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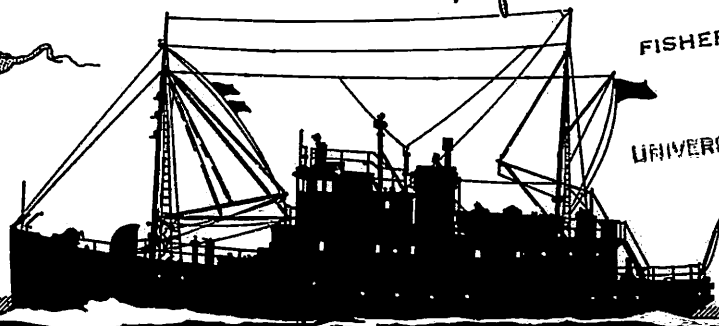
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Technical Report No. 13

## DESALTING OF SEA WATER BY FREEZING PROCESSES

Office of Naval Research  
Contract N8onr-520/III  
Project NR 083 012

Reference 53-1  
November 1953



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DESALTING OF SEA WATER BY FREEZING PROCESSES

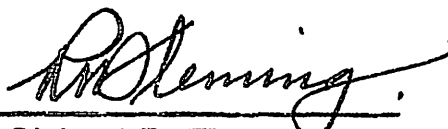
by

Kurt H. Nelson and Thomas G. Thompson

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Office of Naval Research  
Contract N8onr-520/III  
Project NR 083 012

November 1953



Richard H. Fleming  
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## DESALTING OF SEA WATER BY FREEZING PROCESSES

### SUMMARY AND CONCLUSIONS

An experimental laboratory study has been made to determine the extent of the removal of sea salt by freezing processes.

1. Ice formed by freezing sea water under laboratory conditions at different temperatures, from  $-5$  to  $-35^{\circ}$  C. contains from twenty to twenty-five percent of the total salts originally present in sea water.

2. When such ices are allowed to melt in situ at room temperature, marked stratification of waters occurs. The brines at the bottom of the receptacles, representing twenty percent of the total volume, contain sixty percent of the total salts for waters frozen at  $-5^{\circ}$  C. and eighty percent of the total salts when freezing occurred at  $-15$  and  $-15^{\circ}$  C.

3. When the brine is drained from the ice before melting of the ice occurs, the last portions of the water resulting from the melting ice, representing thirty percent of the original volume of sea water, are potable. The total solids of this water are less than that of many municipal water supplies.

4. The yield of potable water may be increased to fifty percent by reprocessing the brackish water from the melting ice. This brackish water represents fifty percent of the original volume of sea water and contains approximately twenty percent of the total salts.

5. The unfrozen brines occupy a volume of twenty percent and contain over eighty percent of the original total salts.

# DESALTING OF SEA WATER BY FREEZING PROCESSES

## INTRODUCTION

The processes occurring during the freezing of sea water differ from those of fresh water. Since the temperature of maximum density of pure water is  $3.98^{\circ}$  C., cooling pure water below this point results in a decrease in the density of the water. When sea salts are added to fresh water, the temperature of maximum density as well as the freezing point are lowered. For an increase of one part per thousand in the salinity, the temperature of maximum density is lowered  $0.215^{\circ}$  C. while the freezing point is decreased only  $0.054^{\circ}$  C. Thus for a water with a chlorinity of 13.67 ‰ or 24.70 ‰ salinity, (1), the freezing point and the temperature of maximum density are identical at  $-1.332^{\circ}$  C. For sea waters of average salinities, the temperatures of maximum densities are below the freezing points. As sea water freezes, fresh water precipitates as crystals of ice containing some occluded salts causing an increase in the density of the liquid phase adjacent to the ice. This denser solution tends to sink and is replaced by water of lesser density. Brine remaining from sea water frozen in the laboratory collects mainly on the bottom of the vessel rather than in the center of the ice cake as in the freezing of fresh or slightly brackish water. The ice formed from sea water has a rather porous nature due to the entrapment of brine.

A search of the literature revealed that data on the distribution of salts in sea ice are rather meager. Malmgren (2) reported

that the salt content of ice produced under natural conditions depends upon the rate of freezing. The concentration of salts decreased with increasing depth below the surface of the ice and also, the salinity of the new ice increased as the air temperatures decreased. Steinbach (3) recently reported results obtained by freezing solutions of artificial sea water containing only sodium chloride.

