

CRITICAL EVALUATION (continued)

Table 1 LiClO₄-H₂O

Expression of Y	
LiClO ₄	$Y = \ln(4x^2/(1+x)^2)$
LiClO ₄ ·H ₂ O	$Y = \ln(27x^2(1-x)/(1+x)^3)$
LiClO ₄ ·3H ₂ O	$Y = \ln(5^5x^2(1-x)^3/(27(1+x)^5))$

Four coefficients, deduced from experimental data by linear regression, were necessary to represent the data. Their values are given in table 2.

Table 2 System LiClO₄-H₂O

Coefficients of fitting equations

Solid Phase	A	B	C	D
LiClO ₄	-226579.46	-1022.4424	1.1542909	6229.9317
LiClO ₄ ·H ₂ O	87977.65	446.3249	0.5600779	-2670.172
LiClO ₄ ·3H ₂ O	-1943.37	0.503708	-8.49846E-3	5.4328

Solubility of anhydrous LiClO₄

Only four data are available so that the coefficients A, B, C, D have been calculated by simple resolution of a system of 4 linear equations and the data above 172 °C must be considered as tentative data. The value 509 K (236 °C) given by Richards and Willard (2) for the melting point of lithium perchlorate has been adopted. The comparison between experimental and calculated values of solubility is presented in table 3.

Table 3 System LiClO₄-H₂OLiquidus of anhydrous LiClO₄

<i>t</i> °C	exp 100w _l calc		exp 100x _l calc		ref
144.2	90.0	90.04	60.4	60.5	7
167.5	91.04	91.06	63.24	63.3	7
172.0	91.11	91.13	63.44	63.5	7
236	100	100	100	100	2

The solid phase in equilibrium with liquid at 144.2 °C is probably anhydrous lithium perchlorate instead of the monohydrate claimed by the authors

(continued)

CRITICAL EVALUATION (continued)

Solubility Curve of $\text{LiClO}_4 \cdot \text{H}_2\text{O}$

The solubility Curve of $\text{LiClO}_4 \cdot \text{H}_2\text{O}$ has been determined only by Simmons and Ropp (7).

The coherence of experimental results is good except for the data close to the melting point.

The congruent melting point is given by solution of the equation :

$$D = -A/T_m - B \ln(T_m) - CT_m \quad [2]$$

The calculated value is 152.2 °C, the experimental value is 149 °C (5).

Table 4 Liquidus of monohydrate $\text{LiClO}_4 \cdot \text{H}_2\text{O}$

t °C	exp100w1calc		exp100x1calc		deviat.x1000	ev.	ref
93.2	70.5	70.53	28.8	28.84	0	R	7
97.3	71.0	70.95	29.3	29.26	0	R	7
108.9	72.8	72.74	31.2	31.12	3.2	R	7
120.7	75.0	75.23	33.7	33.96	- 8.8	R	7
136.9	80.0	79.38	40.4	39.46	22.7	T	7
144.0	82.5	81.47	44.4	42.68	39.8	T	7
144.2	81.53		42.78				**
148.5	85.0	83.02	49.0	45.29	81.7	A	7
149.3	87.5	83.34		45.87			**
149.3		87.48	54.2	54.2	0	R	7
148.5		87.74		54.8			**
144.2	90.0	88.85	60.4	57.45	50.5	A	7*
136.9		89.87		60.03			**
120.7		92.37		67.2			**
108.9		93.38		70.48			**
97.3		94.01		72.66			**
93.2		94.15		73.15			**

* belongs probably to the liquidus of anhydrous LiClO_4 ** calculated by evaluator

R = recommended, T = tentative, A = aberrant value

A comparison between experimental and calculated values of solubility is presented in table

4. The deviation is the quantity $100(x_{\text{exp}} - x_{\text{calc}})/x_{\text{calc}}$. The data have been recommended

when $\text{dev} < 2\%$, considered as tentative when $2\% < \text{dev} < 5\%$ and aberrant above 5 %

Solubility Curve of $\text{LiClO}_4 \cdot 3\text{H}_2\text{O}$

The range of solubility of trihydrate has been determined in totality by Simmons and Ropp

above 0 °C, but in most papers the data are reported at 25 °C. Lithium perchlorate trihydrate

crystallizes from aqueous solutions as short prismatic or needle-like crystals of the hexagonal

form. The critical evaluation is given in table 5. The coherence of experimental results is

good except for the data close to the melting point. The calculated congruent melting point is

95 °C.

