sample with 4mg of additives into the FLASK for each condition of temperature and stir.
- ② Raise the temperature of the test sample up to 95 and 80°C respectively in the oil bath heated in advance.
- ③ Keep refluxing for 30 min, and observe if some precipitation occurs.
- ④ If not, add the specified amount\*<sup>1</sup> of Na<sub>2</sub>CO<sub>3</sub> solution and keep refluxing for 1 hour.
- ⑤ Immediately\*<sup>2</sup>, filter it with filtering paper(No.5C), then take the filtrate and the precipitate (B<sub>11</sub>, B<sub>21</sub>, S<sub>11</sub>, S<sub>21</sub>).

#### (2) S1-2~4, S2-2~4, S3-2~4

- ① Add 0.8, 8, and 80mg of oil with 4mg of additives to 800ml of the test sample for each condition of temperature.
- ② Stir each test sample for 20sec with a mixer and put it into the FLASK.
- ③ Raise the temperature of the test sample up to 95, 80 & 50°C respectively in the oil bath before hand, and observe if some precipitation occurs.
- ④ If not, add the specified amount\*<sup>1</sup> of Na<sub>2</sub>CO<sub>3</sub> solution and keep refluxing for 1 hour.
- ⑤ Immediately\*<sup>2</sup>, filter it with filtering paper(No.5C), then take the filtrate and the precipitate (B<sub>12</sub>, B<sub>13</sub>, B<sub>14</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub>, B<sub>22</sub>, B<sub>23</sub>, B<sub>24</sub>, S<sub>22</sub>, S<sub>23</sub>, S<sub>24</sub>, B<sub>32</sub>, B<sub>33</sub>, B<sub>34</sub>, S<sub>32</sub>, S<sub>33</sub>, S<sub>34</sub>).

\*1 : CASE 1 ( $C_{Cl}$  in  $S_{RB}/C_{Cl}$  in  $S_R$ ) = 1.4 >  $C_{Alk}$  in  $S_{RB}/C_{Alk}$  in  $S_R$

$$V_{R-CaCO_3} = (1.4 C_{Alk} \text{ in } S_R - C_{Alk} \text{ in } S_{RB}) 0.8 / (25.15 \times 10^3)$$

Where,  $V_{R-CaCO_3}$  : Minimum volume of 1/2N Na<sub>2</sub>CO<sub>3</sub> Solution (l)

$C_{Cl}$  : Concentration of chlorine ion (mg/l)

$C_{Alk}$  : Concentration of m-Alkarity (CaCO<sub>3</sub> mg/l)

0.8 : Sampling volume of  $S_{RB}$  (l)

25.15 × 10<sup>3</sup> : Concentration of Na<sub>2</sub>CO<sub>3</sub> solution (CaCO<sub>3</sub> mg/l)

CASE 2 ( $C_{Cl}$  in  $S_{RB}/C_{Cl}$  in  $S_R$ ) = 1.4 =  $C_{Alk}$  in  $S_{RB}/C_{Alk}$  in  $S_R$

THIS WILL BE PRESENTED LATER.

\*2 : Before temperature drops to the limits where the precipitate redissolve again

### 3.4 Measurement

#### 3.4.1 Temperature

- Cooling water inlet ( $T_{\text{CW-IN}}$ ) (°C)
- Cooling water outlet ( $T_{\text{CW-OUT}}$ ) (°C)
- Oil bath ( $T_o$ ) (°C)
- Test sample in the FLASK ( $T_B$ ) (°C)
- Temperature difference ( $\Delta T = T_o - T_B$ ) (°C)

#### 3.4.2 Volume and weight

- $B_b$  (l)
- $B_{11}, B_{21}$  (l)
- $B_{12}, B_{13}, B_{14}, B_{22}, B_{23}, B_{24}, B_{32}, B_{33}, B_{34}$  (l)
- $S_b$  (g)
- $S_{11}, S_{21}$  (g)
- $S_{12}, S_{13}, S_{14}, S_{22}, S_{23}, S_{24}, S_{32}, S_{33}, S_{34}$  (g)

#### 3.4.2 Analysis of constituents

- Sample B = pH(25°C), m-Alk, Cl, Ca, Mg (mg/l)
- Sample S = Cl as NaCl (mg/g)
- Ca as  $\text{CaCO}_3$  (mg/g)
- Mg as  $\text{Mg(OH)}_2$  (mg/g)

### 4. Microscopic observation of precipitate

Select the most appropriate specimen among sample S for examination with SEM to be done at the Research Institute of the University of Petroleum and Minerals.

### 5. Evaluation of results

THIS WILL BE PRESENTED LATER.



RECORDING SHEET FOR TEST(1) OF M-1 EXPERIMENT

Date: , , 1991

Tests No.	S	Temp.	°C	Additives Conc.	mg/l μ l/l	Oil Conc.	mg/l μ l/l	* Note	
No.	Subjects	Sym.	Measuring items	Unit	Results				* Note
1	Formation of p. p. t	pH <sub>S</sub>	pH before heating	25°C*					change into 25 °C  as CaCO <sub>3</sub>  as CaCO <sub>3</sub>  as CaCO <sub>3</sub>
2		pH <sub>F</sub>	pH of filtrate	25°C*					
3		Ak <sub>S</sub>	m-Alk before heating	mg/l*					
4		Ak <sub>A</sub>	m-Alk added	mg/l*					
5		Ak <sub>F</sub>	m-Alk of filtrate	mg/l*					
6		t <sub>o</sub>	Oil bath temperature	°C					=t <sub>o</sub> -t <sub>B</sub>
7		t <sub>B</sub>	Brine temperature	°C					
8		Δt	Temp. difference	°C					
9		T <sub>S</sub>	Starting time	hr	°	'	t=t <sub>B</sub>		
10		T <sub>M</sub>	Time after X min.	hr	°	'	X= min		
11		T <sub>F</sub>	Time after Y min.	hr	°	'	Y= min		
12		V <sub>NA</sub>	V. of Na <sub>2</sub> CO <sub>3</sub> sol. added	l					
13		V <sub>B</sub>	V. of filtrate	l					
14		W <sub>1S</sub>	W. of ppt. after dry'g	g					
15	Results of observa- tion by eyes			—	Sharp	Round	S+R	Adhere	