#### EXPERIMENTAL

Investigations were undertaken in order to obtain quantitative information on the distribution of salts in ice formed by the freezing of sea water, to ascertain the salinity of the waters resulting from the melting of such ice and to examine the nature of any potable waters produced.

In the first series of experiments described below, cylinders of ice, formed by freezing sea water at different air temperatures, were cut into a number of discs. Each disc was allowed to melt and the chlorosity of the water obtained from each disc was determined.

For the second series of experiments, sea water was frozen at different air temperatures and the chlorosity of the water at various depths in the receptacle was determined after the ice had melted in situ.

The sea water in the third series was frozen at different air temperatures and the brine was drained from the ice. As the ice slowly melted, the water was collected in fractions and periodically analyzed.

The sea water used in the experiments was taken near the entrance to Puget Sound and had a chlorinity of 16.60 ‰ (salinity

30.00 ‰). The water was filtered through a sintered glass filter of F porosity before using.

The salt content of the water expressed as grams of chloride ion per liter at 20° C. (chlorosity) was determined by the Mohr method using 0.01 and 0.1 N standard silver nitrate solutions and a few drops of a saturated potassium chromate solution as the indicator.

#### I. Salt Distribution in the Ice

Malmgren's (2) observations on the distribution of salts in sea ice were made on ice formed under natural conditions. As the ice forms in the sea, the resulting denser water sinks and is replaced by more water of the original density. When ice forms in a receptacle of sea water in the laboratory, the water in contact with the ice becomes increasingly concentrated. As it is difficult to duplicate the natural conditions, the distribution of salts in the ice formed under laboratory conditions was investigated by freezing cylinders of ice at different air temperatures.

A paraffined cardboard tube, 6.5 cm. in diameter by 46 cm. high and sealed at the lower end with a rubber stopper, was filled with 1200 ml. of sea water and placed vertically in the freezer. The sides of the tube were insulated with felt to promote freezing from the top surface. When freezing was completed, the tube was removed from the freezer and the brine was drained off by removing the rubber stopper. In all the experiments reported, there was always a minute quantity of brine retained on the surface of the ice. This surface brine was removed and the ice was sliced into discs approximately 1 cm. thick without permitting any melting of the ice. The chlorosity of the

water from each disc was determined after the ice melted. The data are given in Table I and are plotted in Figure 1 for tubes frozen at four different air temperatures.

There was a slight decrease in chloride concentration with depth for the ice cores formed at  $-5^{\circ}$  C. The reverse condition was found to exist for the ice cores frozen at the other temperatures. The effect of the brine retained on the surface of the ice is readily noticeable in the graphs of Figure 1.

As a general statement from the results of this series of experiments, it may be said that twenty to twenty-five percent of the total salts in the initial volume of water are occluded by the ice under laboratory conditions.

Should the ice be permitted to melt, the liquids first forming should contain most of the salts occluded by the ice. This denser solution would gravitate to the brine in the lower portion of the container. To determine the stratification of the waters resulting from the melted ice, the following experiments were devised.

TABLE I

CHLORIDE CONCENTRATION AT VARIOUS DEPTHS IN ICE  
FORMED AT DIFFERENT TEMPERATURES

-5° C.		-15° C.		-25° C.		-35° C.	
Distance Below Surface	Cl gm/l	Distance Below Surface	Cl gm/l	Distance Below Surface	Cl gm/l	Distance Below Surface	Cl gm/l
cm		cm		cm		cm	
1.2	4.67	0.3	15.18	0.9	12.96	0.5	18.25
3.1	4.93	1.4	5.33	2.7	3.36	1.8	3.50
4.5	5.26	3.0	2.52	4.3	3.73	3.1	5.03
6.0	5.29	4.5	3.53	6.0	3.71	4.4	5.26
7.5	4.91	6.0	4.57	7.6	3.86	5.7	5.06
8.9	4.91	7.5	5.00	9.4	4.14	7.0	5.23
10.3	4.80	9.0	5.38	11.2	4.46	8.3	5.32
11.6	4.87	10.5	5.33	12.8	4.55	9.6	5.50
13.0	4.78	12.1	5.75	14.2	4.56	10.9	5.51
14.3	4.89	13.6	5.72	15.8	4.47	12.2	5.81
15.7	4.59	15.2	6.07	17.4	4.54	13.5	5.95
17.1	5.31	16.7	6.85	19.0	5.09	14.9	6.37
18.5	4.47	18.2	6.70	20.6	5.65	16.2	6.47
19.8	4.02	19.7	7.28	22.2	6.13	17.6	6.56
21.1	4.27	21.1	7.03	23.9	6.45	18.9	6.81
22.4	4.12	22.6	7.62	25.5	6.77	20.2	7.08
23.8	4.11	24.1	7.94	27.2	7.38	21.5	7.26
25.1	4.12	25.6	8.49	29.0	9.00	22.9	7.72
26.5	3.80	27.1	9.00	30.7	10.53	24.2	8.22
28.0	4.04	28.6	9.20	32.6	12.53	25.6	8.78
29.5	3.91	30.1	9.03	34.5	16.09	26.9	8.99
30.8	4.68	31.5	10.56	36.4	25.00	28.3	9.56
		33.0	11.50	38.4	48.64	29.7	10.29
		34.5	13.64			31.1	11.20
		35.9	17.97			32.5	13.36
		37.2	19.64			33.9	15.97
						35.3	19.79
						36.7	26.68
						38.5	33.28