(continued)

COMPONENTS: (1) Lithium perchlorate; LiClO ₄ [7791-03-9] (2) Water; H ₂ O; [7732-18-5]	ORIGINAL MEASUREMENTS: Willard, H.H.; Smith, G.F. J. Am. Chem. Soc. 1923, 45, 286-96.												
VARIABLES: One temperature: 298.15 K	PREPARED BY: C.Y. Chan												
EXPERIMENTAL VALUES: Solubility ^a of lithium perchlorate in water at 25.00°C :													
<table><tr><td>mass %</td><td>g/100 cm⁻³ sln</td><td>mol %</td><td>mol dm⁻³</td><td>mol kg⁻¹</td><td>satd sln density/g cm⁻³</td></tr><tr><td>37.385</td><td>47.42</td><td>9.182^b</td><td>4.457</td><td>5.612^b</td><td>1.2683</td></tr></table>		mass %	g/100 cm ⁻³ sln	mol %	mol dm ⁻³	mol kg ⁻¹	satd sln density/g cm ⁻³	37.385	47.42	9.182 ^b	4.457	5.612 ^b	1.2683
mass %	g/100 cm ⁻³ sln	mol %	mol dm ⁻³	mol kg ⁻¹	satd sln density/g cm ⁻³								
37.385	47.42	9.182 ^b	4.457	5.612 ^b	1.2683								
^a The solid phase is a mixture of anhydrous LiClO ₄ and LiClO ₄ ·3H ₂ O . ^b Compiler's calculations.													
AUXILIARY INFORMATION													
METHOD/APPARATUS/PROCEDURE: A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 cm ³ . This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-250°C in a current of air dried with P ₂ O ₅ . Duplicate soly determinations were made, those analyses in which chloride (from thermal decomposition) was found present being rejected.	SOURCE AND PURITY OF MATERIALS: Anhydrous LiClO ₄ was prepared as described in ref. 1. ESTIMATED ERROR: Precision in temp. was ±0.01 °C . REFERENCES: 1. Willard, H.H.; Smith, G.F. J. Am. Chem. Soc. 1922, 44, 2816.												

COMPONENTS:		ORIGINAL MEASUREMENTS:			
(1) Lithium perchlorate; LiClO_4 ; [7791-03-9]		Simmons, J.P.; Ropp, C.D.L.			
(2) Water; H_2O ; [7732-18-5]		J. Am. Chem. Soc. 1928, 50, 1650-3.			
VARIABLES:		PREPARED BY:			
Temperature: 273 - 445 K.		C.Y. Chan			
EXPERIMENTAL VALUES:					
Solubility of LiClO_4 in water at various temperatures:					
t / °C	T ^a / K	mass %	mol % ^a	molality ^a /mol kg ⁻¹	Solid phase
0.0	273.2	29.90	6.736	4.009	$\text{LiClO}_4 \cdot 3\text{H}_2\text{O}$
10.0	283.2	32.88	7.660	4.604	"
20.0	293.2	35.95	8.679	5.276	"
25.0	298.2	37.48	9.216	5.635	"
30.0	303.2	38.87	9.721	5.977	"
40.0	313.2	41.97	10.911	6.798	"
64.6	337.8	50.0	14.48	9.40	"
77.9	351.1	55.0	17.15	11.49	"
89.2	362.4	60.0	20.26	14.10	"
92.3	365.5	62.5	22.01	15.67	"
94.3	367.5	65.0	23.92	17.46	"
95.1	368.3	66.32	25.01	18.51	"
94.8	368.0	66.67	25.30	18.80	"
93.2	366.4	70.0	28.3	21.9	"
92.7	365.9	70.3	28.6	22.2	"
92.5	365.7	70.33	28.64	22.28	"
93.2	366.4	70.5	28.8	22.5	$\text{LiClO}_4 \cdot \text{H}_2\text{O}$
97.3	370.5	71.0	29.3	23.0	"
108.9	382.1	72.8	31.2	25.2	"
120.7	393.9	75.0	33.7	28.2	"
136.9	410.1	80.0	40.4	37.6	"
144.0	417.2	82.5	44.4	44.3	"
148.5	421.7	85.0	49.0	53.3	"
149.3	422.5	87.5	54.2	65.8	"
144.2	417.4	90.0	60.4	84.6	"
167.5	440.7	91.04	63.24	95.50	LiClO_4
172.0	445.2	91.11	63.44	96.33	"
^a Compiler's calculations.					
AUXILIARY INFORMATION					
METHOD/APPARATUS/PROCEDURE:			SOURCE AND PURITY OF MATERIALS:		
Soly measurements at the lower temperatures were carried out by agitating an excess of anhydrous salt with water in a soly tube in a thermostat until constancy in concentration was reached at the set temperature. These determinations were supplemented by results obtained by sealing known amounts of the anhydrous salt and water			Anhydrous LiClO_4 was made using Richards and Willard's method (ref. 1). Two analyses using a modification of Lamb's method (ref. 2) gave values of 100.10 % and 100.15 % LiClO_4 purity.		
			ESTIMATED ERROR:		
			Temp. ± 0.1 °C; soly precision better than $\pm 0.1\%$ (compiler). (continued next page)		

