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SOLUBILITY OF LITHIUM HYDROXIDE IN WATER AND VAPOR PRESSURE OF SOLUTIONS OF LITHIUM HYDROXIDE ABOVE 220 F

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The solubility of lithium hydroxide in water was determined for the temperature interval between 220 and 650 F. The literature furnished data for temperatures below 200 F. A maximum in the curve was found at about 240 F and a minimum at 480 F. The variations in solubility, however, were relatively small. For example, at 40 F, the solubility is 12.7 g LiOH per 100 g H_2O*; at 240 F, it is 17.7; at 480 F, it is 13.4; and at 650 F, it is 16.5.*

The vapor pressures of 4.76 w/o (2.09 molal), 8.59 w/o (3.92 molal), and saturated (approximately 6.25 molal) lithium hydroxide solutions were measured as a function of temperature. At about 688 F, the more dilute solution showed a depression in vapor pressure of about 130 psi, the intermediate 154 psi, and the saturated 158 psi.

The more dilute solution showed a greater deviation from Raoult's law than did the other two.

Vapor-pressure data for sodium hydroxide solutions were compared with those for lithium hydroxide of similar concentration by weight and molality.

INTRODUCTION

Lithium hydroxide is being used as an agent for the control of pH in the primary coolant-water circuits of pressurized nuclear reactors. Basic information on the properties of lithium hydroxide solutions at elevated temperatures is important. For example, both corrosion and heat transfer are related to a buildup of concentrated films or crystalline deposits. Solubility data, therefore, are necessary to help predict the possibility of deposit formation from additions of lithium hydroxide. Similarly, vapor-pressure data are of value in helping establish the conditions under which concentrated films might occur at steam-blanketed areas. The present study was made to furnish data in each of the areas just mentioned.

APPARATUS AND EXPERIMENTAL PROCEDURES

The apparatus for obtaining the solubility data is schematically shown in Figure 1. The reaction vessel (A) was a standard American Instrument Company Type 347 stainless steel autoclave with a drop-in nickel liner. The liner, having a capacity of 700 ml, was made of welded nickel sheet. The autoclave was heated by a resistance-type electric heater (B) controlled by a Foxboro controller (C_2) through a thermocouple (T_1) placed in the heater wall midway between the top and bottom. Heat was supplied to the top of the autoclave and the lines leading from it by a Glas-Col heating mantle (H). The reaction vessel and heater were rocked 30 deg from the vertical at ten strokes per min to agitate the solutions. The head of the autoclave contained a thermocouple well which extended