(NOTE) • Ak<sub>A</sub> = (53) (0.94) (10<sup>3</sup>) V<sub>NA</sub> / 0.8 [mg/l as CaCO<sub>3</sub>]  
 • Concentration of Na<sub>2</sub>CO<sub>3</sub> solution: 1N  
 • To dry at 105°C

COMMENTS	
	Signature

RECORDING SHEET FOR TEST(2) OF M-1 EXPERIMENT

Date: . . . 1991

Tests No	S -	Temp.	°C	Additives Conc.	mg/l μ l/l	Oil Conc.	mg/l μ l/l	* Note
No	Subjects	Sym.	Measuring items	Unit	Results			
1		V <sub>B</sub>	V. of filtrate	l		change into 25 °C as CaCO <sub>3</sub>		
2		pH <sub>F</sub>	pH of filtrate	25°C*				
3		Ak <sub>F</sub>	m-Alk of filtrate	mg/l*				
4		Cl <sub>F</sub>	Cl ion in filtrate	mg/l				
5	Quantity of Ca <sup>2+</sup> Mg <sup>2+</sup> in filtrate	Ca <sub>s</sub>	Ca ion before heat'g	mg/l		CaCO <sub>3</sub>  Mg(OH) <sub>2</sub>		
6		Ca <sub>F</sub>	Ca ion in filtrate	mg/l				
7		Ca <sub>P1</sub>	Ca <sub>s</sub> -Ca <sub>F</sub>	mg/l				
8		W1 <sub>Ca</sub>	2.5(Ca <sub>P1</sub> )(V <sub>B</sub> )	mg *				
9		Mg <sub>s</sub>	Mg ion before heat'g	mg/l				
10		Mg <sub>F</sub>	Mg ion of filtrate	mg/l				
11		Mg <sub>P1</sub>	Mg <sub>s</sub> -Mg <sub>F</sub>	mg/l				
12		W1 <sub>Mg</sub>	2.4(Mg <sub>P1</sub> )(V <sub>B</sub> )	mg *				
13	Quantity of CaCO <sub>3</sub> & Mg(OH) <sub>2</sub> in ppt	W1 <sub>S</sub>	W. of ppt. after dry'g	mg		W1 <sub>S</sub> W1 <sub>NC</sub>		
14		W1 <sub>Cl</sub>	W. of Cl after dry'g	mg				
15		W1 <sub>NC</sub>	W. of NaCl	mg				
16		W2 <sub>S</sub>	W. of actual ppt	mg *				
17			W. of Ca	mg				
18		W2 <sub>Ca</sub>	W. of CaCO <sub>3</sub>	mg				
19			W. of Mg	mg				
20		W2 <sub>Mg</sub>	W. of Mg(OH) <sub>2</sub>	mg				



## MATERIALS(1) FOR #3 M-GROUP MEETING

30 NOV. 1991

## THE SUMMARY OF TEST RESULTS FOR RESEARCH THEMES:M-1

## 1. Viewpoint of the M-1 test

• The degree( $\Delta\text{ALK}_F$ ) of decrease in supersaturated m-Alkalinity

## 2. Test results

TEST No.	TEMP.	CONC. OF ADDITIVES	CONC. OF OIL	M-ALKALINITY			NOTE
				*1 ALK <sub>S</sub>	*2 ALK <sub>F</sub>	*3 $\Delta\text{ALK}_F$	
	°C	ppm	ppm	ppm as CaCO <sub>3</sub>			
S1-b	95	0	0	154.1	112.7	0	
S1-1	95	5	0	154.1	98.9	-13.8	
S1-2	95	5	1	154.1	108.1	-4.6	
S1-4	95	5	100	154.1			
S1-5	95	10	100	154.1			⑤ *5
S1-6	95	5	0	154.1			⑥ *6
S2-b	80	0	0	154.1	108.1	0	
S2-1	80	5	0	154.1	210.5	102.4	
S2-2	80	5	1	154.1	151.8	43.7?	‡4)
S2-4	80	5	100	154.1	211.6	103.5?	‡4)
S2-5	80	3	0	154.1			⑦ *5
S2-3	80	5	10	154.1			● *5
S3-b	50	0	0	154.1	197.8	0	
S3-1	50	5	0	154.1	218.5	20.7	
S3-2	50	5	1	154.1	207.0	9.2	
S3-4	50	5	100	154.1			

- NOTE #1) ALK<sub>S</sub>:m-Alkalinity of brine before heating  
 #2) ALK<sub>F</sub>:m-Alkalinity in filtrate of samples obtained by adding  
 1N NaCO<sub>3</sub> solution for break of supersaturated condition.  
 #3)  $\Delta\text{ALK}_F = (\text{ALK}_F \text{ of S-1} \sim \text{S-6}) - (\text{ALK}_F \text{ of S-b})$   
 #4) Marks ? indicate the re-experiment conditions  
 #5) Marks ● indicate the experiment conditions supplemented  
 #6) Reaction time(Y)= min.

### 3. Evaluation of test results until now

1) At 95°C, while concentration of ADDITIVES is 5ppm, no threshold effect is found.

2) At 80°C, a remarkable threshold effect is found in case where concentration of ADDITIVES is 5 ppm. However, in existence of 1ppm of oil, the threshold effect may be considerably reduced.

If this is the fact, it would be worthy of the greatest result for M-1. At present, there are contradiction in two data obtained. A careful retest will be needed to make it clear.

3) At 50°C, supersaturation state of sea water can be kept and even in existence of oil, there are no changes in the threshold effect by ADDITIVES.