COMPONENTS: (1) Lithium perchlorate; LiClO_4 ; [7791-03-9] (2) Alcohols: (A) Methanol (<i>methyl alcohol</i>); CH_4O ; [67-56-1] (B) Ethanol (<i>ethyl alcohol</i>); $\text{C}_2\text{H}_6\text{O}$; [64-17-5] (C) 1-Propanol (<i>n-propyl alcohol</i>); $\text{C}_3\text{H}_8\text{O}$; [71-23-8] (D) 1-Butanol (<i>n-butyl alcohol</i>); $\text{C}_4\text{H}_{10}\text{O}$; [71-36-3] (E) 2-Methyl-1-propanol (<i>iso-</i> <i>butyl alcohol</i>); $\text{C}_4\text{H}_{10}\text{O}$; [78-83-1]	ORIGINAL MEASUREMENTS: Willard, H.H.; Smith, G.F. <i>J. Am. Chem. Soc.</i> <u>1923</u> , 45, 286-96.
VARIABLES: One temperature: 298.15 K	PREPARED BY: C.Y. Chan

EXPERIMENTAL VALUES:

Solubility^a of LiClO_4 in various alcohols at 25.00°C :

solv in :	methanol	ethanol	1-propanol	1-butanol	2-methyl- 1-propanol
g/100 g sln	64.57	60.28	51.22	44.23	36.73
g/100 cm ⁻³ sln	89.44	79.41	61.49	49.25	38.94
g/100 g solvent	182.25	151.76	105.00	79.31	58.05
mol % ^b	35.44	39.66	37.23	35.59	28.80
mol dm ⁻³	8.406	7.463	5.779	6.646	3.660
mol kg ⁻¹ ^b	17.130	14.265	9.870	7.454	5.457

^a In terms of the anhydrous salt which was the solid phase.
^b Compiler's calculations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE: A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 cm ³ . This tube was then rotated end-over-end in the	SOURCE AND PURITY OF MATERIALS: Lithium perchlorate trihydrate was prepared as described in ref.1. Alcohols were purified by refluxing with calcium and fractional distillation.
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COMPONENTS:

- (1) Sodium perchlorate; NaClO_4 ;
[7601-89-0]
(2) Water; H_2O ; [7732-18-5]

EVALUATOR:

C.Y. Chan
Department of Chemistry
University of Malaya
Kuala Lumpur, Malaysia

CRITICAL EVALUATION

I BINARY SYSTEMS

System $\text{NaClO}_4\text{-H}_2\text{O}$

The original values in mass % of the solubility of sodium perchlorate in water at various temperatures reported by different groups as well as the corresponding calculated mean values are given in Table 1. Original data listed which differed by more than twice the standard deviation of the initial mean at the temperature concerned were omitted in the computation of the final mean values. Twenty-six references to the solubility of NaClO_4 in water at 298 K were found. However, it should be noted that, as seen in Table 1, certain values have been obtained from more than one compilation reference source and in such cases, it is of the evaluator's opinion that the solubility determination had been carried out only once and that these reports had referred to the same result. Only Willard and Smith (6) indicated the precision of their solubility determination ($\pm 0.05\%$). None of the other groups gave any indication of the precision of their experimental results and insufficient information was available for obtaining fair estimates of the error limits of their data in the compilation. The solid phase in equilibrium with the saturated solution at temperatures below 328 K is the monohydrate, $\text{NaClO}_4\cdot\text{H}_2\text{O}$, but at this temperature and above, the solid phase is the anhydrous salt.

Table 1. Solubility of sodium perchlorate in water at various temperatures

<u>T/K</u>	<u>mass %</u>	<u>Solid Phase</u>	<u>Source</u>
273	(60.14) ^a	$\text{NaClO}_4\cdot\text{H}_2\text{O}$	Mikheeva and Titova (79)
"	62.54	"	Freeth (7)
"	62.87	"	Cornec and Dickely (10)
"	62.89	"	Karnaikhov and Makin (20)
<u>Mean:</u>	<u>62.77</u>		

^a rejected in computation of the mean value

(continued)

CRITICAL EVALUATION (continued)

Table 1 (continued)

<i>T/K</i>	<i>mass %</i>	<i>Solid Phase</i>	<i>Source</i>
288	65.51	NaClO ₄ ·H ₂ O	Freeth (7)
"	65.63	"	Cornec and Dickely (10)
"	64.63	"	Carlson (2)
<u>Mean:</u>	<u>65.26</u>		
293	66.84	NaClO ₄ ·H ₂ O	Karnaukhov (21)
"	(67.48) ^a	"	Zaitseva and Lepeshkov (50)
"	67.58	"	Karnaukhov (17)
"	67.60	"	Freeth (7)
"	67.63	"	Cornec and Dickely (10)
"	67.64	"	Kudryakova and Karnaukhov (57)
"	67.65	"	Leboshchina and Kudryakova (85)
"	67.70	"	Molchanov (28)
298.15	67.70		Willard and Smith (6)
298	67.70	"	Chernykh, Ivanov and Alekseeva (51)
"	67.79	"	Lilich and Ovtrakt (27)
"	67.80	"	Lepeshkov and Druzhinina (34)
			Druzhinina and Paraguzova (82)
			Sal'nikova, Karnaukhov and
			Lepeshkov (65); Karnaukhov and
			Sal'nilova (55)
"	67.82	"	Karnaukhov and Makin (20)
"	67.84	"	Bestuzheva, Kinderov and
			Karnaukhov (87); Bestuzheva (88)
298.2	67.86	"	Karnaukhov and Kudryakova (32)
298	67.89	"	Karnaukhov and Tarakanov (56,64)
			Lepeshkov and Tarakanov (70)
"	67.89	"	Andronova (62); Andronova,
			Bogomolova and Gulyakova (40)
"	67.89	"	Smirnov, Ivanov and Chechneva (78)
"	67.92	"	Andronova (84)