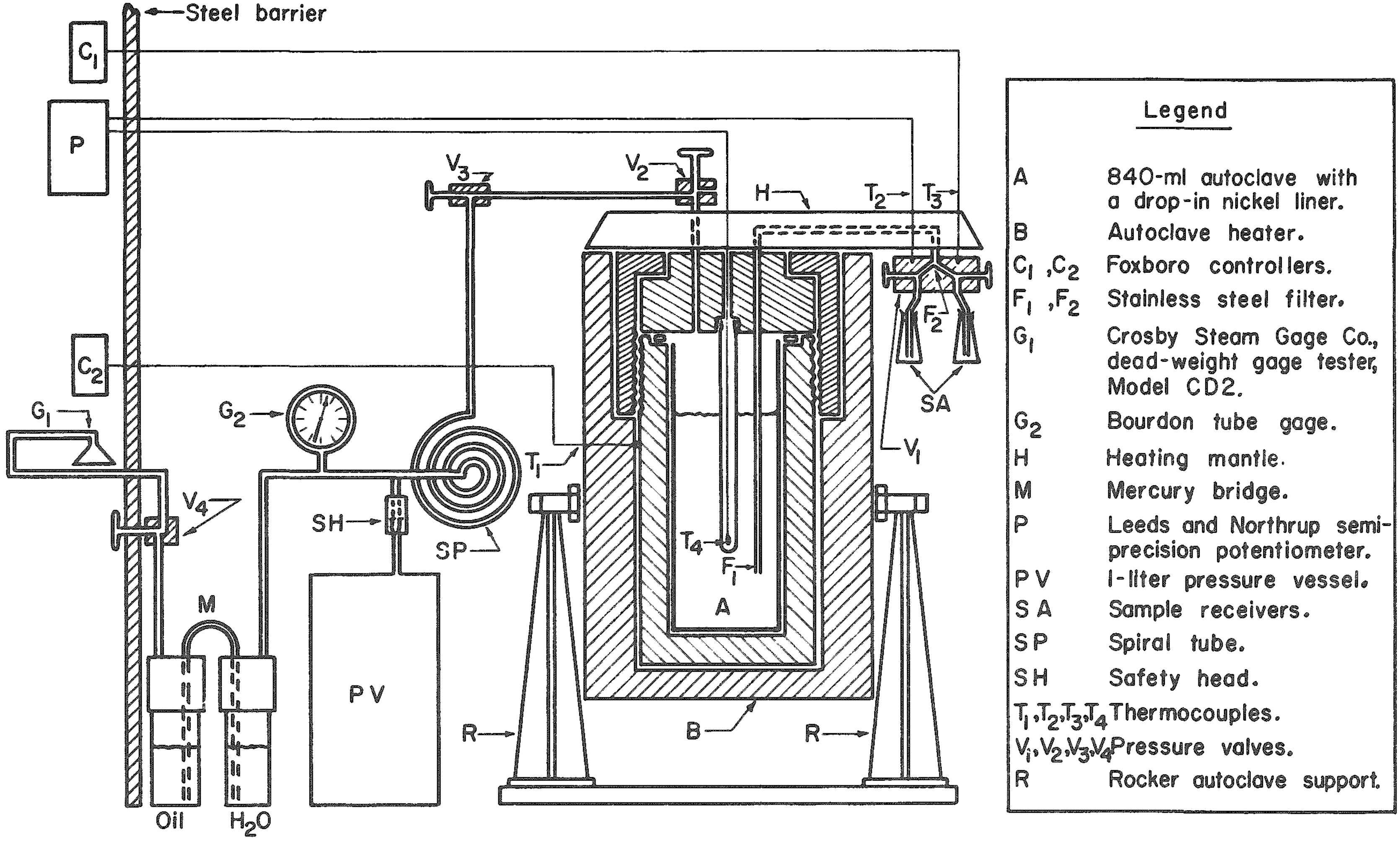

FIGURE 1. APPARATUS FOR SOLUBILITY AND VAPOR PRESSURE STUDIES AT ELEVATED TEMPERATURES

two-thirds of the way into the chamber. Two openings for 1/4-in. high-pressure fittings were also placed in the head. One opening was fitted for measuring pressures and for evacuating; the other opening was fitted for sampling the supernatant liquid in the chamber. The line leading directly from the head of the autoclave included a valve (V_2) for bleedoff, or evacuation, and a shutoff valve (V_3). This line led through a spiral to a Crosby Steam Gage Company Model CD2 dead-weight gage tester (G_1) for measuring the pressure on the system. The condensed vapor from the autoclave was separated from the oil in the gage by a mercury bridge (M). This line also included a 5000-psi Bourdon-type gage (G_2) and a safety head with a 4200-psi rupture disk (SH). The outlet from the safety head led into a 1-liter pressure vessel (PV) which would contain the solution should the temperature or pressure get out of control.

Samples of the liquid were drawn through a pressure tubing line extending through the head of the autoclave down two-thirds of the way into the chamber. A small stainless steel frit (F_1) having a 5-μ rated opening was inserted in the end of this tube to filter the supernatant liquid. The sampling tube leading from the autoclave head contained a three-way Y-type pressure valve (V_1). This valve was heated by two small plate heaters. The temperature of the valve was determined by thermocouples (T_2 and T_3) placed in recesses in the valve. A Foxboro instrument furnished automatic control. Another small stainless steel frit (F_2) was inserted in the sampling line just ahead of the valve (V_1). This valve permitted attachment of two sample receivers (SA) at the same time.

Lithium hydroxide monohydrate, reagent grade, containing 2.2 w/o lithium carbonate was used as a starting material. A quantity of the solid reagent was stirred in distilled water for 10 min, allowed to settle, and the supernatant liquid decanted into a polyethylene bottle. This solution, containing about 10 g of LiOH per 100 g of water, was run into a measuring burette and then through a column containing Rohm and Haas Amberlite XE154 lithium-form ion-exchange resin to remove the carbonate. All vents in the solution line were fitted with tubes containing Ascarite to reduce CO_2 contamination. Carbonate could not be detected in the solution from the ion-exchange column by a double-indicator titration.

The assembled autoclave was evacuated and 500 ml of the lithium hydroxide solution was drawn directly from the ion-exchange column through the sampling valve into the cold autoclave. The solution was heated to 250 F and concentrated by allowing 400 ml of condensed water vapor to escape through the valve (V_2). The autoclave was then cooled, 400 ml of solution was drawn in, the autoclave was reheated, and the water was evaporated. Four successive 400-ml portions of the solution were added and evaporated. The final 400-ml portion was not evaporated. At this stage, the autoclave contained approximately 250 g of purified lithium hydroxide, some in solution and some in crystal form, and 500 g of water.

Solubility Determinations

Sampling of the supernatant liquid at temperature was accomplished by opening the sampling valve and allowing the vapor pressure of the system to force the liquid through the filters into the sample receiver. The receivers were 300-ml Erlenmeyer flasks fitted with two-hole rubber stoppers. A stainless steel tube, with a pressure fitting for attaching to the sampling valve, was inserted through one hole in the stopper. This tube extended nearly to the bottom of the flask. The assembled receiver, consisting of flask, stopper, tube, and about 150 ml of distilled water, was weighed prior to attachment to the sampling valve. About 5 to 7 g of the supernatant liquid was slowly taken into the cold water in the flask. The receiver was then reweighed and the weight of the sample obtained by difference. The inside of the tube was rinsed into the flask and the total LiOH determined by titrating with standard hydrochloric acid using a phenolphthalein indicator. Two receivers were used at each sampling, one to receive the purge solution drawn from the line and the other for the sample.

Three different techniques were used to establish equilibrium:

(1) The solution in contact with the solid LiOH was agitated at temperature for 16 hr before sampling. Samples were taken every 3 hr thereafter until successive analyses showed that equilibrium had been attained. The temperature was then raised 50 F and the procedure repeated.

(2) The second procedure is similar to (1) except that, 1 hr before sampling, 10 g of water vapor was allowed to escape from the head space above the solution. This effected a concentration of the solution and helped confirm a condition of equilibrium.

(3) In the third procedure, the solution was sampled as the temperature was decreased in 50 F intervals from 650 F. Thus, further confirmation of equilibrium conditions could be established.

Since the vapor pressure was too low to force the liquid through the filters at temperatures below 300 F, sampling at these temperatures was accomplished by partially evacuating the sample receiver. The data at 650 F were obtained without a filter in the sampling line. Temperature and pressure conditions at this point were such that the solution would flash evaporate in the line between the filter and the valve when the valve was opened. The close agreement of the successive samples indicated that any suspended material had settled and only solution came through the valve. Samples could not be obtained at 680 F with the present experimental arrangements as the slightest relief of pressure at the valve caused the line to plug.

Vapor-Pressure Determinations

The vapor-pressure determinations were run in a static 1-liter all-nickel autoclave fabricated by Autoclave Engineers.

A temperature-pressure calibration curve for the gage and thermocouple was first determined using distilled water. The data presented later have been adjusted using this calibration curve. Control of the heat to the various sections of the autoclave heater was such that a variation of temperature from top to bottom of the autoclave was no greater than ± 1 F. The dead-weight pressure gage could be read with an accuracy of ± 1 psi at the low pressures to ± 5 at the high pressures.

The autoclave was filled by a procedure similar to that described for the solubility determinations.

