xv ,

.

LIST OF TABLES

Table	Description	Page
1	Sea water constituents (19 6_0 chlorinity) and major constituents relationship with the (34.4 6_0 salinity) present in sea water	7
2	Stages in evaporation of sea water	1 6
3	Solubility data of the system:NaCl-H ₂ O	21
4	Solubility data of the system:Na2SO4-H2O	22
5	Solubility data of the system: NaCl-Na ₂ SO ₄ - H ₂ O at 30°C.	23
6	Solubility data of the system KCl-NaCl-H ₂ O	24
7	Solubility data of the system KCl-MgCl ₂ -H ₂ O at 25°C	2 5
8	Solubility data of the system $KC1-NaC1-MgC1_2$ - H_2O at 25°C	2 6
9	Solubility data of the system NaCl+NH ₄ HCO ₃ = $NaHCO_3 + NH_4Cl$ at 15°C	
10	Solubility data of the system NaCl - K^{C1} - $M_{gC1_2}^{-M_{gSO}}_{4}$ - $Na_2^{SO}_{4}$ - H_2^{O} at 25°C	28
11	Solubility data of the system $Na^{C}1-K^{C}1 - Mg^{C}1_2 - Mg^{S}0_4 - Na_2^{S}0_4 - H_2^{O}$ at 83°C	2 9
12	Composition of Indian sea and well brines	31
13	Direct solar evaporation of bitterns (reduction in volume and rise in concentration of salts)	57
14	Salt fractions collected in direct evaporation of bitterns	58
15	Direct solar evaporation of bitterns (composition of bitterns at various stages)	ı 59
16	Direct solar evaporation of bitterns (composition of salt fractions)	60
17	Solar evaporation of sea bitterns with addition of solid magnesium chloride (MgCl ₂ .6H ₂ O)	64
18	Solar evaporation of mixed bitterns(ratio of mixing 1:1)	65
19	Salt fractions obtained in evaporation of mixed bitterns (ratio of mixing 1:1)	6 6

	xvi Deservición	ne
<u>able</u>	Description	Page
20	Solar evaporation of mixed bitterns (ratio of mixing 2:1)	67
21	Salt fractions obtained in evaporation of mixed bitterns (2:1 ratio)	68
22	Solar evaporation of bitterns treated with mixed salt	70
2 3	Solar evaporation of saturated solution of mixed salt in water	71
24	Salt fractions collected by evaporation of mixed salt solution	72
25 -A	Separation of mixed salt in three fractions to obtain mixed salt with less concentration of sodium chloride	73
В	Bitterns compositions at various stages during precipitation of mixed salt	73
26 - A B	Requirement of solivap green dye (I.C.I data) Requirement of Atul Vaporaid green p x c (Atul ^B ros. Manf. data)	75
2 7	Evaporation of water with and without solivap green	76
28	Effect of concentration of dye on the yield of salt by solar evaporation of similar depths of brines (15 cms.)	78
29	Effect of addition of dye on water and brine evaporation	, 78
30	Decomposition of the dye due to exposure to solar radiation	78
31	Effect of solivap green on evaporation of brine	໌ 79
32	Solar evaporation of 26° Be brine to 38°Be bitterns stage with and without dye addition	80
33	Evaporation of bitterns without dye or magnesium chloride addition	81
34	Evaporation of bitterns with addition of dye but without magnesium chloride addition	82
35	Evaporation of mixed bitterns (1:1 ratio) with addition of dye vaporaid green p x c	83

	٠	
~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	٦.	1
A V		*

Table	xvii Descrip <b>tion</b>	Page
36	Evaporation of mixed bittern (2:1 ratio) with addition of dye (Atul vaporaid green p,x c	84
37	Effect of solivap green and magnesium chloride addition on bitterns evaporation	85
38	Test experimental results (Kandar bitterns)	88
39	Analysis of mixed salt storyed in pit	91
40	Analysis of stored mixed salt under tarpaulin cover	92
41	Area required for 1000 tonnes of mixed salt production	96
42	Evaporation of sea bitterns (approximate values of total chloride, total magnesium and potassium and sodium chlorides	106
.43	Bitterns composition at various densities	106
44	Capital expenditure for storage of mixed salt	117
45	Invariant compositions corresponding to the boundary of carnallite at 25°C and 83°C	123
46	Solubility data for the system:K ^C l-MgCl ₂ - H ₂ O (space model for carnallite)	126
47	Differences in solubilities of NaCl - KCl at 30° and 111.9°C (B.P.)	131
48	Extraction of mixed salt (laboratory results)	140
49 A	Treatment of Kharaghoda mixed salt	145
49 B	Treatment of mixed salt converted to schoenite	146
50	Amount of carnallite crystallisation at various temperature ranges	147
51	Decomposition of carnallite with water (2:1 ratio)	149
52	Decomposition of carnallite with water(1:1 ratio)	150
5 <b>3</b>	Recrystallisation of impure potassium chloride (Ist cycle)	15 <b>1</b>
54	Recrystallisation of impure potassium chloride (IInd cycle)	152
55	Cooling of hot NaCl-KCl solution and collection of fractions between various temperature ranges	153