(continued)

CRITICAL EVALUATION (continued)

Table 1 (continued)

<u>T/K</u>	<u>mass %</u>	<u>Solid Phase</u>	<u>Source</u>
298	(68.84) ^a	NaClO ₄ ·H ₂ O	Druzhinina (67)
<u>Mean:</u>	<u>67.76</u>		
303		"	Abdukarimova, Noguev and Sulaimankulov (74)
"	68.71	"	Freeth (7)
"	68.25		Caven and Bryce (11)
<u>Mean:</u>	<u>68.56</u>		
308	69.8	NaClO ₄ ·H ₂ O	Kudryakova and Karnaukhov (57) Karnaukhov and Guseva (31)
311	70.38	"	Cornec and Dickely (10)
313	70.88	"	Freeth (7)
"	70.87	"	Karnaukov (17)
"	67.63	"	Ivanov (44)
<u>Mean:</u>	<u>70.88</u>		
323	(71.28) ^a	NaClO ₄ ·H ₂ O	Carlson (2)
323	73.26	"	Cornec and Dickely (10)
323	73.16	"	Freeth (7)
323	73.15	"	Zaitseva and Lepeshkov (50)
323	73.53	"	Andronova (62)
323	73.75	"	Andronova (75)
323	73.2	"	Molchanov (28)
323.2	73.2	"	Lepeshkov, Druzhinina and Troitskii (34); Druzhinina (30)
<u>Mean:</u>	<u>73.32</u>	<u>std. dev. : 0.21</u>	
323.95	73.3	NaClO ₄ +NaClO ₄ ·H ₂ O	Freeth (7)
328	73.94	NaClO ₄	Cornec and Dickely (10)
333	74.30	"	Freeth (7)
"	74.2	"	Loseva (71)
<u>Mean:</u>	<u>74.25</u>		
335.8	74.33	NaClO ₄	Molchanov (28)
348	75.00	"	Freeth (7)

(continued)

CRITICAL EVALUATION (continued)

Table 1 (continued)

<i>T/K</i>	<i>mass %</i>	<i>Solid Phase</i>	<i>Source</i>
348	75.01	NaClO ₄	Cornec and Dickely (10)
<u>Mean</u>	<u>75.01</u>		
363	76.27	"	Kudryakova and Lepeshkov (46)
"	75.85	"	Kudryakova and Karnaukhov (57)
"	75.85	"	Karnaukhov and Troitskii (33)
<u>Mean:</u>	<u>75.99</u>		
373 K	76.75	"	Cornec and Dickely (10)
416	79.08	"	Carlson (2)

^a rejected in computation of the mean value

Based on theoretical treatments described in ref. (76), (90) a semi-empirical equation on the form given by Equation (1) was used to fit the combined original data given in table 1, after conversion of the mass % data to mole fractions. Data were selectively rejected until all the values were fitted to within $\pm 2s$ of the calculated value at each selected temperature, s being the standard error defined by $s^2 = (x_{\text{abs}} - x_{\text{calc}})^2 / (N-3)$, where N is the total number of data points.

$$F(x) = a_0 + a_1 (TK)^{-1} + a_2 \ln (TK) \quad (1)$$

where $F(x) = \ln (x^v (1-x)^n / (1 + (v-1)x)^{(n+v)})$, T = temperature, x = mole fraction, $v = 2$ for NaClO₄ (stoichiometric number for salt), the mole ratio water : salt is $n = 1$ when the solid phase is NaClO₄.H₂O and $n = 0$ when the anhydrous salt is the solid phase.

a_0 , a_1 and a_2 are the best-fit parameters, obtained using least-squares linear regression analysis. The results of the analysis are given in Table 2 for $n = 1$ and $n = 0$ in Eq. (1).

Table 2 Values of best-fit parameters with reference to Equation 1,
pertaining to data in Table 1

Temperature :	273 K - 323 K			
Solid phase :	NaClO ₄ .H ₂ O			
Correlation coefficient :	0.999 (37 data points)			
Parameters	$a_0 = -31.828$	std. error in $F(x) = 0.005$		
	$a_1 = 6.33 \times 10^2$	"	"	$a_1 = 1.7 \times 10^2$
	$a_2 = 4.55$	"	"	$a_2 = 0.57$