When vapor-pressure measurements were made on the dilute solutions at increasing temperatures, solution was withdrawn to maintain a minimum head space in the autoclave as expansion occurred. In the determinations using saturated solutions, some of the water vapor was allowed to escape to maintain the head space required. The volume of the head space was not controlled during the determination with decreasing temperatures.

EXPERIMENTAL RESULTS

Solubility Data

The solubility data are presented in Table 1. The analyses of each sample obtained at equilibrium are shown in order to illustrate the reproducibility of the data. The average values are plotted in Figure 2 and show the close agreement obtained by the three different techniques. Values from A. Seidell, Solubilities of Inorganic and Metal Organic Compounds(1), for temperatures below 220 F are included in the plotted data.

The results are quite interesting in several respects. First, the solubility curve shows a maximum value at about 240 F and a minimum at about 480 F and at room temperature. Phase changes could possibly cause the changes in shape of the solubility curve. Additional studies would be required to establish these changes. Other salts such as sodium sulfate and calcium sulfate also exhibit inverse solubility as a function of temperature.

The data also show that increasing the temperature as high as 650 F did not greatly affect the solubility of lithium hydroxide. For example, values range from a minimum of about 12.7 g LiOH per 100 g H_2O at room temperature to a maximum of only about 17.7 g at 240 F.

In contrast to these results, the solubility values of sodium hydroxide show a rapid and fairly uniform increase as temperatures are raised. Values from the International Critical Tables(2) and from Seidell are presented in Table 2 for comparison with those for lithium hydroxide obtained in the present study. At 140 F, the solubility of sodium hydroxide is about 20 times that for lithium hydroxide; at 375 F, it is almost 35 times greater; and at 563 F, it is about 200 times greater.

Hall(3), Rivers(4), and Williams(5) have discussed the effect of steam blanketing and superheat as related to caustic buildup on operating boiler tubes. The solubility values for lithium hydroxide indicate that only a small amount of superheat might be required to result in actually exceeding the solubility limit of lithium hydroxide under stagnant conditions on a heat-exchanger surface. The relatively low solubility of lithium hydroxide may, therefore, make it less hazardous from the corrosion standpoint than sodium hydroxide. That is, it appears that lithium hydroxide solutions no greater than

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(1) References at end.

TABLE 1. SOLUBILITY OF LITHIUM HYDROXIDE IN WATER
(G LiOH per 100 G H_2O)

Run 1, Temperature Increasing		Run 2, Temperature Increasing Plus Evaporation		Run 3, Temperature Increasing Plus Evaporation		Run 4, Temperature Decreasing	
Temperature, F	Solubility	Temperature, F	Solubility	Temperature, F	Solubility	Temperature, F	Solubility
				220	17.5		
					18.1		
					17.5		
					Avg 17.7		
				250	17.7	235	17.8
					17.6		17.5
					17.7		Avg 17.65
					17.7		
					17.8	288	16.6
					Avg 17.7		16.6
							Avg 16.6
300	16.6	300	16.1	300	16.4	330	15.5
	17.0		16.4				15.3
	16.6		16.4				Avg 15.4
	Avg 16.7		Avg 16.3				
350	15.4	355	15.2			375	14.8
	15.35		15.2				14.6
	15.3		Avg 15.2				Avg 14.7
	Avg 15.3						
405	14.1	406	14.7			423	14.0
	14.3		14.3				13.7
	Avg 14.2		14.2				Avg 13.85
			Avg 14.4				
455	13.7	450	13.7			485	13.6
	13.2		13.5				13.45
	Avg 13.45		Avg 13.6				13.6
							Avg 13.5
503	13.6	599	13.6			565	14.4
	13.4		13.5				14.7
	Avg 13.5		Avg 13.55				Avg 14.55
550	14.25		14.1				
	14.0		14.2				
	14.4		Avg 14.15				
	Avg 14.2						
600	14.74	606	14.8	650	16.3	600	14.7
			15.1		16.8		14.8
			15.0		16.2		Avg 14.75
			Avg 14.95		Avg 16.5		