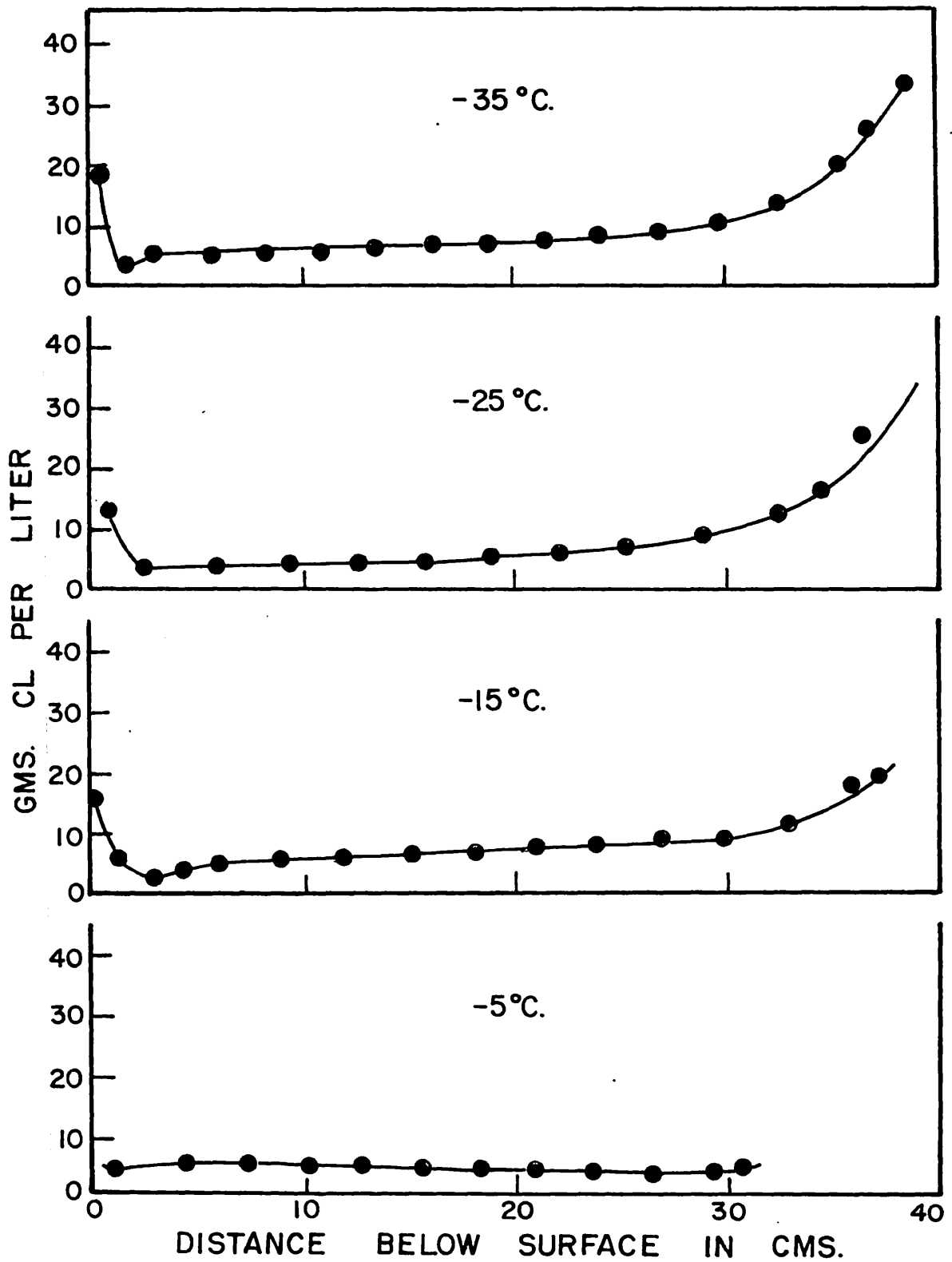


FIGURE 1. Chloride Concentration at Various Depths in Ices Formed at Different Temperatures.

