Monsanto

→ Howard Bergen

FROM ANNISTON, ALABAMA

AUG 8 1969

DATE July 30, 1969

SUBJECT TRIP REPORT - CED Job 1668
MMK Yokkaichi CPH* Plant Start-Up

REFERENCE 8 June - 17 July, 1969
*MMK refers to Aroclor Plant as CPH Plant (Chlorinated Polyphenyl)

TO: D. Danna (3)
ST. LOUIS

cc: J. R. Durland - MMK Tokyo
V. R. Haupt
W. A. Kuhn - WKUHN
H. L. Williams
R. S. Yates - RYATE

SUMMARY:

The June - July MMK Yokkaichi Plant, Aroclor start-up, demonstrated a new site production unit by producing Aroclors 1242, 1248, and 1254. The very satisfactory start-up highlighted good basic design, equipment selection, instrumentation, and an effective preliminary effort with laboratory, training, and equipment preparation. The system produced specification material at approximately 90% of design rate. At this writing, there were no major operation problems. There were, however, a number of minor problems, some of which are listed in the report.

A summary meeting was held at Yokkaichi by Plant Manager, Mr. Murakami. The plant's major current concerns were: varying stability analysis of Aroclor 1242, lack of sufficient design information to give complete engineering service to new users of Santotherm, and the need for additional specification information for the Biphenyl project. Mr. Murakami considered the CPH start-up successful and gave the plant's thanks for the overall MC effort. A summary report was given the MMK Board of Directors, and thanks for the MC effort was given by MMK President, T. Nishikawa, and J. R. Durland.

Permission to take pictures of the operating CPH plant was given by Mr. Murakami. These will allow CED to see the results of their design manual and perhaps help on related projects. A set has been forwarded to Mr. Danna.

Every consideration was given by MMK personnel to make the MC representative's stay a pleasent one.

DSW 199864

OUTLINE OF MAJOR AREA

I. Pre-Design Contact

The adequate MC background effort prior to design was well done and contributed in a major way in the production of this workable manufacturing unit.

Some of this effort is listed below:

What	MMK	Where	Time
1) Laboratory Training	N. Arasaki K. Yamamoto	Anniston Laboratory (G. Miller)	2-1/2 Months
2) Engineering Liaison	M. Okamura	CED (Hinze-Yates) WGK (TSD-Prod.) Anniston (TSD & Production)	Approx. 3 Wks.
3) Production Liaison	M. Tuska jima	Anniston (TSD & Production) WGK (TSD-Prod.)	Approx. 7 Wks.

The understanding barrier, due to language and terminology, etc., requires as much face to face discussion with engineering flow sheets and in-field observations as can be arranged. To minimize this step is to invite major problems in the final unit. In this case, the very observant technically competent MMK personnel with a great deal of MC cooperation, produced a first rate plant. In my opinion, for a complex unit, a CED visit with the MMK design-production team after their definitive design would insure that all know-how had been effectively transmitted.

II. Yokkaichi Observations (See Slides)

A. Plant Location - New site reclaimed land 250 yards from ship channel, all on concrete pilings. Biphenyl unit now in process of being reclaimed.

B. Layout & Safety- Very good from operation, safety, maintenance and limited expansion. MMK use open sewers and recovery traps (recovery trap we should have).

C. Equipment - Good selection - no major problem.

D. Instrument - Good performance - followed CED recommendations.

E. Design Info. - Good - I heard no complaints.

DSW 199865

STLCOPCB4057929

F. Construction - (See Slides) The equipment and lines were insulated and painted; structure, erector set, looked good. No line leaks while on site. All seals worked, no leaks in vacuum system. Most instruments functional and controlled. Insulation covering enameled steel sheeting. All open sewers. Engineering liaison on site. Construction very responsive. Contractors used to keep MMK employe level (900 men) stable. For example, MMK will use contractors to load bagged biphenyl into melter if production personnel not available. One complaint, step treads too short for representative's feet.

G. Operation - Manual — Good - they followed with great care (V. R. Haupt).

H. Pre-Start-up - Planning and Training — They wrote the Book.

I. Manpower - Plan to operate with 2 operators per shift until biphenyl on-stream then one operator plus supervisor, foreman, engineer, T.S. engineer (temp.), laboratory analyst (2), secretary.

J. Production - Approximately 90% of design. Cycle lengthened by B.O.S. to give longer catalyst dissolving time.
Chlorination: 8:1242; 7: 1248; 7: 1254

K. Yield - Matl. Bal. (See Attach.) — 1242 run poor - due to calibration and tank and line filling. 1242-1254 good (97% +) All chlorine, HCl, biphenyl and Aroclor accounted for. No actual losses.

L. Quality (See Attach. Analyses) — Fair to good. Physical Properties in spec.
Electrical - too good. Stability & Inorganic Cl - mixed.

M. Bottleneck (See Bottleneck analyses) — Chlorinator was operated at maximum rate based on free chlorine in HCl. MMK may have to reduce chlorine flow above 1248 gravity.

Next: Jet capacity
Still furnace (can be increased by minor jet change)

N. Research — MMK did a lot of pre-start-up research work on preparation, adsorbent and analysis. MMK research has responsibility of training control laboratory personnel.
MMK research program for 1970 included the following:

STLCOPCB4057930

1. Chlorinator design.
2. Relation of stability values to any measurable variables in the chlorination steps.
3. Catalyst study to maximize electrical properties.
4. Continued adsorbent study for improved electrical properties.
5. Undefined work for heat exchanger use.

O. Production-T.S.

Future Work:

1. Procedure for recovery of:

   A. Aroclor returned from capacitor plants, which has soldering flux and other contaminations.

   B. Returned Santotherm.

2. Studies on the return of poor stability HCl entrained Aroclors and their effect on stability.

3. Installation and start-up of drum washing operation for new and returned drums.
(see attached sheet)

4. Re-refining Aroclor that analyses above specification on Inorganic chlorides or TCC.

P. Not Tested:

1. Production of 1260 - none needed - 1970.

2. Blending operation.

3. Rework of returned material.

4. Drumming operation - July.

5. Drum washing operation - July-August.

Q. Project Expenditure (Est.)

Underspent ---- all projects that Dr. Durland had handled have been underspent. Only the oil additive project, which was changed after start-up, came up to appropriation level. The Board and Dr. Durland are quite pleased. There is a message here for CED.

DSW 199867

STLCOPCB4057931

III. Problem List* (Equipment and Other)

*No major problems - just getting into process understanding and adjustments.

203 - Biphenyl Melt Tank - Do not have ability to mix contents of tank.

Biphenyl quality 68.7°C (one sample) of Yawata Chemical Com may be variable. Some brown color.

201 - Biphenyl Charge Tank -

May give short charge

202 - Chlorination System -

204 - Air cooler oversized - required manual on-off until full load reached.

Chlorine piping required trace and insulation to prevent Biphenyl freeze-up.

Chlorine not always available due to PVC plant replacement requirements. Note that DeNora cell chlorine was a remarkable 97-98%.

HCl produced from chlorinator above 1148 had excessive free chlorine (see attachment). Chlorine feed rate reduction may be required to eliminate.

Possibility that high capacity pump is pulling chlorine into circulating line.