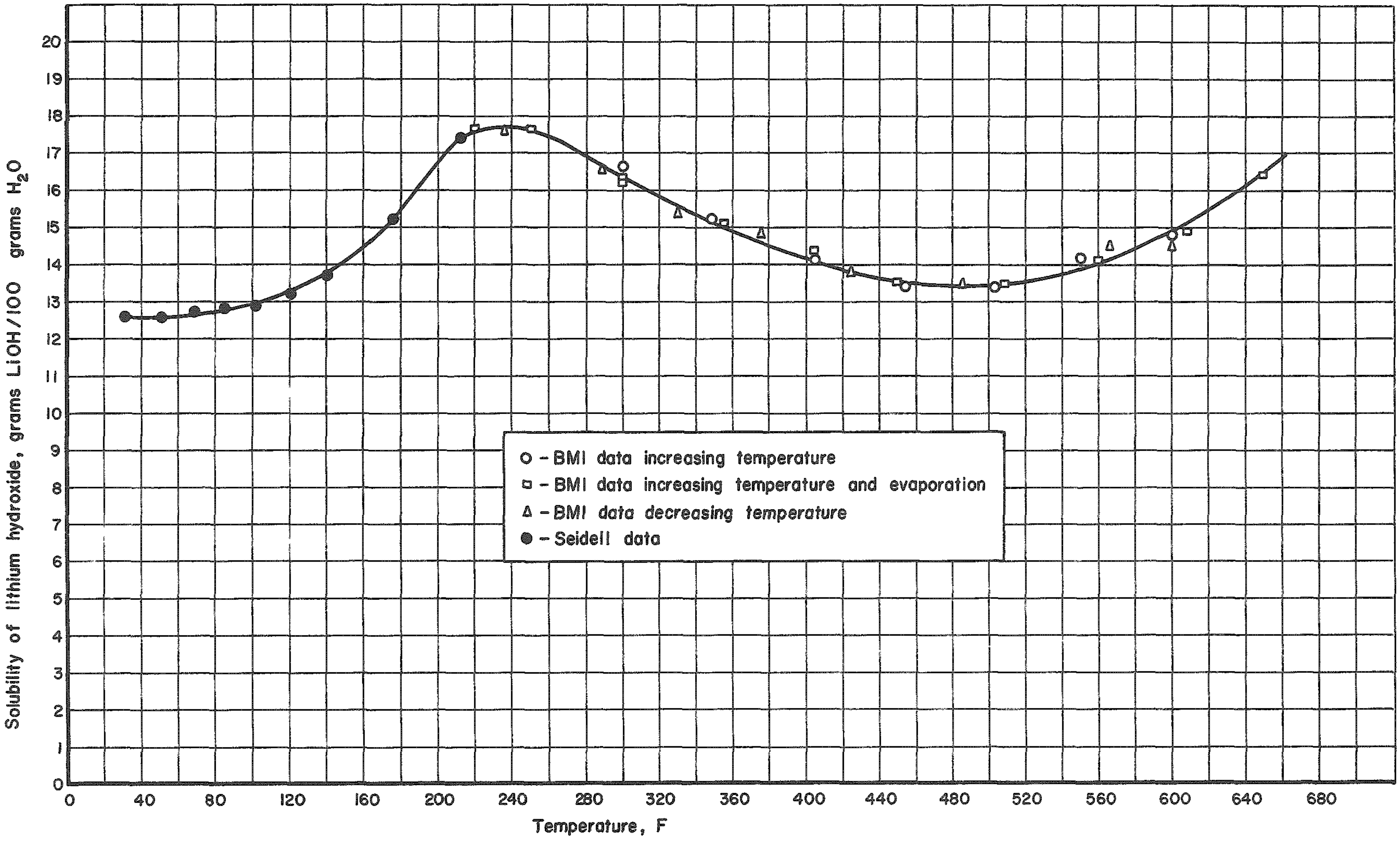

FIGURE 2. SOLUBILITY OF LITHIUM HYDROXIDE IN WATER

about 15 w/o in concentration can be built up before crystallization occurs. Experimental work would be necessary to determine the corrosivity of such lithium hydroxide solutions. In the case of sodium hydroxide, solutions whose concentration is 5 to 10 w/o and greater are required to produce stress-corrosion cracking or general attack. Because sodium hydroxide is very soluble, highly concentrated solutions can be formed at superheated areas.

TABLE 2. SOLUBILITY OF HYDROXIDES IN WATER AT ELEVATED TEMPERATURES(a)

	Solubility, g per 100 g H_2O	
Temperature, F	LiOH	NaOH
140	13.8	292
176	15.3	314
194	16.5	-
212	17.5	328
248	17.7	364
284	16.8	392
302	16.6	-
320	16.0	424
356	15.2	464
374	14.7	496
402	14.2	600
509	13.5	1067
563	14.1	2930
611	15.0	Melting point

(a) From References (1) and (2).

Vapor-Pressure Data

The vapor pressures measured for a saturated lithium hydroxide solution and for solutions containing 5 and 9.4 g of LiOH per 100 g of H_2O are presented in Tables 3, 4, and 5. The values for the depression of the vapor pressure of water, Δp, as affected by the addition of lithium hydroxide are also presented in the tables and graphically illustrated in Figures 3, 4, and 5. Included in the curves are calculated values for Δp obtained by Raoult's law, $p_1 = p_0 \frac{N}{N + n}$. Values for sodium hydroxide as taken from the International Critical Tables(2) are presented for the same molalities and weight per cent concentrations as those used for lithium hydroxide. It should be noted that values were obtained on an ascending temperature schedule and also on a descending schedule.

Table 3 shows that the vapor depression of the 4.76 w/o (2.09 molal) lithium hydroxide solution amounts to about 130 psi at about 685 F. In Figure 3, these Δp values are compared with those for sodium hydroxide.

It can be seen that lithium hydroxide causes somewhat more depression than does sodium hydroxide at equal concentrations by weight. At equal molalities, however, sodium hydroxide causes more lowering of pressure.

TABLE 3. VAPOR PRESSURE OF A SOLUTION OF 5 G LiOH PER 100 G H_2O

Temperature, F	A, Vapor Pressure of H_2O (Steam-Table Data), psia	B, Vapor Pressure of the Solution, psia	Vapor-Pressure Depression ($A - B = \Delta p$), psi
Ascending Temperature			
238	24	23	1
303	70	69	1
363	159	156	3
399.5	245	234	11
424	322	304	18
438	373	352	21
446	405	366	39
474	534	489	45
513	765	710	55
548	1028	967	61
580	1327	1259	68
584	1368	1286	82
599	1538	1451	87
604	1710	1620	90
669	2515	2424	91
672.5	2574	2469	103
680.5	2714	2604	110
687.5	2840	2709	131
Decending Temperature			
683	2763	2634	129
650.5	2215	2109	104
631	1940	1834	106
593	1465	1368	97
579	1310	1224	86
563	1161	1084	77
550.5	1048	984	64
529	878	819	59
504	706	656	50
484	588	538	50
473	529	484	45
461	471	429	42
450	423	385	38
441	386	354	32
433	355	329	26
423	319	303	16
413	286	269	17
407	267	252	15
384	205	194	11
359	151	144	7
334	108	102	6
307	74	70	4
293	60	58	2
277	47	46	1
237	22.5	22	0.5