THE SUMMARY OF TEST RESULTS FOR RESEARCH THEMES:M-1  
(INCLUDED THE ADDITIONAL TEST CONDITION)

4 DEC. 91  
12 DEC. 91

TEST NUMBER	REACTION TIME		TEMP. °C	CONC. OF ADDITIVES ppm	CONC OF OIL ppm	M-ALKALINITY			BLNK
	min					ALK <sub>s</sub>	ALK <sub>F</sub>	ΔALK <sub>F</sub>	
						ppm as CaCO <sub>3</sub>			
S1b1	60		95	0	0	154.1	112.7	0	
S1b2	5		95	0	0	181.7	455.4	0	
S1b3	5		95	0	100	181.7			
S101	60		95	5	0	154.1	98.9	-013.8	S1b1
S102	60		95	5	1	154.1	108.1	-004.6	S1b1
S104	60		95	5	100	154.1	94.3	-018.4	S1b1
S105	60		95	10	100	154.1	140.0	27.3	S1b1
S106	5		95	5	0	154.1	515.0	59.6	S1b2
S107	5		95	5	1	181.7	388.7	-066.7	S1b2
S108	5		95	5	100	181.7	213.9	-241.5	S1b2
S1b4	17		95	0	0	181.7		0	
S109	17		95	5	0	181.7	218.5		S1b3
S110	17		95	5	1	181.7	174.8		S1b3
S111	17		95	5	100	181.7	186.3		S1b3
S112	60		95	3	100	181.7			
S113	5		95	10	100	181.7			
S2b1	60		80	0	0	154.1	108.1	0	
S201	60		80	5	0	154.1	210.5	102.4	S2b1
S202	60		80	5	1	154.1	131.1	23.1	S2b1
S202	60		80	5	1	154.1	151.8	43.7	S2b1
S204	60		80	5	100	154.1	211.6	103.5	S2b1
S204	60		80	5	100	154.1	193.2	85.1	S2b1
S205	60		80	3	0	154.1	89.9	-018.2	S2b1
S203	60		80	5	10	154.1	138.0		S2b1
S206	5		80	5	0	181.7	246.1		S2b1
S207	5		80	5	1	181.7	262.2		S2b1
S208	5		80	5	100	181.7	446.2		S2b1
S3b1	60		50	0	0	154.1	197.8	0	
S301	60		50	5	0	154.1	218.5	20.7	S3b1
S302	60		50	5	1	154.1	207.0	9.2	S3b1
S304	60		50	5	100	154.1	174.8	-23.0	S3b1

THE SUMMARY OF TEST RESULTS FOR RESEARCH THEMES:M-1  
(INCLUDED THE ADDITIONAL TEST CONDITION)

IMPORTANT POINTS

The higher residual m-alkalinity, the less precipitate.  
This leads to scale prevention.

- (Note) •  $ALK_s$  : m-Alk before heating (No precipitation occur.)  
•  $ALK_F$  : m-Alk of filtrate = Residual alkalinity  
•  $\Delta ALK_F$ :  $ALK_F - (ALK_F \text{ obtained by tests indicated in column:BLNK})$   
• BLNK : Test number selected for a comparison  
• Calculated alkalinity in case of adding 1N  $Na_2CO_3$  solution is as shown below.

$$\{(50)(10^3)V_{NA}/V_s + ALK_s\} \text{ ppm as } CaCO_3.$$

Where,  $(50)(10^3)$ : Alkalinity of 1N  $Na_2CO_3$  solution [ppm as  $CaCO_3$ ]

$V_{NA}$  : Amount of 1N  $Na_2CO_3$  added [l]

$V_s$  : Amount of brine before heating [l]

1. Effect of temperature

TEST NUMBER	REACTION TIME		TEMP. °C	CONC. OF ADDITIVES ppm	CONC OF OIL ppm	M-ALKALINITY			BLNK
	min					ALK <sub>s</sub>	ALK <sub>F</sub>	ΔALK <sub>F</sub>	
						ppm as CaCO <sub>3</sub>			
S1b1	60		95	0	0	154.1	112.7	0	
S101	60		95	5	0	154.1	98.9	0	
S102	60		95	5	1	154.1	108.1	0	
S104	60		95	5	100	154.1	94.3	0	
S2b1	60		80	0	0	154.1	108.1	- 4.6	S1b1
S201	60		80	5	0	154.1	210.5	111.6	S101
S202	60		80	5	1	154.1	131.1	23.0	S102
S202	60		80	5	1	154.1	151.8	43.7	S102
S204	60		80	5	100	154.1	211.6	117.3	S104
S204	60		80	5	100	154.1	193.2	98.9	S104
S3b1	60		50	0	0	154.1	197.8	85.1	S1b1
S301	60		50	5	0	154.1	218.5	119.6	S101
S302	60		50	5	1	154.1	207.0	98.9	S102
S304	60		50	5	100	154.1	174.8	80.5	S104

## 2. Effect of reaction time

TEST	REACTION TIME		TEMP.	CONC. OF ADDITIVES	CONC OF OIL	M-ALKALINITY			BLNK
	NUMBER	min				°C	ppm	ppm	
S1b2	5	95	0	0	181.7	455.4	0		
S106	5	95	5	0	154.1	515.0	0		
S107	5	95	5	1	181.7	388.7	0		
S108	5	95	5	100	181.7	213.9	0		
S1b4	17	95	0	0	181.7	131.1	-324.3	S1b2	
S109	17	95	5	0	181.7	218.5	-296.5	S106	
S110	17	95	5	1	181.7	174.8	-213.9	S107	
S111	17	95	5	100	181.7	186.3	-27.6	S108	
S1b1	60	95	0	0	154.1	112.7	-342.7	S1b2	
S101	60	95	5	0	154.1	98.9	-417.6	S106	
S102	60	95	5	1	154.1	108.1	-280.6	S107	
S104	60	95	5	100	154.1	94.3	-119.6	S108	

## 3. Effect of Additives concentration

TEST	REACTION TIME		TEMP.	CONC. OF ADDITIVES	CONC OF OIL	M-ALKALINITY			BLNK
	NUMBER	min				°C	ppm	ppm	
S1b3	5	95	0	100	181.7	179.4	0		
S112	5	95	3	100	181.7	216.2	36.8	S1b3	
S108	5	95	5	100	181.7	213.9	34.5	S1b3	
S113	5	95	10	100	181.7	216.2	36.8	S1b3	