xvii**i** 

,

,

-	Table	Description	Page
	56	Extraction of mixed salt (pilot plant experi- ment data A & B)	156,157
	57	Crystallisation of carnallite in S.W.crystal- liser	15 <b>7</b>
	58	Decomposition of carnallite (pilot plant experiment data)	159
	59	Evaporation of carnallite decomposed liquor to obtain carnallite and 36° Be bittern	1 <b>61</b>
	60	Recrystallisation of impure potassium chloride	163
	61	Regeneration of NaCl - K ^C l solution	164
	62	Treatment of'sel's mixts' to study extraction of different salts	164
	63	Dissolution of'sel's mixts' (laboratory experiment 1)	16 <b>7</b>
	6 <b>4</b>	Dissolution of'sel's mixts' (laboratory experiments 2 & 3)	168
	65	Chilling of'sel's mixts' solution at 10°C	1 <b>7</b> 0
	66	Chilling of'sel's mixts' solution at -5°C	172
	67	Density and analysis of'sel's mixts'solution	175
	68	Dissolution of 'sel's mixts' (pilot plant experiment data)	177
	69	Recovery of epsom salt and Glauber's salt (Pilot plant experiment data)	178
	70	Settling rate of sodium and magnesium sulphate	180
	71	Viscosities of different solutions available in the process	181
	72	Comparative rate of flow with 876 sparkler filter unit	181
	73	^P recipitation of anhydrous sodium sulphate from Glauber's salt (experiment 1)	18 <b>3</b>
,	74	Precipitation of anhydrous sodium sulphate from Glauber's salt (experiment 2)	18 <b>4</b>
	75	System: $K_2SO_4-Al_2(SO_4)_3 - NaCl - H_2O_{at 30°C}$	214
•	76	System: $K_2SO_4$ . Al ₂ (SO ₄ ) ₃ -NaCl-MgCl ₂ - H ₂ O at 30°C	215

,

1 1

## xix

· [

## Table Description System: $K_2SO_4.Al_2(SO_4)_3 - NaCl-MgCl_2-MgSO_4-H_2O$ at 30°C 77 216 78 Preparation of potash alum from mixed salt and aluminium sulphate (laboratory experimental data sheet -1) 220 79 Preparation of potash alum from mixed salt and aluminium sulphate (laboratory experimental data sheet -2) 224 80 Preparation of potash alum from mixed salt and aluminium sulphate (laboratory experimental data sheet -3) 225 81 Potash alum from mixed salt containing high amounts of sodium chloride (laboratory experiments) 226 82 Preparation of potash alum from mixed salt solution (laboratory experiments) 227 83 Preparation of potash alum from bitterns (laboratory experiments - 1)229 84 Preparation of potash alum from bitterns (laboratory experiments - 2) 230 85 Settling rate of aluminium sulphate solution 234 86 Material balance data for potash alum from 235 mixed salt (pilot plant experiment) Analysis of potash alum samples from pilot plant 87 240 experiments 241 88 Crystallisation of potash alum 242 89 Standard specifications of potash alum 260 Material of construction (standard data) 90 91 Testing of corrosion in reaction mixture containing mixed salt and 36° Be bittern 261 at 110°C Protection of metal surface by application of paints (Testing of paints at $110^{\circ}$ C in presence 92 of reaction mixture containing mixed salt and 265 36° Be bittern) Testing of corrosion of common metals for 93 alum process with and without addition of 268 nitric acid

_

1

<u>Table</u>	Description	Page
94	Less soluble double salts of potassium	2 <b>73</b>
95	Solubility of sodium and potassium salts of nitro phenols and related acids	277
96	Solubility of hexanitrodiphenylamine	286
9 <b>7</b>	Precipitation of potassium from sea water using powdered sodium cobaltinitrite reagent	2 <b>92</b>
98	Comparative study of two precipitation processes namely Dipicrylamine and sodium cobaltinitrite for potassium recovery from sea water	294
99	^P recipitation of potassium from sea water, brine and bittern samples as potassium dipicryl- aminate	296
100	Precipitation of potassium as potassium dipicryl- aminate from sea water (laboratory data)	2 <b>97</b>
101	^P recipitation of potassium dipicrylaminate from 29° Be bittern	29 <b>7</b>
102	Washing of potassium dipicrylamine precipitation with water on buchner funnel to remove adhering impurities	298
103	Solubility of potassium dipicrylaminate in water	299
104	Solubility of potassium dipicrylaminate at 28°C in different solutions	300
105	Solubility of free amine (dipicrylamine) in nitric acid at 28°C	301
106	Decomposition of potassium hexanitrodiphenylamine precipitates with nitric acid of different con- centrations at 30°C	30 <b>3</b>
10 <b>7</b>	Decomposition of potassium dipicrylaminate with concentrated nitric acid (at room temperature)	30 <b>4</b>
108	Studies on requirement of acetone and strength of acid for the decomposition of potassium dipicrylaminate precipitates	30 <b>6</b>
109	Decomposition of potassium dipicrylaminate with acetone and acid to obtain a concentrated solution of potassium nitrate in the filtrate	306
1 <b>1</b> 0	Recovery of reagent (dipicrylamine) from active carbon by acetone	308