TABLE 4. VAPOR PRESSURE OF A SOLUTION OF 9.3 G LiOH PER 100 G H_2O

Temperature, F	A, Vapor Pressure of H_2O (Steam-Table Data), psia	B, Vapor Pressure of the Solution, psia	Vapor-Pressure Depression ($A - B = \Delta p$), psi
		Ascending Temperature	
275	45	44	1
289	56.5	55	1.5
298	65	63	2
312	80	78	2
354	141	135	6
360	153	147	6
379	193	181	12
395	233	217	16
406	265	248	17
418	303	283	20
435	364	340	24
497	663	628	35
531	892	849	43
547	1020	957	63
551	1054	964	90
595	1487	1374	113
613	1705	1589	116
644	2128	2094	134
654.5	2280	2139	141
667	2478	2330	148
689	2876	2722	154
		Descending Temperature	
684	2763	2616	147
658	2330	2289	141
649	2193	2079	114
644.5	2133	2028	105
623	1826	1732	94
612	1686	1609	77
572	1247	1185	62
566	1189	1134	55
506.5	722	674	48
480.5	568	534	34
430	343	312	31
396	237	214	23
362	157	141	16
316	85	76	9
251	30	28	2

TABLE 5. VAPOR PRESSURE OF SATURATED LITHIUM HYDROXIDE SOLUTIONS

Temperature, F	A, Vapor Pressure of H_2O (Steam Table Data), psia	B, Vapor Pressure of the Solution, psia	Vapor-Pressure Depression, (A-B=Δp), psi
	Ascending Temperature		
261	36	32	4
263	37	33	4
273.5	45	41	4
282	51	46	5
307.5	75	70	5
328	100	94	6
349	133	124	9
363	158	146	12
387	213	187	26
398	241	212	29
415	281	251	30
433	355	318	37
438	374	334	40
456	431	384	47
470	515	462	48
490	622	561	61
511	752	682	70
528	870	791	79
543	987	904	83
558	1115	1029	86
566	1189	1094	95
574	1266	1161	105
578	1307	1199	108
581	1337	1224	113
620	1786	1664	122
641	2074	1949	125
678.5	2680	2534	146
682	2745	2594	151
685	2800	2649	151
688	2857	2699	158
	Descending Temperature		
684.5	2790	2644	146
683	2763	2619	144
676	2636	2496	140
671	2549	2414	135
666	2464	2329	135
658	2323	2201	132
653.5	2265	2136	129
652	2239	2114	125
639	2045	1922	123
635	1988	1872	116
613	1698	1589	109
588	1511	1312	99
575	1276	1189	87
566.5	1185	1099	86
559	1124	1064	60
547	1020	964	56
518.5	782	734	48
510	744	694	50
480.5	568	522	46
464	486	444	42
452.5	433	396	37
442	389	354	35
433	355	322	33
408	271	254	17
391	223	214	9
333	107	101	6
320.5	90	84	6
275	45	41	4

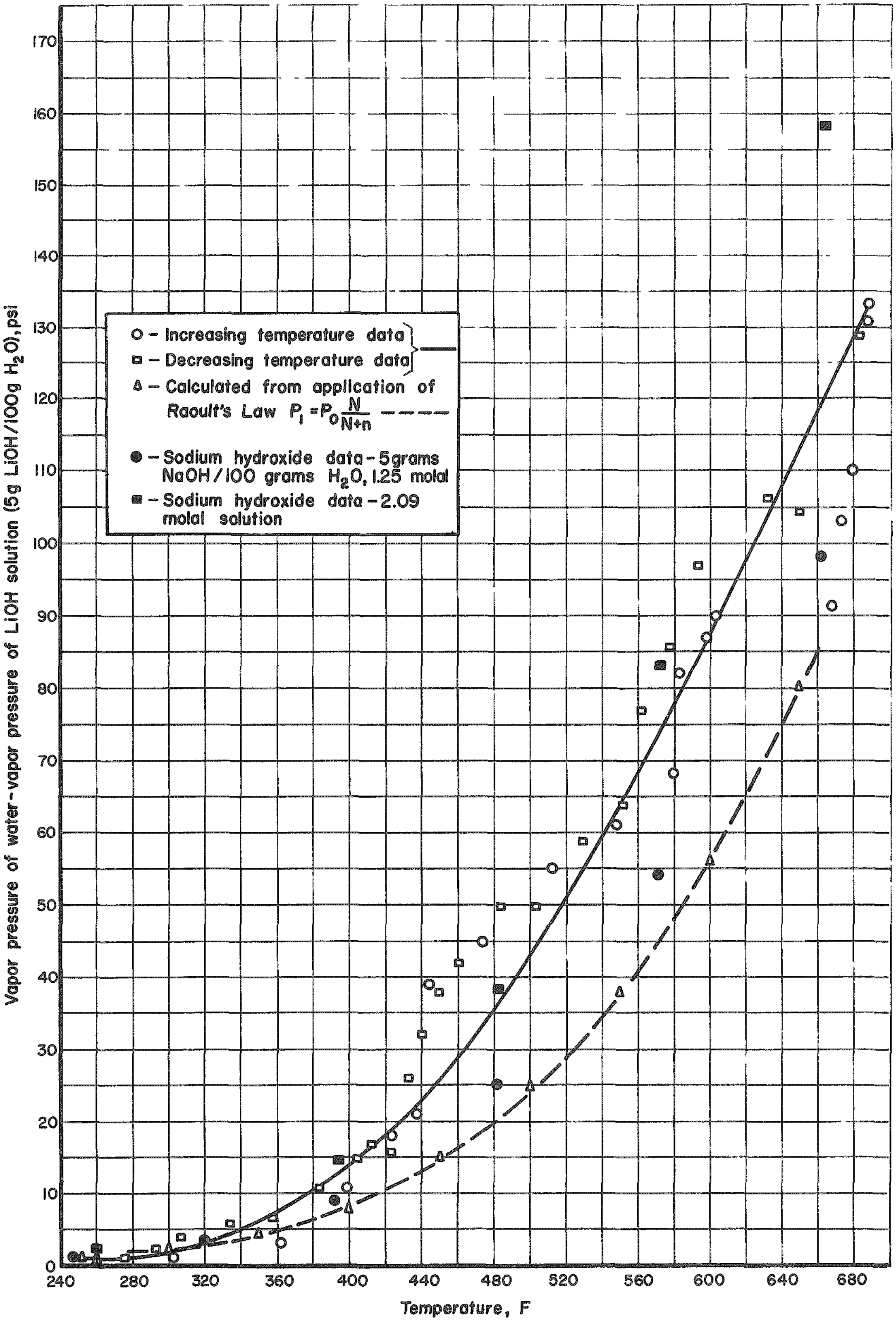

FIGURE 3. DEPRESSION OF THE VAPOR PRESSURE OF WATER BY 5 G LiOH PER 100 G H_2O (2.09 MOLAL OR 4.76 w/o SOLUTION)