(ALK<sub>F</sub> in 60min., 80°C, 3ppm Additives and non-oil of Test No.:S205 is 89.9 ppm as CaCO<sub>3</sub>)

4. Effect of oil concentration

TEST NUMBER	REACTION TIME		TEMP. °C	CONC. OF ADDITIVES ppm	CONC OF OIL ppm	M-ALKALINITY ppm as CaCO <sub>3</sub>			BLNK
	min					ALK <sub>s</sub>	ALK <sub>F</sub>	ΔALK <sub>F</sub>	
S106	5		95	5	0	154.1	515.0	0	
S109	17		95	5	0	181.7	218.5	0	
S101	60		95	5	0	154.1	98.9	0	
S107	5		95	5	1	181.7	388.7	-126.3	S106
S110	17		95	5	1	181.7	174.8	-43.7	S109
S102	60		95	5	1	154.1	108.1	9.2	S101
S108	5		95	5	100	181.7	213.9	-301.1	S106
S111	17		95	5	100	181.7	186.3	-32.2	S109
S104	60		95	5	100	154.1	94.3	-4.6	S101
S101	60		95	5	0	154.1	98.9	0	
S201	60		80	5	0	154.1	210.5	0	
S301	60		50	5	0	154.1	218.5	0	
S102	60		95	5	1	154.1	108.1	9.2	S101
S202	60		80	5	1	154.1	131.1	-79.4	S201
S202	60		80	5	1	154.1	151.8	-58.7	S201
S302	60		50	5	1	154.1	207.0	-11.5	S301
S203	60		80	5	10	154.1	138.0		
S104	60		95	5	100	154.1	94.3	-4.6	S101
S204	60		80	5	100	154.1	211.6	1.1	S201
S204	60		80	5	100	154.1	193.2	-17.3	S201
S304	60		50	5	100	154.1	174.8	-32.2	S301
S206	5		80	5	0	181.7	246.1	0	
S207	5		80	5	1	181.7	262.2	16.1	S206
S208	5		80	5	100	181.7	446.2	200.1	S206
S106	5		95	5	0	154.1	515.0	0	
S107	5		95	5	1	181.7	388.7	-126.3	S106
S108	5		95	5	100	181.7	213.9	-301.1	S106

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)		Discuss contents of report with Dr. Essam	Check report	Test for brushing up of report
17	DEC.	1991		(Goto INDIA)			
18	DEC.	1991					
19	DEC.	1991			(Holiday)	(Holiday)	(Holiday)
20	DEC.	1991	Make report *Introduction		(Holiday)	(Holiday)	(Holiday)
21	DEC.	1991	*Theory			Check report	Make report *Planning
22	DEC.	1991	*Discussion	Brush up report	Check report of M-4 and discuss contents of M-1 report with Saad	Preparation of transportation	*Exp. method
23	DEC.	1991	*Conclusion *Reference *Acknowledge.				*Exp. result
24	DEC.	1991					
25	DEC.	1991					
26	DEC.	1991			(Holiday)	(Holiday)	(Holiday)
27	DEC.	1991			(Holiday)	(Goto YANBU)	(Holiday)
28	DEC.	1991					
29	DEC.	1991				SWCC YANBU FACILITY	
30	DEC.	1991					
31	DEC.	1991			Receive report from Dr. Basu	(Go back to JUBAIL)	
1	JAN.	1992			(Holiday)	(Holiday)	(Holiday)
2	JAN.	1992			(Holiday)	(Holiday)	(Holiday)
3	JAN.	1992			Brush up report with Dr. Essam and Dr. Basu	Check report	
4	JAN.	1992	(Go back to SWCC)				
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)		





実験のPlanningにおいて記載する最大のPoint

Alhammadh's idea

- Additives の効果が油の存在によって、どの様に阻害されるかを定量的に把握することである。
  - Additives の効果には Threshold effect と Crystal distortion とがあるが、前者は換言すると、化学量論的にPrecipitation が起こるべき状態をそれが起こらない状態に保つ効果、即ち過飽和状態を保つ効果であり、後者は例えPrecipitation が起きて、その結晶の形状を伝熱面に付着しない様に変形させる効果である。
  - 後者については、PrecipitateのSEMによる写真観察により行う。
  - 前者については、次の手順でAdditives のThreshold effectを明らかにする。
- 1) Concentration Factor 1.4 (Actual plants におけるそれと同じ値) でもPrecipitation が発生しない温度(60°C) でRaw Sea Water を1.4 倍に濃縮する。

この状態でBrine は過飽和状態で安定である。(Stability Index= )

- 2) この状態のBrine に $\text{Na}_2\text{CO}_3$ を添加してm-Alkalinityを最低値まで減少させる。

この状態でBrine は飽和状態となる。

- 3) この状態のBrine にさらに $\text{Na}_2\text{CO}_3$ を添加していくと、それは全て $\text{CaCO}_3$  のPrecipitationの生成に消費されることになる。そして $\text{CaCO}_3$  の溶解度積に従い、m-Alkalinityは上昇する。
- 4) しかしm-Alkalinityの上昇程度はAdditives の存在及びその滞留時間(反応時間)によって、また油の存在の有無によって大きく異なる。

これらの関係を明らかにすることが本実験の最大のPoint である。

THE MOST IMPORTANT POINT TO BE REFERED IN PLANNING FOR EXPERIMENTS

Alhammadh's idea

- To grasp quantitatively how effect of Additives is retarded due to existence of oil.
  - Effect of Additives involves the threshold effect and the crystal distortion. The former represents an effect which prevents precipitation being broken up stoichiometrically, that is to keep supersaturation. The latter represents an effect which distorts configuration of crystals so that adherence to heat transfer tube wall may not break up even if precipitation occurs.
  - To evaluate the latter based on SEM examination for precipitate.
  - To make threshold effect of Additives clear according to the following procedure.
- (1) To concentrate 1.4 times raw sea water at the temperature(60°C) where no precipitation occurs in case of concentration factor 1.4 simulated to actual plants.