Return of poor stability (TTC 6.0) material from HCl knock-out tank may contribute to lowered stability.

Defination of stability for Therminol (Santotherm) sales is not clear. MC uses trappings (poor stability) to produce Therminol FR-2 (1248).

HCl storage facilities limited chlorinator run.

204 - Off gas drain pot

Required method to measure level.

Acid containing free chlorine could not be used in back washing ion exchange for water treatment.

207-208- Blow Tank

Not as effective as Anniston in reduction of HCl content. Possibly due to tank diameter and length of sparger.

STLCOPCB4057932

215 - Still System -

Circulating pump 221 had rapid failure of Graphite 100 (Spun Graphite) packing. Plant selected and ordered a mechanical seal.

The two burner on-off temperature control on Furnace 222 was ragged.

The dip leg on separator 216 was not long enough to allow complete boil down before loosing seal.

The spare coil was rolled tight with no space and the coils were welded together (CED recommended against this).

218 - Still Receiver -

The jet operated to maximum capacity with no control, i.e., bleed valve remained closed. Probably undersized.

Inorganic chloride on distilled product varied and were higher than U.S. production. The distillation step would be the most likely cause (i.e., lime contact, still temperature, still vacuum, distillation end point, etc.).

223 - 225 - Feed Tank Adsorption System -
226 - 242 -

No recognized problem
1242 and 1248 processed through Silica Gel.
1254 processed through Porocel since 110°C was above Silica Gel limit.

Open question: Life of Silica Gel packing.

Jim Bryant - Special In line Analyzer: Not placed in operation during run due to electrical problems. Remains Research responsibility to place in operation.

Final product analyses showed variations in Inorganic chlorides and TTC stability. Samples are to be exchanged with WGK and Anniston Chief Chemists.

Biphenyl Project:

1. Site being reclaimed.
2. Layout approved early July.
3. Some equipment on order.
   Some major equipment (i.e. Furnace) being held up to resolve some design problems with CED.
4. Most important that CED give good review of layout and hardware.

DSW 199869

STLCOPCB4057933

5. Schedule May - 1969.

6. Look for problems on putting this one on the road.
I think furnace design should be reviewed with Selas at once - Newport coke may be due to increased heat flux over Anniston (M.P.Lux)

Santowax-HB-40

1. Equipment selection and final cost estimate underway.

2. Scheduled - August 1969.

MMK is very anxious to get HB-40 on market.

3. MMK may send representative to CED and Anniston on this one. Not clear.

Competition-

Biphenyl producers: Primary: Yawata
(New Selas Furnace unit reported to be operating 6-9 months with no problems).

By-Product Biphenyl: Mitsubishi-Yoka
Tokyo - Rayon

HB-40 Type - Yawata
Not yet on market

Aroclor - Kanechlor by Kanegafuchi

MMK will push new heat exchange market.
Will push to exceed competitions quality
(i.e., stability and electrical)

Note: Kawechlor practice is to give credit for returned drums.
MMK will follow this proactice using 3 type drums.

1. Zinc line drum for transfer site.

2. Lined drum (2 coats) electrical

3. Steel drum for Santotherm

Kanegafuchi has serious problem in heat transfer market due to the Rice Oil Case. (See attachment)

Burns Severson

:md
Attachments

DSW 199870

STLCOPCB4057934

Trip Report - Burns Severson
CED Job 1668

Attachments

66 Slides of CPH Plant (D. Danna) sent separately and tape.

1. Chlorination Cycles 1242
2. Analysis - 1242
3. Chlorinator Cycles 1248
4. Blow Tank Resulsts - 1248
5. Analyzer - Receiver - 1248
6. Analysis after treatment 1248- 1254

7.-11. Cycle Analysis to determine bottleneck

12. Annunicator
13. Annunicator
14. Free Chlorine vs. level of chlorination
15. Discussion on use of Purchased By-Product Biphenyl and Hydrogen Clean-Up
16. Drum Cleaning Equipment
17. Bran Oil Poisoning
18. Personnel List - Yokkaichi Plant

DSW 199871

STLCOPCB4057935

CPH Z9-1 元位 1242

CHLORINATION Cycle plus Lab. Tests

1242	A201	A202	A203	A204	A205	A206	A207	A208
Dry wt. 乾燥重量 kg	5000	〃	〃	〃	〃	〃	〃	〃
amount of Cl 使用量 kg	4210	4370	4330	4320	4340	4310	4300	447
100% Cl 換算 kg	1650	1760	1690	1730	1749	1725	1702	1780
time Cl₂ in 通入開始	11⁰⁰	13²⁰	10³²	10⁴²	13⁵²	9³⁵	9⁰⁵	9⁰⁵
200 #	12⁰⁰	14⁴³	11³²	11⁴²	11⁰¹	10³⁵	10⁰¹	10⁰⁵
290 #	13⁰⁰	15²³	12³²	12⁴²	12⁰²	11³⁵	11⁰²	11⁰⁵
Finish 反応終了	17⁴⁵	19⁰⁵	15⁰⁶	16⁰⁰	15⁴⁸	16⁰⁷	14⁴⁶	15⁰⁰
Reaction time 反応時間	6⁴⁵	5⁴⁵	5³²	5¹³	5⁴¹	5³²	5⁴¹	5⁵⁵
Sp. gr. 比重 65°C	1.340	1.347	1.350	1.351	1.349	1.342	1.360	1.361
Date 月日	6.16	6.17	6.18	6.19	6.20	6.21	6.24	6.25
Cool time 冷却時間	9⁴⁵	11⁰⁵	7⁰⁵²	7⁰⁵⁵	6⁰⁵⁸	8⁰⁵⁰	7⁰⁹	7⁰⁵
合計	2 hrs Dustbed + Clay Treat ↓							
d_4^{25}	1.378	1.377	1.392 (1.392)	1.390 (1.390)	1.392 (1.392)			1.401
TCC ppm	1.09 1.12	0.56	2.54	4.54	3.31			2.05
HS ppm	0.25	0.35	0.89	0.89	0.89			0.78
ε	4.99	5.00	4.97	5.67	5.25			4.98
tan δ %	0.4	1.66	0.67	0.68	0.78			0.31
VR $\times 10^{13}$ VR $\times 10^{13}$ Ω-cm	2.9	1.65	2.8	0.73	6.62			1.45

1142

After flow 硬化後のフロー

Acidity 酸度 0.22 mg KOH/gr

Moisture 水分 400 ppm

Acidity 酸度 0.