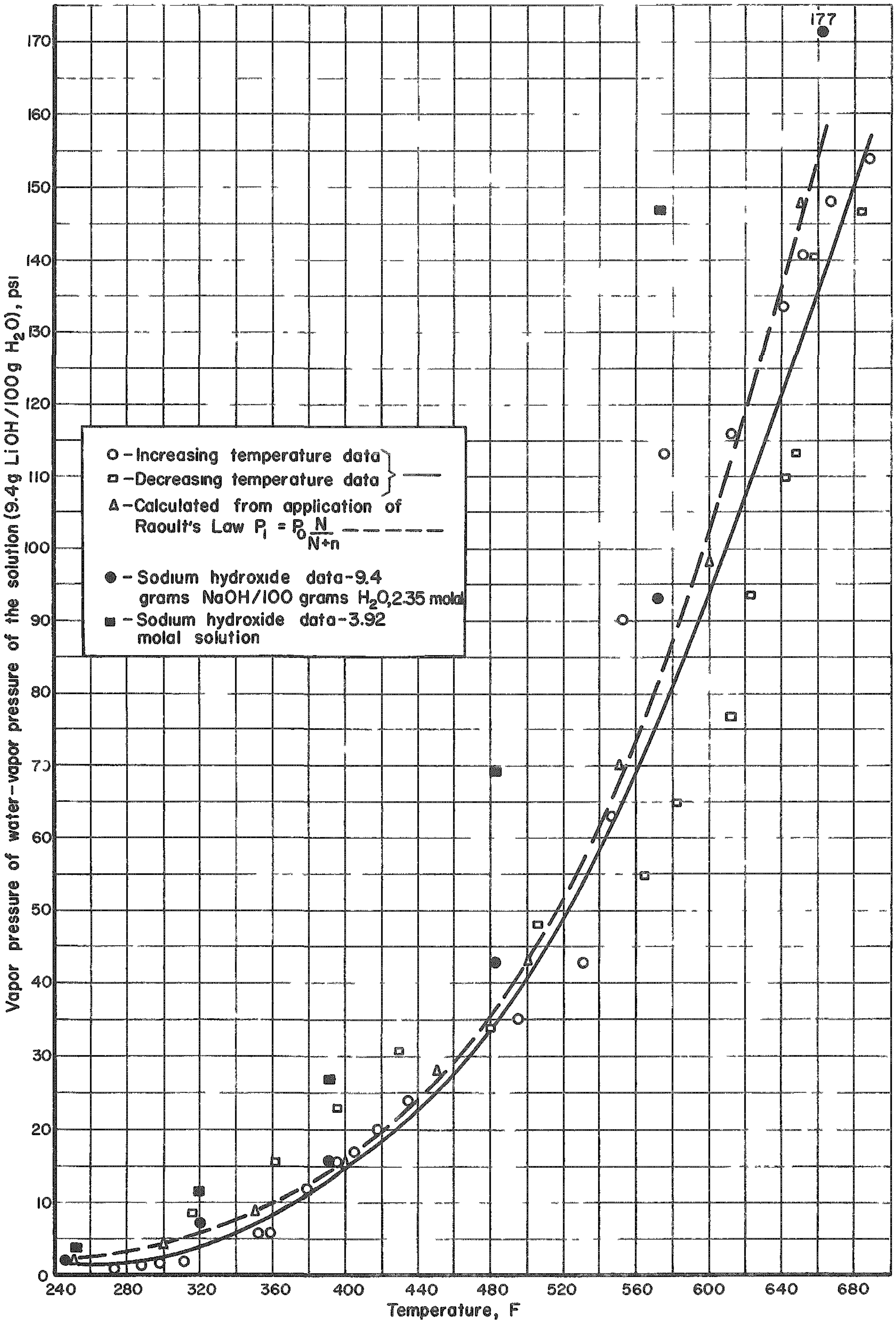

FIGURE 4. DEPRESSION OF THE VAPOR PRESSURE OF WATER BY 9.4 G LiOH PER 100 G H_2O (3.92 MOLAL OR 8.59 w/o SOLUTION)