In this state, brine is kept stable in supersaturation. (Stability Index= )

- (2) To decrease m-alkalinity at minimum by adding  $\text{Na}_2\text{CO}_3$  to the above brine.

In this state, the brine is in saturated condition.

- (3) All amount of  $\text{Na}_2\text{CO}_3$  further added to the above brine may be consumed for formation of  $\text{CaCO}_3$  precipitates. According to solubility product of  $\text{CaCO}_3$ , m-alkalinity increase.
- (4) However, the extent of m-alkalinity increase varies too much due to existence of Additives or oil, retention time (reaction time) and temperature.

Intercorrelation between different parameters (Temp., Time, Additives concentration, Oil concentration and m-Alkalinity) is the most important point in this study.



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	
	<ul style="list-style-type: none"> <li>• Dr. Essam E. F. El-Sayed</li> <li>• Saad Al Sulami</li> <li>• Tadatsugu Hamada</li> </ul>	<ul style="list-style-type: none"> <li>• Dr. A. A. Nomani</li> <li>• Yoshio Hamao</li> <li>• Tadatsugu Hamada</li> </ul>
1.	Introduction	
	<ul style="list-style-type: none"> <li>• On basis of outline of Introduction in inception report</li> <li>• In addition to above, refer to it that scale prevention has something close to do with decision for plants size in design.</li> </ul>	<ul style="list-style-type: none"> <li>• On basis of outline of Introduction in inception report</li> <li>• In addition to above, refer to application of suitable method for oil analysis.</li> </ul>
2.	Theory(This title could be changed to more appropriate one. Result of literature survey shall be included.)	
	<ul style="list-style-type: none"> <li>• Technological explanation concerning acid &amp; chemical dosing methods (To emphasise importance of alkalinity for water conditioning. To explain threshold effect and crystal distortion in chemical dosing method)</li> <li>• Fouling factor peculiar to methods above. (On basis of measurement in operating MSF plant in Saudi-Arabia)</li> </ul>	<ul style="list-style-type: none"> <li>• 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)</li> <li>• To present analytical methods for oil in water. To explain advantage or disadvantage for each. To suggest effectiveness of GC/MS.</li> </ul>
3.	Planning and procedure of experiments	
	<ul style="list-style-type: none"> <li>• Experiment conditions</li> <li>• Measurement items</li> <li>• Experiment apparatus (e. g. configuration)</li> <li>• Experimental procedure</li> </ul>	
4.	Results	
	<div style="border: 1px solid black; padding: 5px; display: inline-block;">INCOMPLETION</div>	

### 3. EXPERIMENT PROCEDURES

#### 3.1 PLANNING

(Already explained)

#### 3.2 EXPERIMENT CONDITIONS

(Prepare Tables for experiment conditions)

#### 3.3 EXPERIMENT PROCEDURES

(To explain the following contents)

- Sampling location and quality of raw sea water --(Map or Figure & Table)
- To prepare 1.4 times of raw sea water as TDS (brine), where no ppt occur at 60°C
- Intercorrelation between m-alkalinity and 1N Na<sub>2</sub>CO<sub>3</sub> addition in the above brine
- To make it sure that no ppt was broken out even if the brine was kept for 30 min. at the temperature specified in the experiment conditions
- To break out ppt by 1N Na<sub>2</sub>CO<sub>3</sub> addition and then kept the temperature and the duration, specified in the experiment conditions
- Separation method of ppt
- To divide ppt for composition analysis and SEM examination
- To search for correlation between composition of ppt and existence of oil, together with mass balance check from analytical data for filtrate and ppts

### 4. EXPERIMENT RESULTS

(Prepare the following tables and figures)

<TABLE>

- Quality of the brine before and after tests
- Table for evaluation of effect of temperature
- Table for evaluation of effect of reaction time
- Table for evaluation of effect of Additives concentration
- Table for evaluation of effect of oil concentration
- Analytical data of ppt

<FIGURE>

- Correlation between 1N Na<sub>2</sub>CO<sub>3</sub> addition and m-alkalinity
- Correlation between residual m-alkalinity and temperature
- Correlation between residual m-alkalinity and reaction time
- Correlation between residual m-alkalinity and Additives concentration
- Correlation between residual m-alkalinity and oil concentration
- Correlation between ppt occurrence and residual m-alkalinity
- Correlation between composition of ppt and oil concentration
- Others

## 5. DISCUSSION

### 5.1 Evaluation on Effect of Additives

The aims of this experimental research are to examine and compare by carrying out laboratory experiments, the properties of the additives currently used to prevent scaling, when oil is present in the sea water.

Here, at first, authors wish to review the evaluation on effect of Additives used in this experiment. As stated in chapter 2, Additives have two effects i.e. ① threshold effect ② crystal distortion. In other words, ① represents to easily keep super-saturated state and ② represents to inhibit scaling to tube wall by changing crystalline configurations.

It is unlikely possible to identify for ② in this experiment. This will be discussed afterward, based upon the results of SEM examination. ① can be made sure by analysis for water quality, namely, in case where threshold effect come into force more M-Alkalinity than critical concentration for  $\text{CaCO}_3$  ppt, which is stoichiometrically calculated. It is difficult to estimate the amount of  $\text{CaCO}_3$  ppt from m-alkalinity change especially from data which are obtained in a test unit where fluid continuously flow in and out, but in case of a batch test like this experiment where the amount of precipitation for m-alkalinity is known.

### 5.2 Evaluation of precipitation by residual m-alkalinity

From the above point of view, regarding the amount of ppt as value obtained by subtraction of residual m-alkalinity from ones in the initial state. An analysis results for influence of each factors on residual m-alkalinity ( $\text{ALK}_F$ ) are shown in Fig. 5\*\*\*-5\*\*\*.