## II. Salt Distribution in Waters After Ice Melted

The receptacles used for freezing the sea water were glass cylinders 20 cm. in diameter, 60 cm. high and had a capacity of over 18 liters.

After samples of 15 liters each were frozen, the cylinders were removed from the freezer and the ice was allowed to melt without draining off the remaining brine. When the melting was completed, samples were pipetted at approximately 6 cm. intervals beginning at the surface of the water. At about 10 cm. from the bottom of the cylinder, samples were taken at 3 cm. intervals.

As convection currents would tend to occur during the melting and freezing processes, and particularly if melting was too rapid, the above experiments were first conducted at  $-15^{\circ}$  C. under different conditions whereby the rate of freezing and/or melting was altered by the use of insulation material around the cylinders. Other than a slight decrease in chloride concentration in the surface layer of the water for the slower melting under insulated conditions, no noticeable difference was found between insulated and uninsulated conditions. The results were generally reproducible to within five to ten percent.

In Table II are data, graphically presented in Figure 2, showing the desalinification produced by freezing samples to temperatures of  $-5$ ,  $-15$ , and  $-45^{\circ}$  C. followed by melting of the ice at room temperature. In these experiments the containers were well insulated to insure slow freezing and melting. There was marked desalting of the water in the upper portions of the cylinders in all cases. The brine remaining at  $-5^{\circ}$  C. had a concentration considerably less than the brines obtained at lower temperatures, but occupied a much greater volume.

TABLE II

CHLORIDE CONCENTRATION OF SAMPLES TAKEN AT  
VARIOUS DEPTHS IN CYLINDERS AFTER MELTING OF ICE

Air Temp. of Refrigerator	$-5^{\circ}$ C.		$-15^{\circ}$ C.		$-45^{\circ}$ C.	
Total Water Above Sampling Point liters	Depth cm	Cl gm/l	Depth cm	Cl gm/l	Depth cm	Cl gm/l
0.4	1.1	0.90	1.3	0.44	1.1	0.52
2.0	6.2	1.32	6.5	1.31	6.2	1.09
4.0	12.5	3.53	12.7	3.25	12.5	2.78
6.0	18.7	6.05	19.0	4.50	18.7	4.92
8.0	24.9	10.65	25.3	7.27	24.9	7.77
10.0	31.3	22.13	31.7	11.84	31.3	11.43
12.0	37.6	37.02	38.1	25.92	37.6	23.76
13.0	40.9	39.93	41.4	46.06	40.9	40.87
14.0	44.2	40.18	44.7	67.25	44.2	69.22
15.0	48.1	40.22	49.0	73.96	48.1	85.42