Moisture 水分 30

Circulating time after cat. add. 触媒添加後循環時間

57' 65' 57' 42' 47' 116' 20' 80

Initial temp. of fd 仕込温度 122° 122° 125° 120° 120° 122° 122° 122°

1 Red Nick 元売掛金回収

17 19 30 EF

Cat. % 触媒量 0.10 0.10 0.07 0.07 0.07 0.07 0.10 0.1

Reaction Temp 反応温度 155° 〃 〃 〃 〃 〃

DSW 199872

STLCOPCB4057936

(2)

Distilled + Treated 1242 From Column And From Storage Tank

9=69
#2 4242
CB = Chlorinated Y

Sample	HC Spec	目標	9/208 6/66 11hr	9/208 1/07 12hr	9/208 5:0	16:30	17:00	17:30
APHA	40 WT	10	5	5	5			
Sp. Gr	1.381~1.392	1.381	1.388	1.389	1.389	1.387	1.387	1.387
酸度 Acidity	0.01 WT	0.01 WT	0.000	0.000	0.000			
水分 Moisture	25 WT	50 WT	5	20	20			
屈折率 R.I	1.6240~1.6260	1.6227	1.6242	1.6245	1.6240	1.6244	1.6245	1.6245
Ind Prod 鉄	0.25 WT		0.114	0.061	0.157	0.047	0.067	0.026
Pour P 流動点	-12°C WT	-14	-15°C	-22°C	-17°C			
ε	4.72~4.90		4.96	4.79	5.01			
x10^13 V.R	0.02×10^13 WT	1	1.65×10^13	5.27	2.58			
HS	0.5 WT	0.5	0.51	0.02	0.61	0.96	0.03	0.03
HS	0.4	0.4						
TCI	0.55	0.5	0.39	1.13	0.22			
dielec range 誘電率範囲	10% 32.4~ WT		326	325	325			
	90% 36.5~		346	345	346			
SO4	ナシ							
P.S	34KV WT		79	63	56			
Flash 引火点	170~200		179	180	186			
Consum 溶解度	Al 0.0 WT		0.0	0.0	0.0			
	APHA 60 WT		15~20	5	5 WT			
	酸度 0.010 WT		0.00	0.00	0.00			
	無機塩 0.05		0.061	0.133	0.103			
Visc 粘度	32~34 SUS		16.26 cst	16.1 cst	16.2	16.1	16.4	16.3
Sp.Heat 比熱	0.3							
PF 100°C	2.3	-	-					
	5.0	0.5	2.18	2.17	6.49			
Evaporation loss 蒸発減量			20 mg		0.03			

DSW 199873

STLCOPCB4057937

1248 Chlorination Data + Properties of Lab Treated

12-8 Batch date Material for Each Batch 69.7.5

		94501	94502	94503	94504	94805	94806	94807	
HB		6.26	7.1	7.1	7.2	7.2	7.3	7.2	-
ウエハー	Kg	3000	〃	〃	〃	〃	〃	〃	-
Cl_2	Kg	5480	5360	5330	4940	5110	5220	5470	5111
100% HCl	Kg	2190	2300	2270	2120	2260	2240	2360	2246
Catalyst	%	0.01 酸化鉄	0.01 〃	0.01 〃	0.01 〃	0.01 〃	0.01 酸化鉄	0.01 酸化鉄	0.01 -
Stadch 145 NIP	hr	9'03	10'00	20'00	8'30	17'05	7'30	17'20	-
200°		10'03	11'00	21'00	9'30	20'05	8'30	18'20	-
290°		11'05	13'00	22'00	10'20	21'05	9'20	19'20	-
Finish (5%)		16'58	16'47	5'33	16'51	1'56	14'10	3'53	-
Reaction time		6'53	6'49	6'33	6'25	6'31	6'40	6'33	6'42
Cycle time From start 200 to temp 207		9'24	9'20	9'25	9'00	11'21	9'53	9'07 1242	9'49 7'13
Starting temp	°C	121	130	127	128	103	130	122	126
Cat. circulation	min	78	75	95	60	85	70	75	77
Reac. Temp	°C	155	157	155	154	156	150	155	156
Sp. gr. 60°C		1.419	1.425	1.424	1.423	1.426	1.401	1.415	1.415

↓ Kneb Completed from Chlorinator Sample

Analysis d_4^{125}			1.455	1.455	1.453	1.454	1.457	1.447	1.452
TCC	%	1.12	0.62	0.71	0.63	0.61			0.79
HS	ppm	0.96	0.66	0.74	0.64	0.29	0.23	0.20	0.69
ε		4.71	4.69	4.72	4.71	4.77	4.77	4.76	4.73
tanδ	50Hz %	0.69	0.39	0.74	1.75	0.24	2.01	1.60	1.23
VR $\times 10^{13}$	Ω-cm	5.4	9.36	4.72	3.07	1.55	1.2	1.26	3.60
SnO_2 Cl	ppm	0.0							

Lab = Line + Dish + Stick Cpl.

Cycle time = 200°C to 290°C

DSW 199874

STLCOPCB4057938

O. Blow Tank - Effect of Blow on Hcl + Hcl recovery

	BEFORE AIR BLOW			AFTER AIR BLOW				
LS-4 NO	酸価 (mg/g)	[illegible]	[illegible] (kg)	酸価	[illegible]	[illegible]	[illegible] (kg)	[illegible]
94201	Acidity mgKOH/gr	HCl mg/gr	Total Acid / Kilo / B	Acidity mgKOH/gr	HCl mg/gr	HCl Kg/B	HCl removed Kg/B	Hcl Recovery % Calc.
94202								
94203								
94204								
94205								
94206								
94207								
94208	1.519	0.98	4.58	0.133	0.086	0.40	4.18	91.4 (%)
94801	1.342	0.87	4.59					
94802	1.143	0.74	3.71	0.177	0.127	0.67	3.24	82.9 (%)
94803	1.313	0.85	4.09	0.189	0.187	0.99	3.50	78.0 (%)
94804	1.195	0.77	4.06					
94805	1.247	0.80	4.23	0.271	0.175	0.87	3.35	79.4 (%)
94806	1.496	0.97	4.11	0.187	0.121	0.64	4.47	87.5 (%)
94807	1.154	0.73	3.66	0.230	0.148	0.78	3.08	79.8 (%)
95401	0.404	0.26	1.53					
95402				0.090	0.058	0.34		
95403				0.208	0.18	1.06		
95404	1.006	[illegible]	9.10					
95405								
95406								
95407								

AIR Flow Rate 160 m³/hr
Blow Temp 120°C ~ 140°C

DSW 199875

STLCOPCB4057939

1248 [illegible] After distillation
(Condenser Outlet)

ITEM 項目	7/2 [illegible] 217 94801~2	7/2 6:15 217 94801~2	7/2 14:40 220-220 94801~2	7/2 20:20 217 94804	7/3 [illegible] 217 94804	7/4 21:15 217 1248 1248	7/4 [illegible] 218 1248 1248	7/4 [illegible] 217 948
PF 1000Hz	0.42	0.87	2.62	0.57			2.72	[illegible]
PF 50Hz								
In. Or. Cl	0.826	0.895	0.251	1.950	0.475		2.114	[illegible]
ε 1000Hz	4.68	4.70	4.70	4.70			4.70	[illegible]
TR $\times 10^{-2}$	7.66	1.79	1.59	17.4			0.375	0.[illegible]
HS [illegible]	0.37	0.33	0.90	0.20	0.22		0.210	0.52
TCC	0.27	0.23					0.66	0.72
水 Moisture	23	29	30	29	34	39	25	62
酸価 Acidity	0.000	0.000	0.001	0.000	0.000	0.000	0.000	0.000
ATHA	5247	〃	〃	〃	5247		5	5247
比重 Sp. Gr					1.110	1.395		
粘度 Viscos								
屈折率 Rf. I								