#### 5.2.1 Effect of Retention Time on m-alkalinity ( $\text{ALK}_F$ )

The plot in Fig. 5\*\*\* show the correlation between  $\text{ALK}_F$  and retention time i.e. 5min, 17min, and 60min. after the moment when ppt is forced to occur by addition of 1N  $\text{Na}_2\text{CO}_3$  into brine prepared in advance to be 5ppm of additives (initial alkalinity is 1,000+181.7ppm as  $\text{CaCO}_3$ ).

First, in 5min test, comparing blank value (•) to 5ppm(+) for additives the bigger  $\text{ALK}_F$  in the latter represents that additives is retarding the growth of ppt. But in the presence of oil, ppt is likely to easily grow (m-alkalinity to reduce), especially in case of 100ppm oil, reduction rate of m-alkalinity reaches to 3 times of no oil.

On the other hand, as retention time longer, the above difference in m-alkalinity reduces gradually. In 60min. test, m-alkalinity at every condition converges in around 100ppm as  $\text{CaCO}_3$ .

It suggests that both additives and oil have something little to do with ppt stoichiometrically. This, especially for additives, supports the established theory. And also it would be identified for oil, being no affinity with water, as easily anticipated.

As far as 60min's concerned, the presence of oil has nothing to do with occurrence of ppt, and effect of additives will be limited to crystal distortion.

Actually, there can not be such a state as the above. The retention time of brine in the actual plants (20,000t/d class) is considered to be 2 or 3min. The most important data would be for 5min test.

Of course the occurrence of ppt cannot directly connect with increase of fouling factor. No matter how much ppt may occur, if additives effectively work on crystal distortion, scaling rate to heat transfer tubes will be retarded. However, it is sure that occurrence of ppt is the primary factor to increase fouling factor. Such viewpoint as possibility on initiation of precipitation by oil is important.

Evaluating 5 min. data from the above point of view, it could be stated that the presence of oil becomes the cause of promotion of precipitation and the higher oil concentration the much ppt.

Cases where taking retention time of brine into account are of S. Steinberg et al other than this experiment.

[WILL BE EXPLAIN BY DR. ESSAM ABOUT  
RESULT OF THE DATA-ANALYSIS WHICH  
BE CARRIED OUT BY S. STEINBERG ET AL]

Fig. 5.1 Effect of retention time  
on quantity of scale precipi-  
tated

Fig. 5.1 shows correlation between retention time and quantity of scale precipitated. In Fig. 5.1, it is clear that there is a close relation between precipitation rate and retention time at the presence of additives.

This trend is similar to this experiment.

And it is easily anticipated that the extend of increase in precipitation rate with progress of retention time varies due to the kind of additives.

Keen competitions on sale of many kind of additives have been extended in SADI-ARABIA, where there are the biggest demands in the world, and in Japan who has biggest products for MSF plant in the world.

The present circumstances make author perplexed to choose a more suitable ones. Taking it into consideration, it is thought that evaluation in this experiment is an effective way being its simplicity and low-price. It is needed to make its effectiveness sure by systematic tests in future.

### 5.2.2 Effect of Additives Concentration on Alkalinity

With plots, correlation between residual m-Alkalinity after 5 min. while additives contact with ppt, and concentration of additives at 95°C and 100ppm of oil is shown in Fig.5.\*\*\*. From the results, it is clear that more than 3ppm of additives are no use to prevent ppt. This is well coincident with the practice of 3 or 4 ppm additives at Al-Jubail Phase-2 plants. It proves by chance that scaling prevention at this plant has been effectively and economically controlled. And also it leads to prove propriety of this evaluation.

### 5.2.3 Effect of Temperature on Alkalinity

Fig.5\*\*\* shows correlation between residual m-alkalinity after 60min while additives contact with ppt, and temperatures at 5ppm of additives, 1 and 100ppm at oil together with a plot of blank value. These data were obtained by 60min test which has been done on a hypothesis that contacting time of additives with ppt, would not have so much influence on m-alkalinity. Strictly speaking, 5min test should be done.

However, high temperature stage has an importance effect of temperature on scaling. Now that those data have already obtained as stated in 5.2.1 and it takes much time to prepare brine, an experiment was suspended this time. The following can be concluded from the above data.

- 1) Oil has not so much effect on m-alkalinity at 50°C and 60min. of retention time. the higher concentration of oil, the lower residual m-alkalinity. Oil promotes growth of ppt to some extent.
- 2) At, 80°C and 60°C of retention time, while almost the same extent of decrease for m-alkalinity as 50°C, was found under free of oil, the higher oil concentration, the more m-alkalinity decrease compared to 50°C.
- 3) At 95°C and 60min. of retention time, each m-alkalinity decrease to a certain level, independent of presence of additives and oil concentration.

### 5.2.4 Effect of Oil Concentration on m-alkalinity

Fig.5\*\*\* shows correlation between residual m-alkalinity and oil concentration at 5min. and 60min. of retention time with parameter of temperature. Evaluation on this is made in 5.2.1 to 5.2.3.

Reference to the fact that particle size of ppt becomes very small under 100ppm of oil should be made. Some of ppt have passed through No.5C filter paper in the case where experimental condition is 100ppm of oil concentration, 80°C and 60min. of retention time. That is the reason why filtrate indicate an abnormal value in m-alkalinity under S204 of experimental condition. 0.45 micron filter was used in the case where S104 of experimental condition is 95°C, 100ppm of oil and 60min. of retention time.

Therefore, such a plot is neglected when drawing correlation curves.