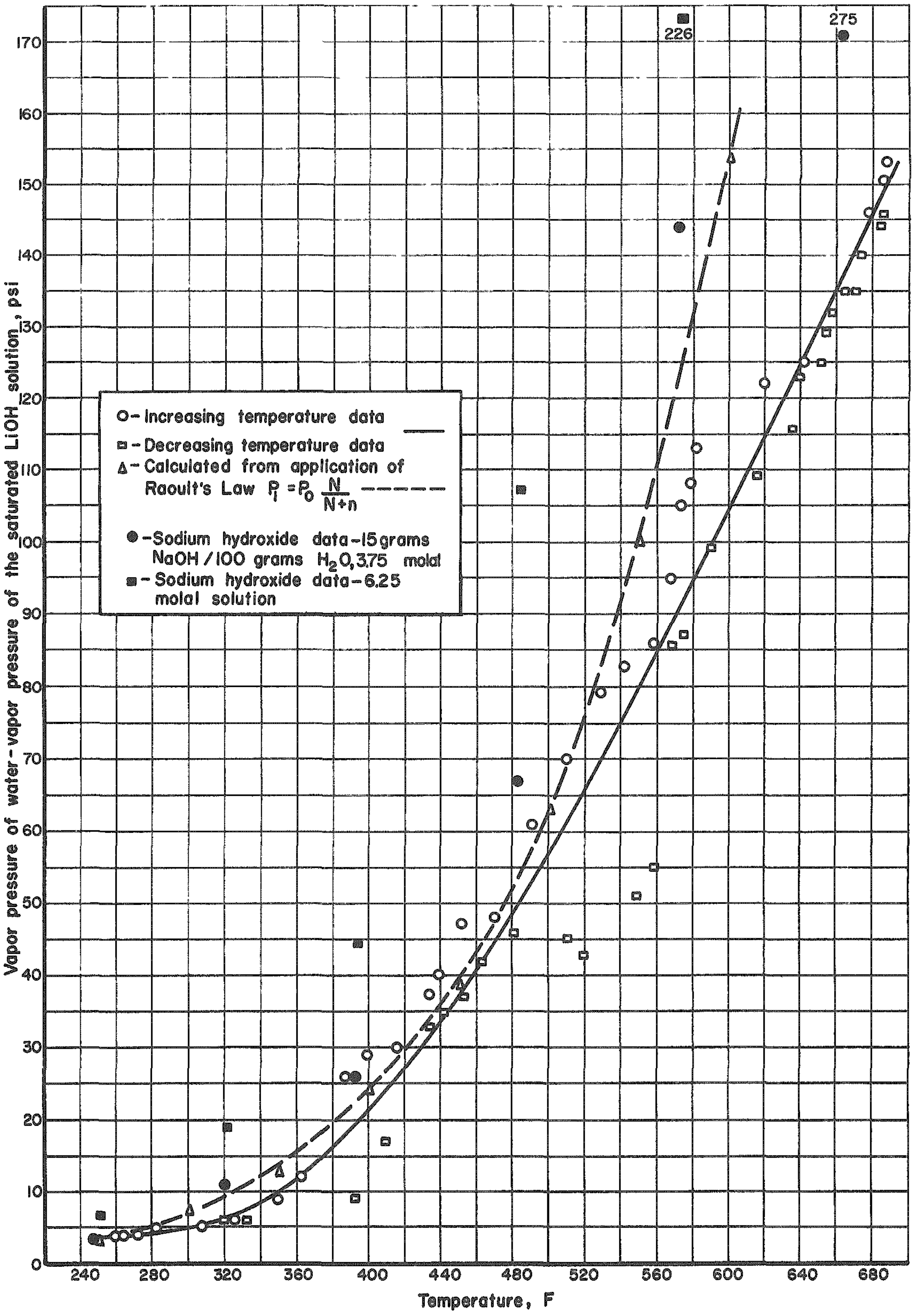

FIGURE 5. DEPRESSION OF THE VAPOR PRESSURE OF WATER BY SATURATION WITH LiOH (APPROXIMATELY 6.25 MOLAL OR 13 w/o SOLUTION

It is interesting that the distortion in the Δp curve (Figure 3) near 480 F occurs at the same temperature as the minimum in the solubility curve (Figure 2). The cause of this effect is not understood.

Figure 3 shows that the deviation from Raoult's law is positive and appreciable for 4.76 w/o lithium hydroxide solutions. Because of the limited scope of the project, attempts have not been made to interpret the data from a thermodynamic standpoint. The data, however, may be amenable to thermodynamic estimations of activity values and dissociation constants.

Tables 4 and 5 and the corresponding figures show that the vapor depression for a 8.59 w/o (3.92 molal) solution and for a saturated solution (about 6.25 molal) of lithium hydroxide amounts to about 154 psi and 158 psi, respectively, at about 689 F. In general, over the entire temperature range studied, the saturated solution showed slightly greater vapor-depression values than did the 8.59 w/o solution.

It can be noted that the values calculated for Raoult's law lie appreciably above the average LiOH curve (Figure 5) for the saturated solution and correspond fairly closely to those for the 8.59 w/o (3.92 molal) solution (Figure 4).

Figure 6 compares graphically the absolute vapor pressure of water with that of the lithium hydroxide solutions for pressures between 1800 and 2500 psia. This is the pressure range of particular interest in reactor engineering.

Undoubtedly, these colligative properties of lithium hydroxide solutions are dependent on the activities of the species as well as on the degree of dissociation and association. Thus, the positive and negative deviations from Raoult's law noted for the various solutions can probably be explained.

It should be noted that the Δp values for sodium hydroxide are much higher than those for the lithium hydroxide solutions. Table 6 summarizes the values for lithium and sodium hydroxide at three concentrations expressed as molality, grams per 100 grams H_2O, and weight per cent. It can be seen that, at 662 F, the vapor depression for a 20 w/o (6.25 molal) sodium hydroxide solution is about three times as great as for a lithium hydroxide solution of the same molal strength. More dilute solutions showed smaller differences. In other words, sodium hydroxide shows a greater lowering of vapor pressure than does lithium hydroxide at the same molal concentrations. In addition, the extent of this difference increases as the concentration of the solution rises. It should be mentioned that the data for sodium hydroxide, as given in the International Critical Tables(2), have been confirmed recently by Kiyana and Kitahara.(6)

DISCUSSION

In addition to establishing the solubility and vapor-pressure relationships for lithium hydroxide between 220 and 650 F, this research has indicated several areas for continuing study:

(1) One is concerned with establishing the phase changes which seem to occur at elevated temperatures.