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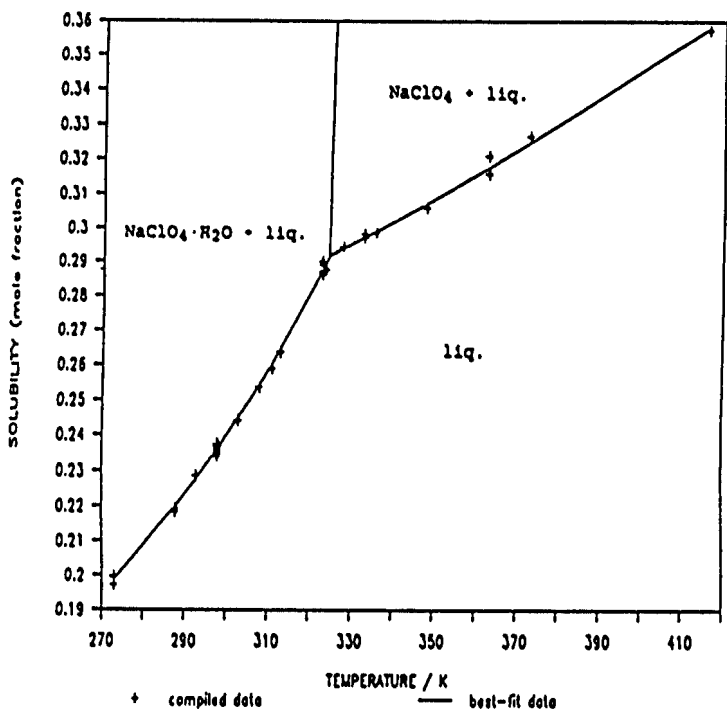
CRITICAL EVALUATION (continued)

Table 2 (continued)

Temperature :	328 K - 416 K		
Solid phase :	NaClO ₄		
Correlation coefficient :	0.996 (11 data points)		
Parameters	$a_0 = -17.854$	std. error in F(x) = 0.008	
	$a_1 = 4.09 \times 10^2$	" " "	$a_1 = 3.4 \times 10^2$
	$a_2 = 2.355$	" " "	$a_2 = 0.94$

The average difference between calculated and observed values was 0.38 % of the observed solubility value.

Figure 1. Solubility-temperature plot for the NaClO₄-water system
Solid line represents smoothed data based on Equation 1.



The solubility-temperature plot is shown in Figure 1 and Table 3 lists recommended and tentative smoothed values of the solubility of sodium perchlorate in water, computed based on Equation 1 using the appropriate parameters at the selected temperatures. The recommended values are for the solubilities at 298.15 K and 323.15 K, where original data were

(continued)

CRITICAL EVALUATION: (continued)

reported by more than three independent groups at each of these temperatures.

Table 3. Smoothed data calculated for the solubility of NaClO_4 in water at selected temperatures.

$t/^{\circ}\text{C}$	T/K	Solubility		Solid phase	Status
		mol fraction	molality		
0	273.15	0.1987	13.77	$\text{NaClO}_4 \cdot \text{H}_2\text{O}$	tentative
5	278.15	0.2056	14.36	"	"
10	283.15	0.2126	14.99	"	"
15	288.15	0.2200	15.66	"	"
20	293.15	0.2278	16.38	"	"
25	298.15	0.2362	17.16	"	recommended
30	303.15	0.2451	18.02	"	tentative
35	308.15	0.2545	18.95	"	"
40	313.15	0.2647	19.98	"	"
45	318.15	0.2757	21.13	"	"
50	323.15	0.2876	22.41	"	recommended
51.8	324.95	0.2922	22.91	$\text{NaClO}_4 \cdot \text{H}_2\text{O} +$ NaClO_4	tentative
55	328.15	0.2942	23.14	NaClO_4	"
60	333.15	0.2975	23.50	"	"
65	338.15	0.3008	23.87	"	"
70	343.15	0.3041	24.26	"	"
75	348.15	0.3075	24.65	"	"
80	353.15	0.3109	25.04	"	"
85	358.15	0.3144	25.45	"	"
90	363.15	0.3179	25.87	"	"
95	368.15	0.3215	26.30	"	"
100	373.15	0.3251	26.74	"	"
105	378.15	0.3287	27.19	"	"
110	383.15	0.3324	27.64	"	"
115	388.15	0.3362	28.11	"	"

(continued)

CRITICAL EVALUATION (continued)

Table 3. (continued)

t/°C	T/K	Solubility		Solid phase	Status
		mol fraction	molality		
120	393.15	0.3400	28.59	NaClO ₄	tentative
125	398.15	0.3438	29.08	"	"
130	403.15	0.3477	29.59	"	"
135	408.15	0.3516	30.10	"	"
140	413.15	0.3556	30.63	"	"
145	418.15	0.3596	31.17		

The peritectic transition from the monohydrate to anhydrous sodium perchlorate was determined to occur at 324.96 K and at this point the mole fraction of the salt in the solution at equilibrium is 0.2922, molality was 22.91 mol kg⁻¹, based on the evaluator's analysis. It should be noted that these values are slightly higher than the values of 0.2877 (mole fraction) and 22.42 mol kg⁻¹ calculated from Freeth's data (7) at 323.95 K for the saturated solution in equilibrium with both the anhydrous salt and its monohydrate. The peritectic temperature reported by Cornec and Dickely (10) was 325.90 K.