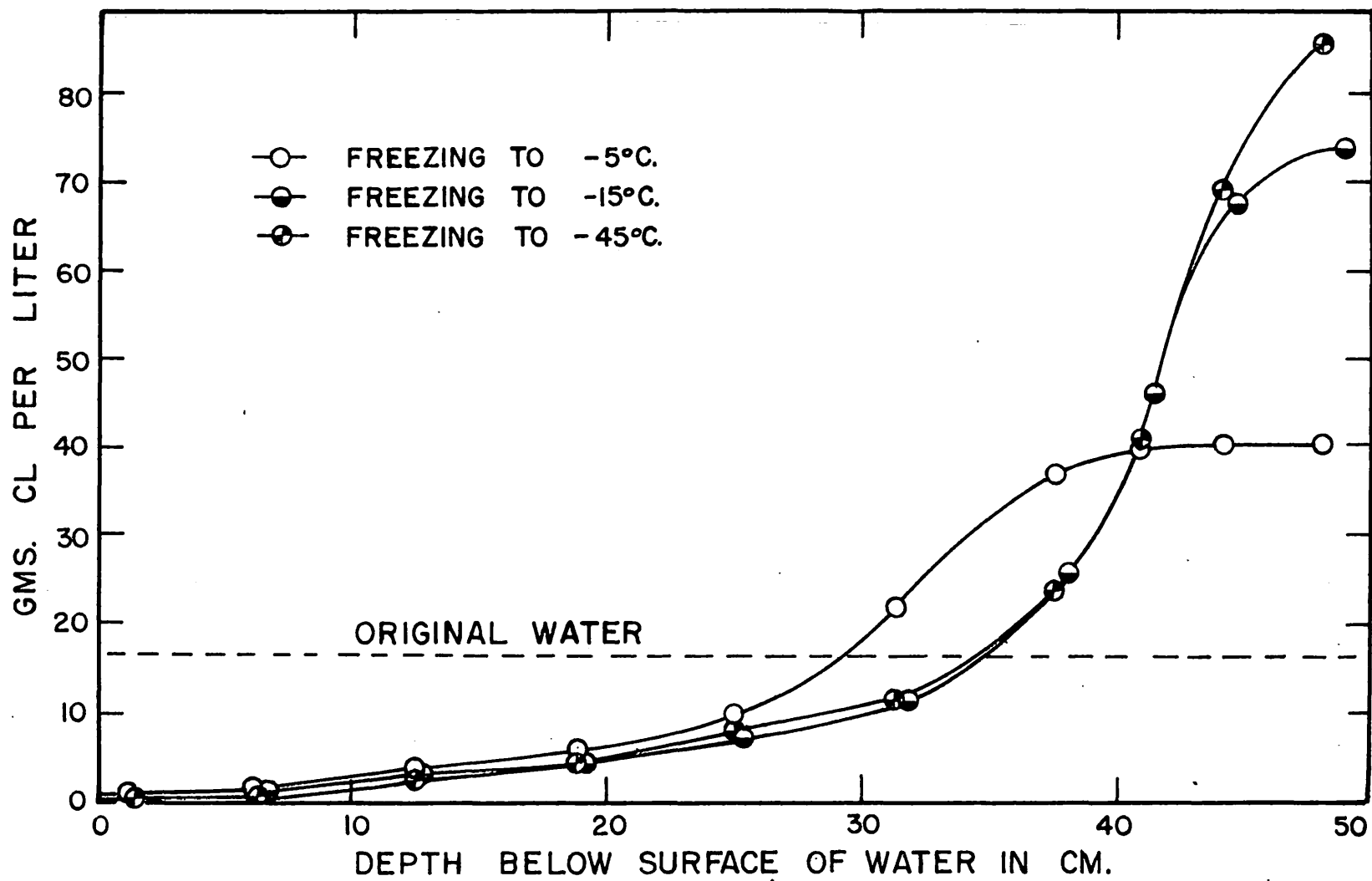


FIGURE 2. Effect of Freezing Sea Water at Various Temperatures.

### III. Removal of Salts from Melting Ice

Although the first experiments indicated that the ice had a high salt content, the first water from the slowly melting ice after removal of the brine would contain a very high percentage of the occluded salts and brine. The first ice to melt would be that immediately adjacent to the interstitial salts and brine retained in the ice. The resulting liquid, gravitating through the ice, would have a high salinity. The remaining ice would then have only exceedingly small fractions of the original salts. Water formed by the later melting of the residual ice would be fresh water. To ascertain the extent of the removal of salts from the ice and the quantities of fresh water formed, the following experiments were conducted. The sea water was frozen to the desired temperature and the residual brine was drained from the ice. The ice was then allowed to melt with the water being removed as it formed.

The apparatus consisted of a special container of Lucite and a Technicon fraction collector of the photoelectric drop counting type. The container was constructed by cementing a conical Lucite bottom to a Lucite pipe 12.5 cm. in diameter and 50 cm. high. A short section of 6 mm. diameter Lucite tubing was cemented in a hole of similar size in the center of the conical bottom. Drainage of the container was through a stopcock fastened to the Lucite tubing by rubber tubing. A Lucite screen of 6 mm. mesh was attached to three Lucite rods and could be removed from the container. The screen and container are shown in Figure 3.