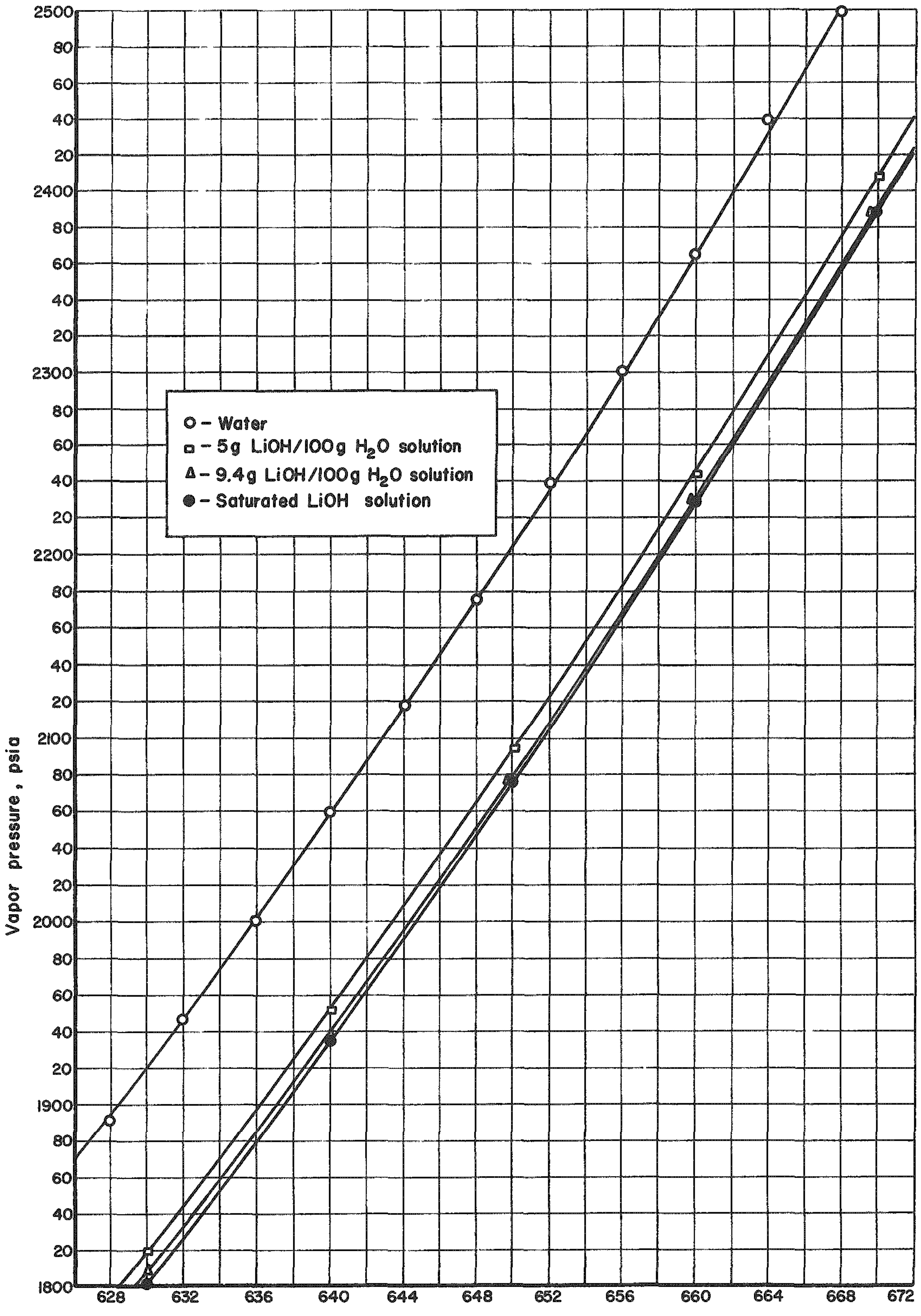

FIGURE 6. PORTIONS OF THE VAPOR-PRESSURE CURVES BETWEEN 1800 AND 2500 PSIA FOR LITHIUM HYDROXIDE SOLUTIONS

TABLE 6. VAPOR-PRESSURE-DEPRESSION DATA FOR LITHIUM AND SODIUM HYDROXIDE SOLUTIONS OF EQUIVALENT CONCENTRACTIONS(a)

	LiOH	NaOH		LiOH	NaOH		LiOH	NaOH	
Concentration									
Grams per 100 Grams H_2O	5	5	8.4	10	10	15.6	15(b)	15	25
Weight Per Cent	4.76	4.76	7.83	8.59	8.59	13.5	13.04	13.04	20.0
Molality	2.09	1.25	2.09	3.92	2.35	3.92	6.25	3.75	6.25
Vapor-Pressure Depression (Δp), psi									
248 F	1	1.2	1.95	1	2.25	4	4	3.6	6.7
320 F	4.5	3.6	6.3	3	7.15	11.5	7	11.2	18.8
392 F	12.5	9	14.5	13	16	27	18	26	44
482 F	35.5	25	38	34	42.5	69	50	67	107
572 F	73	52	83	74	93	147	90	144	226
662 F	120	98	158	140	177	280	137	275	433

(a) NaOH data calculated from vapor-pressure data in the International Critical Tables; LiOH data calculated from smooth curves in Figures 3, 4, and 5 of this report.

(b) Saturated, approximately.

(2) Another is the establishment of the boiling-point-elevation data for solutions of different concentrations.

(3) A third is an investigation of stress-corrosion cracking as related to lithium hydroxide concentration.

Since several observations made during this experimental study may be of interest to others doing similar work, details of these observations are recorded here.

Some lithium hydroxide solution was inadvertently introduced into the annular space between the nickel liner and the stainless steel autoclave wall during some of the solubility runs. Visual examination of the stainless steel thus exposed showed no indication of stress-corrosion cracking.

The solid lithium hydroxide remaining in the autoclave and that which collected in the lines during cooling was slow to redissolve even in hot water. Often, it was necessary to break up the solid mass with a chisel in order to get it out of the autoclave.

The initial run with the dilute solution in the new all-nickel autoclave showed evidence of corrosive attack, especially in the vapor space. A slow increase in the pressure of the system at constant temperature indicated a generation of gas. Inspection of the cold autoclave showed a gray powdery film on the surface of the nickel. This did not happen in succeeding runs. Apparently, a conditioning of the fresh nickel surface was necessary.

In the initial solubility runs, the stem packing of the sampling valve was above the threads and allowed the solution to come in contact with the threads. The valve froze due to corrosion of the threads. Replacement with a valve having the stem packing below the threads prevented further freezing.

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