Cornec and Dickely (10) also reported solubility data in mamount concentration units and saturated solution densities (see compilation) over the temperature range 273 K - 373 K while similar data were reported by Carlson (2) at 288 K, 323 K and 416 K. Carlson's solubility values of 8.79 mol dm⁻³ at 288 K and 10.08 mol dm⁻³ at 323 K are somewhat lower than Cornec and Dickely's values of 8.91 mol dm⁻³ and 10.46 mol dm⁻³ at the corresponding temperatures. There is better agreement between Willard and Smith's value (6) of 9.301 mol dm⁻³ at 298.15 K, Cornec and Dickely's value of 9.33 mol dm⁻³ and the value of 9.309 mol dm⁻³ converted from the recommended molal solubility (Table 3) using Willard and Smith's value of 1.6821 g cm⁻³ for the saturated solution density (6) at the same temperature. Table 4 compares solubility values (in mol dm⁻³) reported by Cornec and Dickely (10) and those calculated using the smoothed data given in Table 3 and Cornec and Dickely's saturated solution density values at various temperatures.

(continued)

COMPONENTS: (1) Sodium perchlorate; NaClO ₄ ; [7601-89-0] (2) Water; H ₂ O; [7732-18-5]	ORIGINAL MEASUREMENTS: Willard, H.H.; Smith, G.F. <i>J. Am. Chem. Soc.</i> <u>1923</u> , 45, 286-96.												
VARIABLES: One temperature: 298.15 K	PREPARED BY: C.Y. Chan												
EXPERIMENTAL VALUES: Solubility ^a of sodium perchlorate in water at 25.00°C :													
<table><tr><td>mass %</td><td>g/100 cm⁻³ sln</td><td>mol %</td><td>mol dm⁻³</td><td>mol kg⁻¹ m₁</td><td>satd sln. density/g cm⁻³</td></tr><tr><td>67.70</td><td>113.88</td><td>23.57^b</td><td>9.301^b</td><td>17.118^b</td><td>1.6821</td></tr></table>		mass %	g/100 cm ⁻³ sln	mol %	mol dm ⁻³	mol kg ⁻¹ m ₁	satd sln. density/g cm ⁻³	67.70	113.88	23.57 ^b	9.301 ^b	17.118 ^b	1.6821
mass %	g/100 cm ⁻³ sln	mol %	mol dm ⁻³	mol kg ⁻¹ m ₁	satd sln. density/g cm ⁻³								
67.70	113.88	23.57 ^b	9.301 ^b	17.118 ^b	1.6821								
<p>^a The solid phase was mixture of the anhydrous salt and that crystallized from the sat. sln (probably NaClO₄.H₂O).</p> <p>^b Compiler's calculations.</p>													
AUXILIARY INFORMATION													
METHOD/APPARATUS/PROCEDURE: A satd sln of the salt was prepared at a temperature slightly above 25°C and sealed together with about 1 g of the anhydrous salt in a solubility tube, capacity 18-20 cm ³ . This tube was then rotated end-over-end in the thermostat bath at 25.00°C for 24-48h and stood vertically to allow the solids to settle. Samples of the clear satd sln were then analysed for solute content by an evaporation-to-dryness method using Pt crucibles. The salt was dried to const. wt. at 250 °C in a current of air dried with P ₂ O ₅ . Duplicate soly determinations were made, those analyses in which chloride was found present being rejected.	SOURCE AND PURITY OF MATERIALS: Anhyd. NaClO ₄ was prepared from pure sodium carbonate and slight excess of purified HClO ₄ (ref.1) by crystallization above 50 °C (ref. 2). After centrifugal separation, the solid was dried in a current of dry air at 250 °C.												
	ESTIMATED ERROR: Precision in temp. was ± 0.01 °C Precision in solubility data was ± 0.05 %.												
	REFERENCES: 1. Willard, H.H.; <i>J. Am. Chem. Soc.</i> <u>1912</u> , 34, 1480. 2. Willard, H.H.; Smith, G.F. <i>J. Am. Chem. Soc.</i> <u>1922</u> , 44, 2816.												