DSW 199876

STLCOPCB4057940

1248 + 1254 after treatment

(6)

1248 精製 [illegible] (After column treatment)

1248 Silicagel
1254 Porocel

1248 ← 1254

	7/2 17:00	7/3 6:30	7/3 9:30	7/4 8:20	7/4 8:30	7/4 11:15	7/5 13:30	7/7 8:[illegible]
	227	227	227	227	227	227	227	227
項目	94801-2	94803-4	94803-5	94806	94806	94807	94807	94[illegible]
PF 100ml	[illegible]							
PF	0.35	0.36	0.27	[illegible]	0.33	0.38	0.26	0.28
In Or. Q	0.035	0.031	0.045	0.06	0.065	0.060	0.045	0.0[illegible]
Σ	4.68	4.70	4.70	4.75	4.75	4.70	4.72	4.29
[illegible] $\times 10^{12}$	[illegible]	17.4	107	36.7	69.4	43.5	65.0	[illegible]
HS ppm	0.[illegible]	0.20	0.28	0.[illegible]	0.49	0.48	0.35	0.21
TCC			0.49	0.52	0.66	0.30	0.33	0.4[illegible]
moisture	16	18	22	28	26	[illegible]	19	28
[illegible] Acidity	0.000	0.000	0.000	0.000	0.000	0.000	[illegible]	0.000
APHA	5	5.67	"	"	"	5~10	5.[illegible]	5
[illegible] Sp. Gr 63°C			1.001		1.004	1.001		1.50[illegible]
[illegible]								
[illegible]								

DSW 199877

STLCOPCB4057941

7

Analysis of Cycles to Determine Bottle Neck

i.e. Chlorinator

1 Biphenyl 溶融

1 溶解[illegible]

2 [illegible]

2 (Chlorinator)

1 [illegible]

2 Biphenyl 仕込量 [illegible] 8% up

3 HCl [illegible]

高温[illegible]		11C Design	Design
1242	4300 T/y	5000 T/y	11 H
1248	3720 T/y	4560	10
1254		4350	9.6
1260		4140	9.1

[illegible]

1) [illegible]

2) [illegible]

3) [illegible]

4) [illegible]

5) [illegible]

3 [illegible]

1 [illegible]

[illegible]

1242	[illegible]	6000 T/y
1248	[illegible]	5450 T/y
1254		

DSW 199878

STLCOPCB4057942

STILL

	1212	1218	1234
Charge from blow tank	16 min	16 min	16 min
Still pump circulate	5	5	5
Prepare add lime	5	5	5
Lime add	5	5	5
Prepare fire on	5	5	5
Heat up	100	110	141
Distillation			
Boil down	132	130	100
Circulate after fire off	10	10	10
Vacuum release	5	5	5
Mortar	25	25	25
Prepare for next charge	10	10	10
Distillation time	40° [illegible]	46° 12'	
other work except still	5°18'	5°06'	5°37'
	45.3 hr	51.3 hr	
Batches	8	7	6
Finish product kg	86,454	87,600	
Capacity kg/hr	805	732	
Year [illegible]	6000 T/Y	5450 T/Y	

DSW 199879

STLCOPCB4057943

クロリネーション 塩素注入 — CHLORINATION

Big Chart

No.		1242	1248	1254	$\bar{X}$	
1.	シフォン槽入口	0.27	0.36	0.21	0.29	Pipeing charge
	1-1 シフォン手前指示線	0.06	0.08	0.06	0.07	TT Stg to meas T.
	1-2 クロリネーター注入管	0.09	0.12	0.03	0.08	Pre pipe for cha.
	1-3 クロリネーター移設	0.08	0.09	0.07	0.08	Actual cha. Time
	1-4 クロリネーター槽内管	0.05	0.07	0.05	0.06	Pre Cl to Cir. Lk
2.	クロネター循環時間	1.20	1.38	1.33	1.30	Cir. Lake Time
	2-1 槽場後到達時間迄	0.03	0.21	0.12	0.12	Before Cat
	2-2 槽場後到達時間迄	1.17	1.17	1.21	1.18	after Cat
3.	反応時間	5.54	6.46	8.33		← Chlorination
	3-1 反応槽後エヤー迄	0.24	0.22	0.26	0.24	Limit air Calculation
	3-2 段下ムの出口迄	4.29	5.51	6.56		Until fully clean
4.	エヤーフロー	2.34	3.04	2.53	2.49	← Air flow
	4-1 反応槽より導槽	0.21	0.33	0.33	0.29	Pipe to Transfer
	4-2 ポンプへ移送	0.05	0.07	0.13	0.08	Transfer to Blk Tank
	4-3 エヤー管槽	0.08	0.16	0.07	0.10	Pipe Air flow
	4-4 エヤーフロー	2.59	2.02	2.23	2.15	Blow
	Batch 合計時間	10.14	12.03	13.20	—	← IN clock flow
	Cycle time *	7.53	9.23	11.04	—	

* Cycle time は 4-2 の時を言う。

Design

	6+1	7½+1	9+1	85%
	Batch	C.T	Inst. Rate	7440 M/Y
1242	4657	7.9	577	4300 T/Y
1248	4700	9.4	500	3720
1254		11.1		

DSW 199880

STLCOPCB4057944

Temperature

	1242	1248	1252
	°C	°C	°C
Biphenyl Melting Tank	80. ±5	80 ±5	80 ±5
Biphenyl Mix Tank	100 ±5	100 ±5	100 ±5
Chlorinator (Middle)	162.	162	162
(Bottom)	162.	162	162
Chlorinator Circulation Line Air Cooler Inlet	(162)	(162)	(162)
Air Cooler Outlet	155 ±1	155 ±1	154.5 ±0.5
Chlorinator Hel gas Outlet	100 ±3	102. ±4	96.1 ±6
Hel gas Condenser Hel gas Outlet	49.3±4	52.1±3	50.7 ±4
Hel gas Condenser Circulation Water (out)	45±5	49 ±3	48.5 ±4
Aeration Tank	135±10	135 ±10	135. ±10
Still			
Still Pot (Bottom)	229 ±4	241 ±3	263.0 ±7 ±2
Heater Circulation Line Heater Inlet	228.5 ±4	239.7 +6 −3	263.2 ±7 ±6
Heater Outlet	233.5±4	244.2 ±3	267.7 ±3 ±5
Condenser Inlet	217.5±2	227.0 ±4	252.0
Outlet	85.5±0.5	85.3 ±0.5	86.5 ±1
Condenser Circulation Water	86.0±1	86.5 ±1	86.3 ±0.5
Surge Tank	69.5 ±0.5	82.0 ±0.5	109.5 ±0.5
Absorber Inlet	70.5 ±0.5	83.8 ±0.5	110.0 ±0.5
Outlet	(70. ±1)	(83.5 ±1)	(109.5 ±1)
Storage Tank	80 ±10	80 ±3	