5.3 Evaluation on Crystal Distortion by SEM examination.

5.4 Evaluation on Fouling Factor in the case where sea water is contaminated by oil

6 Conclusion

7 Recommendation for future work

8 References

9 Acknowledgements



RECORDING SHEET FOR TEST(1) OF M-1 EXPERIMENT

Date: 17. Nov 1991

Tests No	S	Temp.	°C	Additives Conc.	mg/l μl/l	Oil Conc.	mg/l μl/l	Note
	1-61		95					
No	Subjects	Sym.	Measuring items	Unit	Results			# Note
1	Formation of p.p.t	pH <sub>s</sub>	pH before heating	25°C	7.685 (22)			change into 25 °C
2		pH <sub>F</sub>	pH of filtrate	25°C	8.505 @ 25.3			
3		AK <sub>s</sub>	m-Alk before heating	mg/l	154.1 @ pH = 4.17			as CaCO <sub>3</sub>
4		AK <sub>A</sub>	m-Alk added	mg/l	1027.5			as CaCO <sub>3</sub>
5		AK <sub>F</sub>	m-Alk of filtrate	mg/l	112.7 @ pH=4.15			as CaCO <sub>3</sub>
6		t <sub>o</sub>	Oil bath temperature	°C	107			
7		t <sub>B</sub>	Brine temperature	°C	95			
8		Δt	Temp. difference	°C	12			=t <sub>o</sub> -t <sub>B</sub>
9		T <sub>s</sub>	Starting time	hr	8°20'	t=t <sub>B</sub>		
10		T <sub>M</sub>	Time after X min.	hr	8°50'	X=30min		
11		T <sub>F</sub>	Time after Y min.	hr	9°50'	Y=60min		
12		V <sub>NA</sub>	V. of Na <sub>2</sub> CO <sub>3</sub> sol. added	l	0.01625			
13		V <sub>B</sub>	V. of filtrate	l	0.796			
14		W <sub>s</sub>	W. of ppt. after dry'g	g	0.796			
15		Results of observation by eyes				Sharp	Round	S+R
					Yes			Yes

(NOTE) •  $AK_A = (53)(0.94)(10^3)V_{NA} / 0.8$  [mg/l as CaCO<sub>3</sub>]  
 • Concentration of Na<sub>2</sub>CO<sub>3</sub> solution: 1N  
 • To dry at 105°C

COMMENTS \* Volume of filtrate has decreased significantly from 816ml [actual] to 796 ml [measured]. Some of this loss is due to sample spoiling in fume hood during filtration.

*[Signature]*  
Signature

\* Chloride was not measured.

RECORDING SHEET FOR TEST(2) OF M-1 EXPERIMENT

Date: 17. Nov 1991

Tests No	S <sub>b</sub>	Temp.	95 °C	Additives Conc.	0 mg/l μl/l	Oil Conc.	0 mg/l μl/l	* Note
No	Subjects	Sym.	Measuring items	Unit	Results			
1		V <sub>B</sub>	V. of filtrate	l	0.796			change into 25 °C as CaCO <sub>3</sub>
2		pH <sub>F</sub>	pH of filtrate	25 °C	8.505 @ 25.3			
3		Al <sub>F</sub>	m-Alk of filtrate	mg/l	112.7 @ pH=4.15			
4		Cl <sub>F</sub>	Cl ion in filtrate	mg/l	33.365			
5	Quantity of Ca <sup>2+</sup> Mg <sup>2+</sup> in filtrate	Ca <sub>s</sub>	Ca ion before heat'g	mg/l	630			CaCO <sub>3</sub>
6		Ca <sub>F</sub>	Ca ion in filtrate	mg/l	253			
7		Ca <sub>P1</sub>	Ca <sub>s</sub> - Ca <sub>F</sub>	mg/l	377			
8		Wl <sub>Ca</sub>	2.5(Ca <sub>P1</sub> )(V <sub>B</sub> )	mg	754			
9		Mg <sub>s</sub>	Mg ion before heat'g	mg/l	1967			
10		Mg <sub>F</sub>	Mg ion of filtrate	mg/l	1875			
11		Mg <sub>P1</sub>	Mg <sub>s</sub> - Mg <sub>F</sub>	mg/l	92			
12		Wl <sub>Mg</sub>	2.4(Mg <sub>P1</sub> )(V <sub>B</sub> )	mg	177			
13	Quantity of CaCO <sub>3</sub> & Mg(OH) <sub>2</sub> in ppt	Wl <sub>s</sub>	W. of ppt. after dry'g	mg	796			Wl <sub>s</sub> - Wl <sub>NC</sub>
14		Wl <sub>Cl</sub>	W. of <del>Cl</del> after dry'g	mg	9.8			
15		Wl <sub>NC</sub>	W. of NaCl	mg	25			
16		W2 <sub>s</sub>	W. of actual ppt	mg	771			
17			W. of Ca	mg	S 275		A 169	
18		W2 <sub>Ca</sub>	W. of CaCO <sub>3</sub>	mg	768			
19			W. of Mg	mg	S 13		A 62	
20		W2 <sub>Mg</sub>	W. of Mg(OH) <sub>2</sub>	mg	144			

RECORDING SHEET FOR TEST(1) OF M-1 EXPERIMENT

Date: 9.20.1991

Tests No	S	Temp	°C	Additives Conc.	mg/l μl/l	Oil Conc.	mg/l μl/l	Note	
	162		95		0		0		
No	Subjects	Sym.	Measuring items	Unit	Results			* Note	
1	Formation of p.p.t	pH <sub>s</sub>	pH before heating	25°C	7.653 @ 23.1				change into 25°C
2		pH <sub>F</sub>	pH of filtrate	25°C	8.125 @ 2.3				
3		AK <sub>s</sub>	m-Alk before heating	mg/l	181.7 @ pH=4.16				
4		AK <sub>A</sub>	m-Alk added	mg/l	996.4				as CaCO <sub>3</sub>
5		AK <sub>F</sub>	m-Alk of filtrate	mg/l	455.4 @ pH=4.18				as CaCO <sub>3</sub>
6		t <sub>o</sub>	Oil bath temperature	°C	7.1				
7		t <sub>B</sub>	Brine temperature	°C	95				
8		Δt	Temp. difference	°C	16				= t <sub>o</sub> - t <sub>B</sub>
9		T <sub>s</sub>	Starting time	hr	9°45'	t = t <sub>e</sub>			
10		T <sub>M</sub>	Time after X min.	hr.	10°15'	X = 30 min			
11		T <sub>F</sub>	Time after Y min.	hr	10°20'	Y = 5 min			
12		V <sub>NA</sub>	V. of Na <sub>2</sub> CO <sub>3</sub> sol. added	l	0.01				
13		V <sub>B</sub>	V. of filtrate	l	0.49				
14		W <sub>s</sub>	W. of ppt. after dry'g	g	0.357				
15		Results of observation by eyes			Sharp	Round	S+R	Adhere	
				✓			✓		