COMPONENTS: (1) Sodium perchlorate; NaClO ₄ ; [7601-89-0] (2) Water; H ₂ O; [7732-18-5]	ORIGINAL MEASUREMENTS: Freeth, F.A. Rec. Trav. Chim. Pays-Bas <u>1924</u> , 43, 475-507.																																																																																			
VARIABLES: Temperature: 241 - 348 K. Composition.	PREPARED BY: C.Y. Chan																																																																																			
EXPERIMENTAL VALUES: Solubility system NaClO ₄ -H ₂ O at various temperatures :																																																																																				
<table><tr><th colspan="4">Solubility of NaClO₄</th><th>Solid</th></tr><tr><th>t/ °C</th><th>mass %</th><th>mol % ^a</th><th>molality^a/ mol kg⁻¹</th><th>phase</th></tr><tr><td>75</td><td>75.0</td><td>30.62</td><td>24.50</td><td>NaClO₄</td></tr><tr><td>60</td><td>74.3</td><td>29.84</td><td>23.61</td><td>"</td></tr><tr><td>50.8</td><td>73.3</td><td>28.77</td><td>22.42</td><td>NaClO₄ + NaClO₄.H₂O</td></tr><tr><td>50</td><td>73.16</td><td>28.63</td><td>22.26</td><td>NaClO₄.H₂O</td></tr><tr><td>40</td><td>70.88</td><td>26.37</td><td>19.88</td><td>"</td></tr><tr><td>30</td><td>68.71</td><td>24.42</td><td>17.94</td><td>"</td></tr><tr><td>15</td><td>65.51</td><td>21.84</td><td>15.51</td><td>"</td></tr><tr><td>0</td><td>62.54</td><td>19.72</td><td>13.64</td><td>"</td></tr><tr><td>-0.3</td><td>10</td><td>1.6</td><td>0.91</td><td>ice</td></tr><tr><td>-6.8</td><td>20</td><td>3.5</td><td>2.0</td><td>"</td></tr><tr><td>-11.1</td><td>30</td><td>5.9</td><td>3.5</td><td>"</td></tr><tr><td>-17.8</td><td>40</td><td>8.9</td><td>5.5</td><td>"</td></tr><tr><td>-22</td><td>45</td><td>10.7</td><td>6.7</td><td>"</td></tr><tr><td>-32</td><td>56</td><td>15.8</td><td>10.4</td><td>NaClO₄.H₂O + ice</td></tr></table>					Solubility of NaClO ₄				Solid	t/ °C	mass %	mol % ^a	molality ^a / mol kg ⁻¹	phase	75	75.0	30.62	24.50	NaClO ₄	60	74.3	29.84	23.61	"	50.8	73.3	28.77	22.42	NaClO ₄ + NaClO ₄ .H ₂ O	50	73.16	28.63	22.26	NaClO ₄ .H ₂ O	40	70.88	26.37	19.88	"	30	68.71	24.42	17.94	"	15	65.51	21.84	15.51	"	0	62.54	19.72	13.64	"	-0.3	10	1.6	0.91	ice	-6.8	20	3.5	2.0	"	-11.1	30	5.9	3.5	"	-17.8	40	8.9	5.5	"	-22	45	10.7	6.7	"	-32	56	15.8	10.4	NaClO ₄ .H ₂ O + ice
Solubility of NaClO ₄				Solid																																																																																
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60	74.3	29.84	23.61	"																																																																																
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-32	56	15.8	10.4	NaClO ₄ .H ₂ O + ice																																																																																
^a Compiler's calculations.																																																																																				
AUXILIARY INFORMATION																																																																																				
METHOD/APPARATUS/PROCEDURE: The saturation apparatus was similar to that used by Van't Hoff (ref.1) and samples of clear satd sln were taken using a weight-pipette. Na ⁺ was determined as Na ₂ SO ₄ by addition of pure sulphuric acid to the sln in silica basins and evaporating at a low red heat. All analyses were carried out in duplicate. Solid phase compositions were determined using Schreinemakers' method. Gas-heated thermostats were used and thermometers were checked against N.P.L. Standards.		SOURCE AND PURITY OF MATERIALS: NaClO ₄ was prepared from very pure ammonium perchlorate (% purity not stated) and an aqueous sln of pure NaOH. Source and other details not given.																																																																																		
		ESTIMATED ERROR: No estimation.																																																																																		
		REFERENCES: 1. Van't Hoff, J.H. Zur Bildung der Ozeanischen Salzablagerungen Wieweg, Braunschweig <u>1905</u> , 1; <u>1902</u> , 2.																																																																																		

(continued next page)

COMPONENTS:

- (1) Sodium perchlorate; NaClO_4 ;
[7601-89-0]
(2) Water; H_2O ; [7732-18-5]

ORIGINAL MEASUREMENTS:

Freeth, F.A.

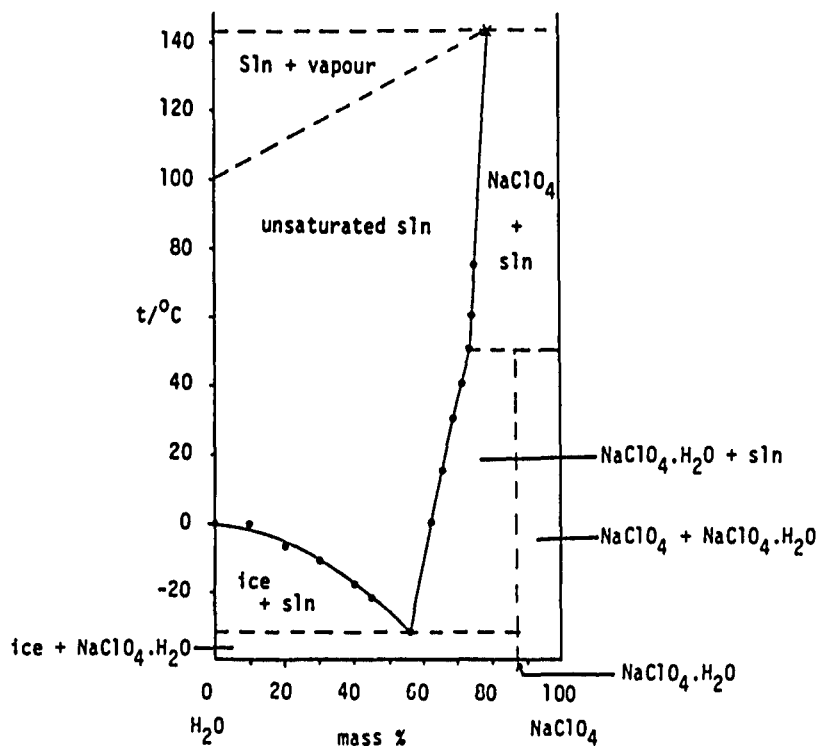
Recl. Trav. Chim. Pays-Bas 1924,
43, 475-507.