DSW 199881

STLCOPCB4057945

	1242		1248		1257
Theoretical production of Anodes	1,670 kg/B		1,891 kg/B		
Biphenyl charge	2,555 kg/B		2,865 kg		
Batches 1 run	8 B		7 B		
Sum of Biphenyl	22,850 kg		19,997		
Theoretical production of Anodes	38,200 kg (4780 kg/B)		37,900		
Theoretical Cl_2	1,211 kg/B		1,029		
Sum of Cl_2	34,500 kg		36,950		
YIELD					
Finished Product	36,457 kg	95.4 %	37,600 kg	99.2	
	4,557 kg/B		4,700 kg/B		
Loss					
Still pump heat 221	385.3 kg	1.02 %	189.0	0.5	
still pres. 220 " "	59.7	0.15			
drawn from HCl line 252	229.5	0.61			
catch pot 212	1.0		–		
Pipe line	352.5	0.92	99.0	0.21	
HCl pipe line	0.9				
Chlorinator drain	48.7	0.11			
Sample	152.2	0.40			
Sub total	1,232.8	3.2	227.8	0.61	
Tar (Mortar)	7.7	1.9	1013.1	2.71	
		100.49			

DSW 199882

STLCOPCB4057946

Arrangement of Announciater, MMK Yokkaich

(As to the Instrument)

Shut Down	Pressure of Nitrogen (Low)	Liquid Level of Crude surge TANK (223). (High & Low)	Temp. of circulating Water of Still Condenser (Low)	Pressure of still pot (High) 215	Pressure of Fuel oil (Low)	Temparature of bottom of Chlorinator (High)	Pressure of Chlorine Feed (High & Low)
	Pressure of Instrument Air (Low)	Flow rate of Feed Arochlor to Adsorber (Low)	Liquid Level of still Reciever (High & Low)	Outlet Temp. of Still Heater (High)	Misfire of Burner	Temparature of Middle of chlorinator (High)	Flow Rate of Chlorine Feed (High & Low)
	Pressure of 13 Kg/cm² steam (Low)	Inline Analizer (High)		Liquid Level of still pot (High & Low)	Pressure of Fuel oil of Burner (Low)	Temp. of circulating Liquid of chlorinator (Low)	Totalizer of Feed Chlorine (80% of Total)
						Pressure of off gas of chlorinator (HCl) (High)	

(12)

DSW 199883

STLCOPCB4057947

Arrangement of Announciator, MMK, Yokkaich

(As to the electricity)

Interruption of generating Station	Stoppage of Source of electricity	Stoppage of electrolisys plant		
Stoppage of Circulating Pomp of chlorinator	Stoppage of Circulating Pomp of Still Pot	Stoppage of Circulating Pomp of Water of still condenser		

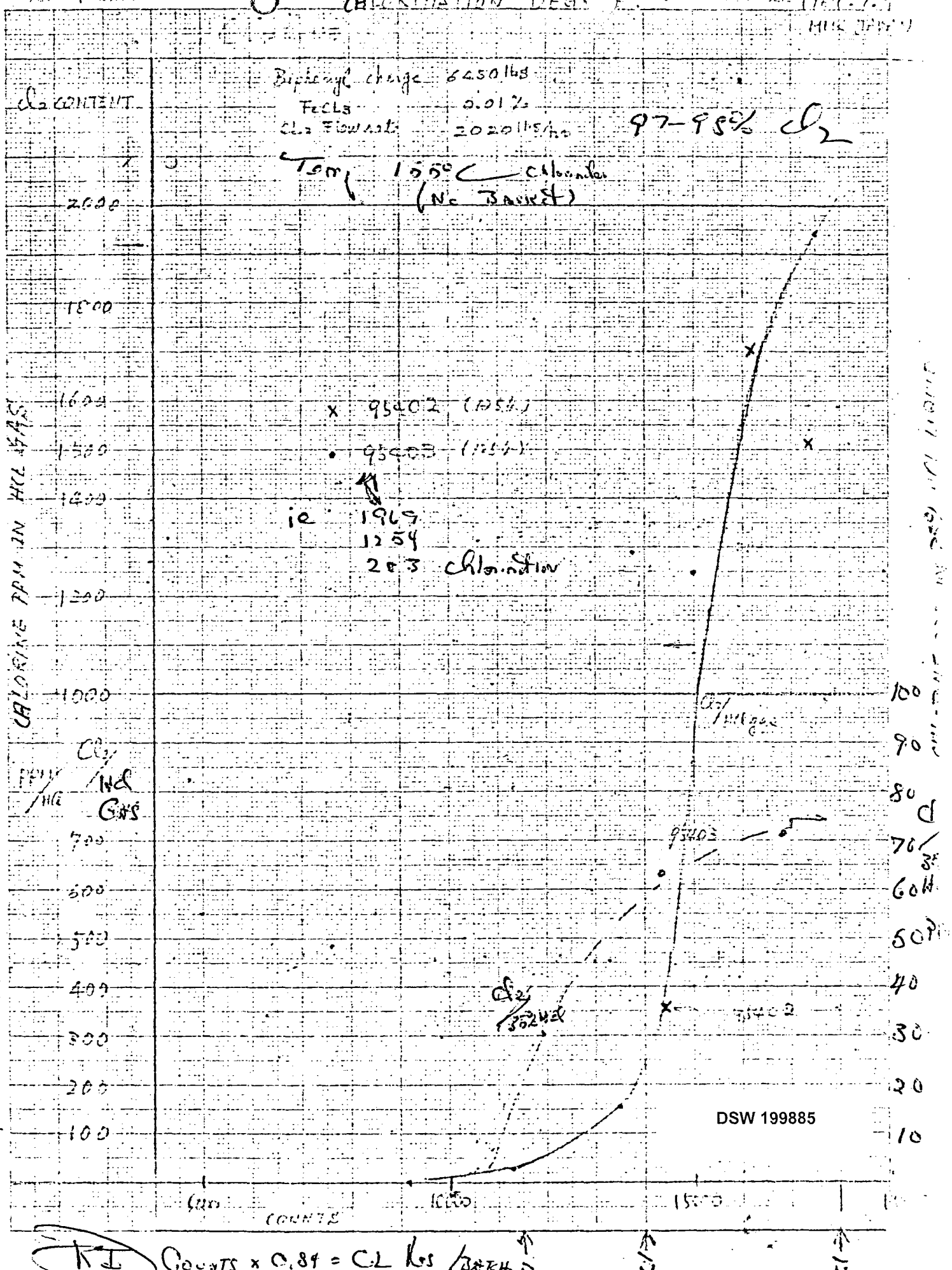
(13)

DSW 199884

STLCOPCB4057948

(14)

RELATION BETWEEN UNREACTED CHLORINE AND CHLORINATION DEGREE



KI Counts x 0.84 = CL lbs /BATCH

DSW 199885

STLCOPCB4057949

15 Answer to 3 questions to Mr. K. Matsuo (Supt.) 10 Jan 69 (Page 1)

PART I PURCHASED BIPHENYL

I Question

CONSIDER USE OF REFINED BY-PRODUCT BIPHENY FROM EITHER MITSUBISHI-YOKA PETROCHEMICAL COMPANY OR FOR TOYO-RAYON COMPANY TO PRODUCE THERMINOL GRADE OPH AT MMK YOKKACHI PLANT.