(NOTE) • AK<sub>A</sub> = (53)(0.94)(10<sup>3</sup>)V<sub>NA</sub> / 0.8 [mg/l as CaCO<sub>3</sub>]  
 • Concentration of Na<sub>2</sub>CO<sub>3</sub> solution: 1N  
 • To dry at 105°C

COMMENTS	adhere on the lower hemisphere.
	Signature

wt of #5 filter paper = 1.124g  
 " " " " + ppt = 1.467g } → 0.343g  
 wt of millipor = 0.088g  
 " " " " + ppt = 0.102g } → 0.014g  
 wt. of ppt for Ca & Mg = 0.00331g

RECORDING SHEET FOR TEST(2) OF M-1 EXPERIMENT

Date: . . . 1991

Tests Na	S <sub>1</sub> -b <sub>2</sub>	Temp.	°C	Additives Conc.	mg/l 0 μl/l	Oil Conc.	mg/l 0 μl/l	# Note
No	Subjects	Sym.	Measuring items	Unit	Results			
1		V <sub>B</sub>	V. of filtrate	l	0.49		change into 25° C as CaCO <sub>3</sub>	
2		pH <sub>F</sub>	pH of filtrate	25°C	8.125 @ 2.2-3			
3		AK <sub>F</sub>	m-Alk of filtrate	mg/l	45.5.9 @ pH = 4.18			
4		Cl <sub>F</sub>	Cl ion in filtrate	mg/l	31,042			
5		Ca <sub>s</sub>	Ca ion before heat'g	mg/l	630			
6	Quantity of Ca <sup>2+</sup> Mg <sup>2+</sup> in filtrate	Ca <sub>F</sub>	Ca ion in filtrate	mg/l	318		CaCO <sub>3</sub>	
7		Ca <sub>P1</sub>	Ca <sub>s</sub> - Ca <sub>F</sub>	mg/l	312			
8		W <sub>1Ca</sub>	2.5(Ca <sub>P1</sub> )(V <sub>B</sub> )	mg	390			
9		Mg <sub>s</sub>	Mg ion before heat'g	mg/l	1967			
10		Mg <sub>F</sub>	Mg ion of filtrate	mg/l	1902			
11		Mg <sub>P1</sub>	Mg <sub>s</sub> - Mg <sub>F</sub>	mg/l	65			
12		W <sub>1Mg</sub>	2.4(Mg <sub>P1</sub> )(V <sub>B</sub> )	mg	78			
13		Quantity of CaCO <sub>3</sub> & Mg(OH) <sub>2</sub> in ppt	W <sub>1s</sub>	W. of ppt. after dry'g	mg	357		Mg(OH) <sub>2</sub>
14	W <sub>1c1</sub>		W. of <sup>Na</sup> Cl after dry'g	mg	41			
15	W <sub>1NC</sub>		W. of NaCl	mg	104			
16	W <sub>2s</sub>		W. of actual ppt	mg	253			
17			W. of Ca	mg	<sup>s</sup> 68	<sup>A</sup> 221		
18	W <sub>2Ca</sub>		W. of CaCO <sub>3</sub>	mg	361			
19			W. of Mg	mg	<sup>s</sup> 72	<sup>A</sup> 9		
20	W <sub>2Mg</sub>		W. of Mg(OH) <sub>2</sub>	mg	97			

RECORDING SHEET FOR TEST(1) OF M-1 EXPERIMENT

Date: 6 Dec. 1991

Tests No.	S <sub>1</sub> - b <sub>3</sub>	Temp.	95 °C	Additives Conc.	0 mg/l μ l/l	Oil Conc.	100 mg/l μ l/l	† Note		
No.	Subjects	Sym.	Measuring items	Unit	Results					
1	Formation of p.p.t	pH <sub>s</sub>	pH before heating	25 °C	7.653 @ 23.1		change into 25 °C			
2		pH <sub>F</sub>	pH of filtrate	25 °C	8.567 @ 22.2					
3		AK <sub>s</sub>	m-Alk before heating	mg/l	181.7 @ pH = 4.5		as CaCO <sub>3</sub>			
4		AK <sub>A</sub>	m-Alk added	mg/l	1000		as CaCO <sub>3</sub>			
5		AK <sub>F</sub>	m-Alk of filtrate	mg/l	179.4 @ pH = 4.17		as CaCO <sub>3</sub>			
6		t <sub>o</sub>	Oil bath temperature	°C	108					
7		t <sub>B</sub>	Brine temperature	°C	94					
8		Δt	Temp. difference	°C	14		= t <sub>o</sub> - t <sub>B</sub>			
9		t <sub>S</sub>	Starting time	hr	8°00'	t = t <sub>B</sub>				
10		t <sub>M</sub>	Time after X min.	hr	8°30'	X = 30 min				
11		t <sub>F</sub>	Time after Y min.	hr	8°35'	Y = 5 min				
12		V <sub>NA</sub>	V. of Na <sub>2</sub> CO <sub>3</sub> sol. added	l	0.01					
13		V <sub>B</sub>	V. of filtrate	l	0.488					
14		W <sub>s</sub>	W. of ppt. after dry'g	g	0.475					
15		Results of observation by eyes				Sharp	Round	S+R	Adhere	
						✓		✓		

(NOTE) • AK<sub>A</sub> = (53)(0.94)(10<sup>3</sup>)V<sub>NA</sub> / 0.8 [mg/l as CaCO<sub>3</sub>]  
 • Concentration of Na<sub>2</sub>CO<sub>3</sub> solution: 1N  
 • To dry at 105 °C

COMMENTS	
	Signature

wt of #5 filter paper = 1.119 g  
 " " " + ppt = 1.583 g → 0.464  
 wt of millipore = 0.036 g  
 " " " + ppt = 0.097 g → 0.11  
 wt of 100 mesh CaCl<sub>2</sub> = 0.00440