EXPERIMENTAL VALUES:(continued)

COMMENTS AND/OR ADDITION DATA:

The temperature-composition phase diagram for the system $\text{NaClO}_4\text{-H}_2\text{O}$
(Pressure = 101325 Pa) is given below.

x - ref. B. Carlson



COMPONENTS:					ORIGINAL MEASUREMENTS:	
(1) Sodium perchlorate; NaClO ₄ ; [7601-89-0]					Cornec, E.; Dickely, J.	
(2) Water; H ₂ O; [7732-18-5]					Bul. Soc. Chim. (France) 1927, 41, 1017-27.	
VARIABLES:					PREPARED BY:	
Temperature: 273 - 373 K					C.Y. Chan	
EXPERIMENTAL VALUES:						
Solubility of sodium perchlorate in water at various temperatures :						
t/ °C	g dm ⁻³	Solubility of NaClO ₄			density/ g cm ⁻³	Solid phase
		mass %	mol % ^a	mol dm ⁻³		
100	1330	76.75	32.69	10.86	1.758	NaClO ₄
75	1318	75.01	30.63	10.76	1.757	"
55	1298	73.94	29.45	10.60	1.756	"
50	1281	73.26	28.73	10.46	1.749	NaClO ₄ .H ₂ O
38	1206	70.38	25.90	9.85	1.713	"
25	1142	67.63	23.67	9.33	1.683	"
15	1091	65.63	21.93	8.91	1.663	"
0	-	62.87	19.94	-	-	"
^a Compiler's calculations.						
AUXILIARY INFORMATION						
METHOD/APPARATUS/PROCEDURE:						
No details of saturation method given. The saturated solutions were evaporated in a water-bath and the solids dried in an oven at 110 °C, cooled and weighed in stoppered flasks.						
Transition temperature detn: A mixture (about 40g) of hydrated sodium perchlorate with a small amount of water was put in a wide test-tube placed inside a wider tube, and the whole apparatus was placed in a thermostated oven set at a few degrees under the transition temperature. Then some crystals of the salt were added. The transition temperature which remained steady to ± 0.03 °C for at least 20 min. was measured using a Baudin thermometer.						
SOURCE AND PURITY OF MATERIALS: Commercial sodium perchlorate was purified by several recrystallizations before use.						
ESTIMATED ERROR: Not stated.				REFERENCES:		
(continued next page)						

COMPONENTS: (1) Sodium perchlorate; NaClO ₄ ; [7601-89-0] (2) Water; H ₂ O; [7732-18-5]		ORIGINAL MEASUREMENTS: Cornec, E.; Dickely, J. Bul. Soc. Chim. (France) 1927, 41, 1017-27.				
EXPERIMENTAL VALUES: (continued)						
COMMENTS AND/OR ADDITIONAL DATA The transition temperature for the transformation NaClO ₄ .H ₂ O = NaClO ₄ was measured as 52.75 °C. Compositions of super-saturated aqueous NaClO ₄ solutions (solid phase was the anhydrous salt) are as follows :						
t / °C		mass % (1)	mol % (1) ^a			
38		72.83	28.28			
25		72.21	27.66			
15		71.68	27.14			
^a Compiler's calculations.						
COMPONENTS: (1) Sodium perchlorate; NaClO ₄ ; [7601-89-0] (2) Water; H ₂ O; [7732-18-5]		ORIGINAL MEASUREMENTS: Carlson, B. Festkrift ed. Klason, P., (Norstedt, Stockholm, 1910), 262-3.				
VARIABLES: Temperature: 288 K, 323 K and 416 K.		PREPARED BY: C.Y. Chan				
EXPERIMENTAL VALUES: Solubility system NaClO ₄ -H ₂ O at various temperatures :						
t/ °C	g dm ⁻³	Solubility of NaClO ₄		density/	Solid phase	
		mass %	mol % ^a	mol dm ⁻³		
15	1076	64.63	21.19	8.79	1.666	NaClO ₄ .H ₂ O
50	1234	71.28	26.75	10.08	1.731	"
143	1414	79.08	35.74	11.55	1.789	NaClO ₄
^a Compiler's calculations.						
AUXILIARY INFORMATION						
METHOD/APPARATUS/PROCEDURE: No details given.				SOURCE AND PURITY OF MATERIALS: Not stated.		
ESTIMATED ERROR: Not stated.				REFERENCES:		