	<b>xxx</b> 17 i	
Table	Description	Page
111	Recovery of dipicrylamine from end liquor using n-butanol	309
1 <b>12</b>	Recovery of potassium as potassium dipicryl- aminate from sea water (bench scale experiment)	311
1 <b>13</b>	Comparative study of raw material requirements	312
114	Decomposition of potassium dipicrylaminate precipitates	316

LIST OF FIGURES

		HIDT OF FIGORID	
	FIGURE	Description	PAGE
	1	System: NaCl - H ₂ O	21
	. 2	System: $Na_2SO_4 - H_2O$	21
	3	System: Na ₂ SO ₄ - NaCl - H ₂ O	23
	4	System: NaCl - KCl - H ₂ O (space model)	24
	5	System: NaCl - KCl - H ₂ O, 20°C isotherm and boiling point isobar	24
	6	System: KC1- MgCl ₂ - H ₂ O, 25°C isotherm	.25
	7	System: KCl - NaCl - $MgCl_2 - H_2O$ at 25°C	26
	8	System (Reciprocal salt pair): $NaCl + NH_4HCO_3 = NaHCO_3 + NH_4Cl at 15°C$	27
	9	Quinary system: NaCl - $KCl - MgCl_2 - MgSO_4 - Na_2SO_4 - H_2O$ at 25°C	28
	10	Quinary system: $NaCl-KCl-MgCl_2 - MgSO_4 - Na_2SO_4 - H_2O$ at 83°C	29
	11	Quinary system: $NaCl-KCl-MgCl_2 - MgSO_4 - Na_2SO_4 - H_2O$ at 25°C, showing path of mixed salt separation during solar evaporation of bitterns	49
	12	Per cent transmittance of solivap green in water at different wave lengths, (showing the absorption region of solar radiation and the requirement of dye to get complete absorption)	76
,	12	Percentage transmittance of solivap green, B.A.S.F green PLX, Atul Vaporaid green pxc at various wave lengths	76

· ·

## x**xxii**

.

.

、

____

FIGURE	Description	PAGE
14	· · · · · · · · · · · · · · · · · · ·	TROD
14	Field scale experiments at Kamdar Salt Works (Bhavnagar Salt Works, Bhavnagar), showing crude and mixed salt pans and arrangement of channels	8 <b>7</b>
15	Evaporation and dilution of 30°Be and 36° Be bitterns during monsoon	94
16	Density variations of the surface layers of the stored 30° and 36° Be bitterns during monsoon	94
17	Lay out details of mixed salt area at experimental salt farm	95
18	General layout of mixed salt pans	96
19	Photographs showing layout, mixed salt pan bed, collection and storage of mixed salt	96
20	Concentration of various salts in bittern during solar evaporation of bitterns	106
21	Conditions for mixed salt separation (7.76 to 10.9 per cent magnesium and 21 to 28.6 per cent chloride concentrations	106
22	Concentrations of magnesium chloride verses other salts (NaCl, KCl, $MgSO_4$ ) during solar	
	evaporation of bitterns	106
23	System: KCl - MgCl ₂ - H ₂ O (space model for carnallite)	125
24	System: NaCl - KCl - H ₂ O at 30°C isotherm and boiling point isobar	130
25	Flow sheet for regeneration of NaCl -KCl saturated solution	164
26 -	Flow diagram and the compositions of solids and solutions in the process for the recovery of potassium chloride and byproducts from mixed salt	195
27	Photograph showing potassium chloride pilot plant	195
28	Photographs showing arrangements of equipments in commercial potassium chloride unit at Kandla	

~

	-	
xxiii		

FIGURE	Description	PAGE
29	System: $K_2SO_4$ -Al ₂ (SO ₄ ) ₃ -H ₂ O (polytherm)	216
30	Solubility of aluminium sulphate potassium sulphate and potash alum at various temperatures	216
31	Solubility of potash alum at 0° and 25°C	216
32	Process flow sheet for the recovery of potash alum from mixed salt	235
33	Process flow sheet for the recovery of potassium salt from sea (brine and bitterns) by precipitation with calcium dipicrylaminate reagent	2 <b>92</b>
34	Flow sheet showing arrangement of vessels in bench scale experiments	311

•

, , ,