BASIS:

1. PRICE COMPETITION FOR HEAT EXCHANGE FLUIDS VERY STRONG AND WILL EFFECT PROFIT MARGINS.

2. A BIPRODUCT REFINED BIPHENYL WILL BE AVAILABLE FROM TOYO-RAYON COMPANY IN SEPTEMBER. THE QUALITY IS REPORTED TO BE GOOD ENOUGH FOR KONAN CHEMICAL COMPANY TO USE IN PRODUCTION OF PARA PHENYL PHENOL (PPP) KONAN NOW USING ANNISTON BIPHENYL. THE PRICE IS ESTIMATED TO BE 50-70¥ PER KILO OR AN ESTIMATED 60-70% OF YAWATA BIPHENYL COST.

3. THE BIPRODUCT SOURCE IS REPORTED TO BE STILL BOTTOMS FROM CYCLOHEXANE PARAXYLENE PROCESS (NYLON) THE ANNUAL PRODUCTION IS ESTIMATED TO BE 9 M POUNDS PER YEAR

15

DSW 199886

PROBLEMS TO RESOLVE

1. OBTAIN ACTUAL SPECIFICATIONS OF BY-PRODUCT BIPHENYL FROM KOYO-RAYON.

2. OBTAIN SAMPLE OF REPRESENTATIVE MATERIAL.

3. IDENTIFY IMPURITIES.

4. DETERMINE IF ANY HEALTH HAZARDS WILL RESULT FROM THE PRESENCE OF CHLORINATED IMPURITIES TO OPERATIONS OR TO CUSTOMER.

5. DETERMINE BY LABORATORY PREPERATION AND TESTS IF THERMAL STABILITY PROPERTIES ARE CHANGED. LABORATORY TO USE LABPREPARED CPH FROM YAWATA BIPHENYL AS CONTROL

6. DETERMINE THERMINOL STABILITY SPECIFICATIONS FOR FR SERIES. NORMAL PRODUCTION IN US OF FR GRADE IS SAME AS FOR ELECTRICAL GRADE.

7. DETERMINE HOW TO COMPLETLY SEPERATE PRODUCTION OF ELECTRICAL GRADE CPH BY YAMATA BIPHENYL FROM THERMINOL GRADE CPH BY BY-PRODUCT BIPHENYL

8. DETERMINE IF: LENGTH OF ELECTRICAL AND THERMINOL RUNS; CLEAN-OUT OF BIPHENYL STORAGE TANK; CHANGE OF FILTER CARTRIDGES AND SILICA GEL; DRAINING OF LINES AND PUMPS; USE OF CLEAN-OUT BATCHES AND OTHER CHANGE-OVER COSTS WILL REDUCE SAVINGS OF LOWER COST BIPHENYL ENOUGH TO STOP ITS USE.

DSW 199888

1/5

STLCOPCB4057952

PART II PURCHASED BIRHENYL

II CAN PURCHASED BIRHENYL BE ADDED TO FURNACE STREAM TO ALLOW HIGHER CONVERSION TO TERPHENYL AT LOWER COST THAT CONVERSION FROM BENZENE

Question

ANSWER

NO - NOT AT PRICE OF BIRHENYL AND BENZENE

PRICE ESTIMATE

BENZENE 25¢ / KILO

PURCHASED BY PRODUCT BIRHENYL 50¢ / KILO

BIRHENYL PRICE NEED TO BE CLOSE TO BENZENE PRICE

BACKGROUND INFORMATION

1. ADDITION OF BIRHENYL TO BENZENE STREAM TO FURNACE CAN BE DONE

A: REQUIRE PREMIX OPERATION

ONE WAY - MELT TANK TO RECEIVE AND MELT BAG OR TANK TRUCK BIRHENYL. RATIO CONTROL MOLTEN BIRHENYL AND BENZENE IN IN-LINE MIXING & PROVIDE SAFE GUARDS ON MIXING CONTROL TO AVOID PLUGGING FURNACE

THIS OPERATION WILL REQUIRE CAPITAL AND HIGH LABOR IF BAGS ARE USED.

DSW 199889

STLCOPCB4057953

2. PRODUCTION GAINS WOULD BE:

A: REDUCTION OF FURNACE FUEL REQUIRMENT SINCE HEAT TO PRODUCE RECYCLE BIPHENYL WILL NOT BE REQUIRED.

B: INCREASE CAPACITY OF FURNACE BY ELIMINATION (OR REDUCTION) OF RECYCLE PRODUCT BACK TO FURNACE. GAIN WILL BE ~~IN PROPORTION~~ IN PROPORTION TO BIPHENYL IN-PIT.

C. SOME YIELD IMPROVEMENT FROM ELIMINATION OF TARS AND CARBON MONOXIDE FROM PRODUCTION OF RECYLE BIPHENYL

DSW 199890

O

QUESTION No. 2 Question

IS THERE ANY ADVANTAGE (IMPROVED REACTION RATE) TO USE MOLECULAR SIEVE TREATMENT OF CHLORINE PLANT HYDROGEN WHEN USED IN HB-40 HYDROGENATION?

1) ANNISTON REVIEW OF PROJECT FOR MR. MURAKAMI INDICATED THERE MAY BE SOME ADVANTAGE.

2) CHLORINE PLANT HYDROGEN ANALYSIS YOKKAICHI

H_2 - 99.97%
CO 17-22 PPM
CO_2 20-30 PPM
N_2 0.02
O_2 0.01
Hg 3×10^{-6} Gram/m^3

ESTIMATE:

(A) BASED ON H_2 ANALYSIS IT IS DOUBTFUL THAT ANY MAJOR GAIN IN HB-40 PRODUCTION RATE WOULD OCCUR BY INSTALLING AT THIS TIME A CLEAN-UP SYSTEM

(B) YOKKAICHI CO is 17-22 PPM
ANNISTON - AFTER CLEAN UP is Less than 10 PPM

(C) SYSTEM WAS DESIGNED TO OPERATE AT 50 PPM. NOT MUCH DIFFERENCE, IN LABORATORY AUTOCLAVE, BETWEEN 30-40 PPM CO + 10 PPM CO. IN

DSW 199891

STLCOPCB4057955

5. SOME RATE IMPROVEMENT AT ANNISTON IS DUE TO HIGHER AVERAGE PRESSURE OF HYDROGEN FROM BIPHENYL HYDROGEN COMPRESSOR THAT WAS OBTAINED ON THE CHLORINE PLANT COMPRESSOR.

6. SINCE ONLY 70% OF THE PROCESSED HYDROGEN CAN BE USED IN HB-40 THE YOKKAICHI TUBULAR UNIT ~~WILL NOT~~ MAY NOT BE A GOOD SUPPLY SOURCE UNTIL SALES OF BIPHENYL AND TERPHENYL PRODUCTS ARE WELL ESTABLISHED.

ESTIMATE

1. IT IS DOUBTFUL THAT A MAJOR GAIN IN HB-40 OUTPUT ~~COSTS~~ WOULD OCCUR FROM INSTALLING A CLEAN-UP SYSTEM FOR YOKKAICHI CHLORINE PLANT HYDROGEN.

ACTION

1. WRITE G. MILLER - ANNISTON FOR INFORMATION ON AMOUNT OF RATE IMPROVEMENT THAT COULD BE ACHIEVED FROM TREATING MM HYDROGEN

DSW 199892

15-8

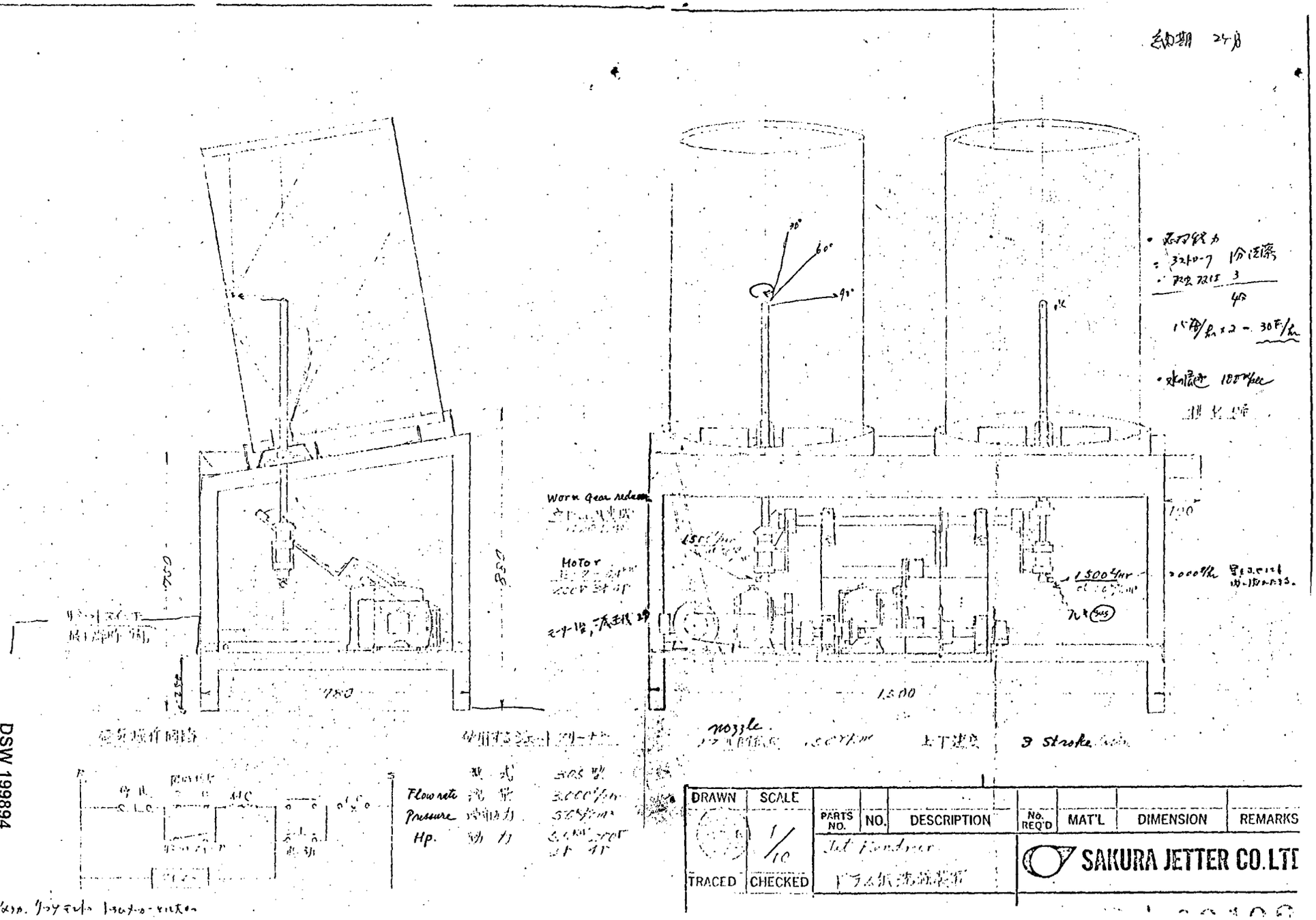
STLCOPCB4057956

2. TUBOLER UNIT #7 PLANS TO LEAVE SPACE FOR LATTER INSTALLATION OF HIGH PRESSURE STAGE COMPRESSOR AND HYDROGEN CLEAN UP SYSTEM.

DSW 199893

15-9

STLCOPCB4057957



DRAWN	SCALE	PARTS NO.	NO.	DESCRIPTION	No. REQ'D	MAT'L	DIMENSION	REMARKS
	1/10			Jet Sweeper				
TRACED	CHECKED							

SAKURA JETTER CO.LTD

DSW 199894

STLCOPCB4057958

This man has also been arrested, the spokesman said, and has made confessions.

Details of the confessions will be released after investigations are complete, the spokesman said.

U.S. Pullout Begins

Continued From Page 1

after the announcement by President Richard M. Nixon and President Thieu on Midway Island that 25,000 Americans would be sent home by the end of August.

Nixon said last month that he hoped to beat a timetable proposing the withdrawal of all American combat forces by the end of 1970.

The farewell ceremony was attended by the American military commander, Gen. Creighton W. Abrams, U.S. Ambassador Ellsworth Bunker and South Vietnam's Vice President Nguyen Cao Ky.

Gen. Abrams told the men of the "Old Reliables"—the nickname for the 9th Infantry Division—that in their nearly three years of service in the Mekong Delta, men of the division had conducted themselves magnificently in action against a tough, wily and determined enemy.

"You laid the groundwork for the Republic of Vietnam's armed forces to carry on your important role in the defense of their nation," he said.

The first unit to leave Vietnam is the 3rd Battalion, 60th Infantry Regiment of the 9th Division.

Their departure symbolized the reversal of a process that began almost exactly 19 years ago. Since Jan. 1, 1961 36,866 Americans have died in Vietnam.

On July 15, 1950, the first U.S. military mission arrived in Saigon to liaison with French forces fighting the Viet Minh.

From this handful of observers, the figure steadily increased over the years and reached a peak of 543,000 last August. It has remained at a plateau of roughly 540,000 for more than a year.

This week, there were 539,500 men in Vietnam, not counting 35,700 U.S. sailors and Coast Guard men in ships in the South China Sea.

Lull Being Watched

WASHINGTON (AP)—The White House said Monday the lull in fighting in Vietnam is being watched carefully and the matter of U.S. troop withdrawals is under constant review.

But press secretary Ronald L. Ziegler gave no definite confirmation to reports that if the lull continues the withdrawal of U.S. forces might be speeded up.

Ziegler said he had no information to provide either, on a report that some of the

Patient Dies

FUKUOKA (Kyodo)—A 13-year-old Yamaguchi Prefecture boy suffering from a skin disease—one of 864 recognized patients in the rice bran oil poisoning case—died Tuesday.

Motohiro Miyoshi of Mine, was the first fatality among the victims in the sensational poisoning case blamed on a chlorine-containing compound that accidentally got into the product.

Miyoshi collapsed at about 11:30 a.m., soon after he went out of his house too seek fresh air. He had complained of headache and vomiting after he got home from school.

He was already dead when a doctor was called.

An autopsy was held on the body in the evening at Yamaguchi University Hospital.

The doctors who performed the autopsy said the boy died of a heart attack and his death may not be directly related to the poisoning.

Miyoshi had pimple-like eruptions all over the face and dark pigmented skin all over his body.

He was not considered a serious case and was examined every 10 days at the Yamaguchi University Hospital in Ube.

He was said to have consumed an average 1.4 liters of the rice bran oil produced and sold by Kanemi Soko Co., of Fukuoka between March 1967 and October 1968. He was said to be excessively fond of fatty dishes.

Miyoshi and three other members of his family are among 864 recognized patients in the poisoning from 12 prefectures.

About 13,000 persons who ingested the oil have been examined.

Tokyo Tech. Prof. Kills Self in Woods

A professor of Tokyo Institute of Technology hanged himself Tuesday, supposedly out of excessive fatigue from the diehard campus strife, Zushi police said.

Kazuo Niira, 49, a professor of physics at the state-run university, was found dead in a woods in the precincts of Jinmuji Temple in Zushi, Kanagawa Prefecture, at about 3:15 p.m. by a passerby.

He had left no suicide note, but a notebook found in his bag near the body contained scribblings with a description of his strife-weary mind.

ness circles are calling for some restrictive measures to curb U.S. imports from Japan and a number of protectionist bills are being introduced on the congressional floor, he explained.

The U.S. Government, however, thinks that efforts should be made to expand trade, not to restrict it, Meyer said, and added that he hoped the U.S. and Japan could maintain mutually beneficial trade relationships.

What the U.S. wants in economic relations with Japan is to bring the trade balance, currently some $1,100 million in favor of Japan, in a more proper line, he said.

A reporter mentioned the report that only three Cabinet members of the Nixon Administration will take part in the 7th meeting of the Joint Japan-U.S. Committee on Trade and Economic Affairs and asked whether this indicates decreased interests of the U.S. in the ministerial committee.

The U.S. envoy said he has not received the final information on the composition of the U.S. delegation to the meeting but that he is sure that there will be no decrease of American interests in Eastern Asia.

He regretted, however, that certain Cabinet members would have to join the presidential delegation which plans to visit Romania and six Asian countries at the same time.

But he said U.S. Secretary of State William P. Rogers and Foreign Minister Kiichi Aichi would also hold discussions on Okinawa.

He added that in the meeting held between Nixon and Aichi in Washington in early June the President said Eastern Asia will be the most important area for the U.S. in the remainder of the 20th century.

Prior to the question and answer session, the U.S. ambassador read a brief, prepared statement. He said in the course of the statement that he hopes that the sufferers of the heavy rains in western Japan will be able to return to normal life at an early date.

Sea Limits

PORT OF SPAIN (AP) — Legislation aimed at extending Trinidad and Tobago's territorial sea limits from 3 miles to 12 miles will be introduced this week, said acting Prime Minister Arthur Robinson.

Beautiful Sapphire Mink Stoles
From 45,000 to 150,000 yen!!
Nicola's International Furs
Tokyo 585-2941—ask for Fur Department

DSW 199895

STLCOPCB4057959

MITSUBISHI MONSANTO CHEMICAL COMPANY

June 69

****

Yokkaichi Plant

Administrative Department Director K. Hirai Assist. to Director T. Watanabe	General Affairs Sec.	Manager	K. Hirai (conc.)
		Assist. Mgr.	S. Jyo
	Personnel Sec.	Manager	K. Hirai (conc.)
		Assist. Mgr.	I. Kubo
		"	M. Danno
	Accounting Sec.	Manager	K. Kusahara
		Assist. Mgr.	O. Uemura
	Purchasing Sec.	Manager	S. Ishikawa
		Assist. Mgr.	F. Inaba
	Traffic Sec.	Manager	K. Yano
		Assist. Mgr.	K. Iseki
PVC Production Department Gen. Superintendent K. Matsuo Assist. to Gen. Sup't. M. Tsukajima SKE-Jimo T. Si Yoshika Yo-540-Ki	Coordination Sec.	Superintendent	K. Migono
		Supervisor	S. Takasu
	Plant Technical Service Sec. - VCM	Superintendent	M. Hatano
		Supervisor	S. Takasu (conc.)
		"	K. Ohura
	Plant Technical Service Sec. - PVC	Superintendent	K. Matsuo (conc.)
		Supervisor	K. Miyati
	Electrolysis Sec.	Superintendent	[illegible]
		Supervisor	[illegible]
	VCM Sec.	Superintendent	[illegible]
	PVC Sec.	Superintendent	[illegible]
		Supervisor	[illegible]
	Petroleum Additives Sec.	Superintendent	Y. [illegible]imoto
PER Production Department Gen. Superintendent Y. Terajima Assist. Gen. Supt.	Coordination Sec.	Superintendent	Y. Hinago (conc.)
		Supervisor	K. Hirohata
	Plant Technical Service Sec.	Superintendent	H. [illegible] (conc.)
	Styrene Polymer	Superintendent	Y. Hinago (conc.)
		Supervisor	[illegible]
	[illegible]		

YOKKAICHI PLANT

Manager

. Murakami

DSW 199896

STLCOPCB4057960

[illegible]

Assist. Gen. Supt.
Y. [illegible]
H. [illegible]

Engineering & Maintenance Dept.

Gen. Superintendent
S. Ishii

Assist. Gen. Supt.
Y. Kitagawa
[illegible] Sakurai

Plants Control Staff

Director
[illegible] Murakami (conc.)

YOKEAICHI RESEARCH & DEVELOPMENT DEPARTMENT

Director T. Morikawa

Assist. Director
H. Kada ✓ Mink Coord. Springfield Dr - U 1211-54

Assist. to Director
H. Yamamoto

[illegible]	[illegible]	[illegible] (conc.)
	Supervisor	S. [illegible]
[illegible]	Superintendent	[illegible]
	Supervisor	T. Hashiguchi
[illegible] Matching Sec.	Superintendent	K. Nojima
Coordination Section	Superintendent	Y. Nishida
	Supervisor	T. Fukuda
Engineering Sec.	Superintendent	M. Okumura
	Supervisor	S. Kawamura
Electrical Sec.	Superintendent	M. Sakurai (conc.)
Maintenance Sec.	Superintendent	K. Kuramoto
	Supervisor	K. Dairaku
	"	S. Kawamura (conc.)
Instrumentation Sec.	Superintendent	K. Shiraishi
	Supervisor	A. Yamamoto
Utilities Sec.	Superintendent	K. Kuramoto (conc.)
	Supervisor	I. Jimbo
Plants Control Sec. Coordination	Manager	H. Takeuchi
	Assist. Mgr.	S. Itani
Operation Evaluation Sec. Cost	Manager	T. Ichiriki
	Assist. Mgr.	K. Takano
Safety Sec.	Manager	K. Goto
Materials Std. Sec.	Manager	S. Kobayashi
Research Section A	Manager	H. Kada (conc.)
	Assist. Mgr.	M. Tsuji
Research Section B	Manager	H. Kojima
	Assist. Mgr.	H. Kakitani
Research Section C	Manager	H. Mitsuoka
	Assist. Mgr.	T. Tsushima
Research Section D	Manager	S. Sakurai — N. Wport
	Assist. Mgr.	H. Uragami
	"	K. Horie

DSW 199897

STLCOPCB4057961