The container, filled with five liters of sea water, was

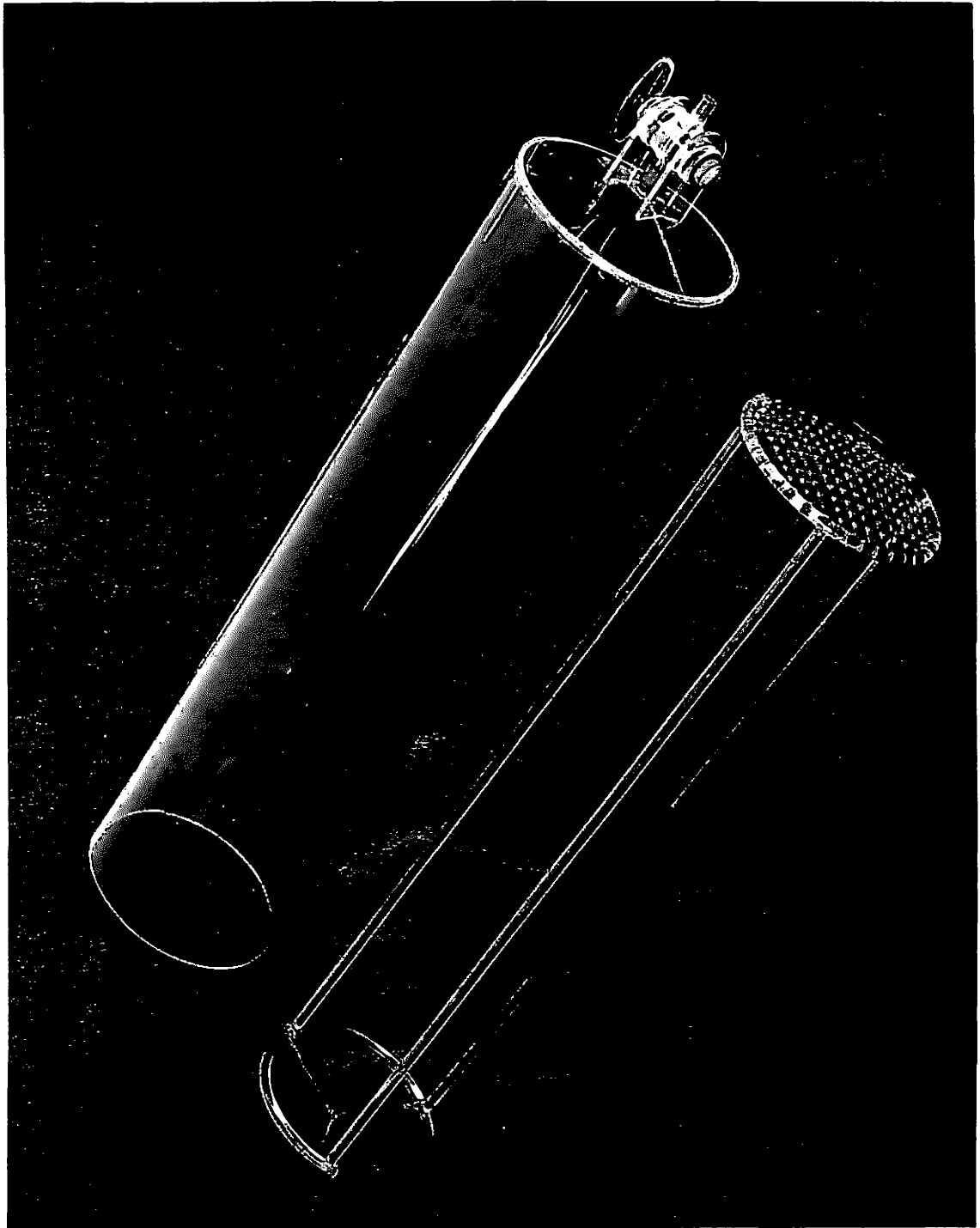


FIGURE 3. Container and Screen Used in the Continuous Removal of Water from Melting Ice.

placed in the freezer. A glass tube, 50 cm. long and sealed at one end, was set in place in the container. This tube later provided an air inlet to facilitate the drainage of brine and water from the melting ice. When thermal equilibrium had been attained, the container was removed from the freezer and placed on the Technicon fraction collector. The sealed end of the long glass tube was broken off and the fraction collector was turned on. After attaching a glass delivery tube, the stopcock was used to regulate the flow of brine from the container. When the brine had drained, the stopcock was fully opened. The volume of brine or water collected in each test tube (about 20 ml.) was controlled by adjusting the fraction collector. The screen supporting the ice was raised sufficiently so that none of the ice would be suspended in the water resulting from its melting.

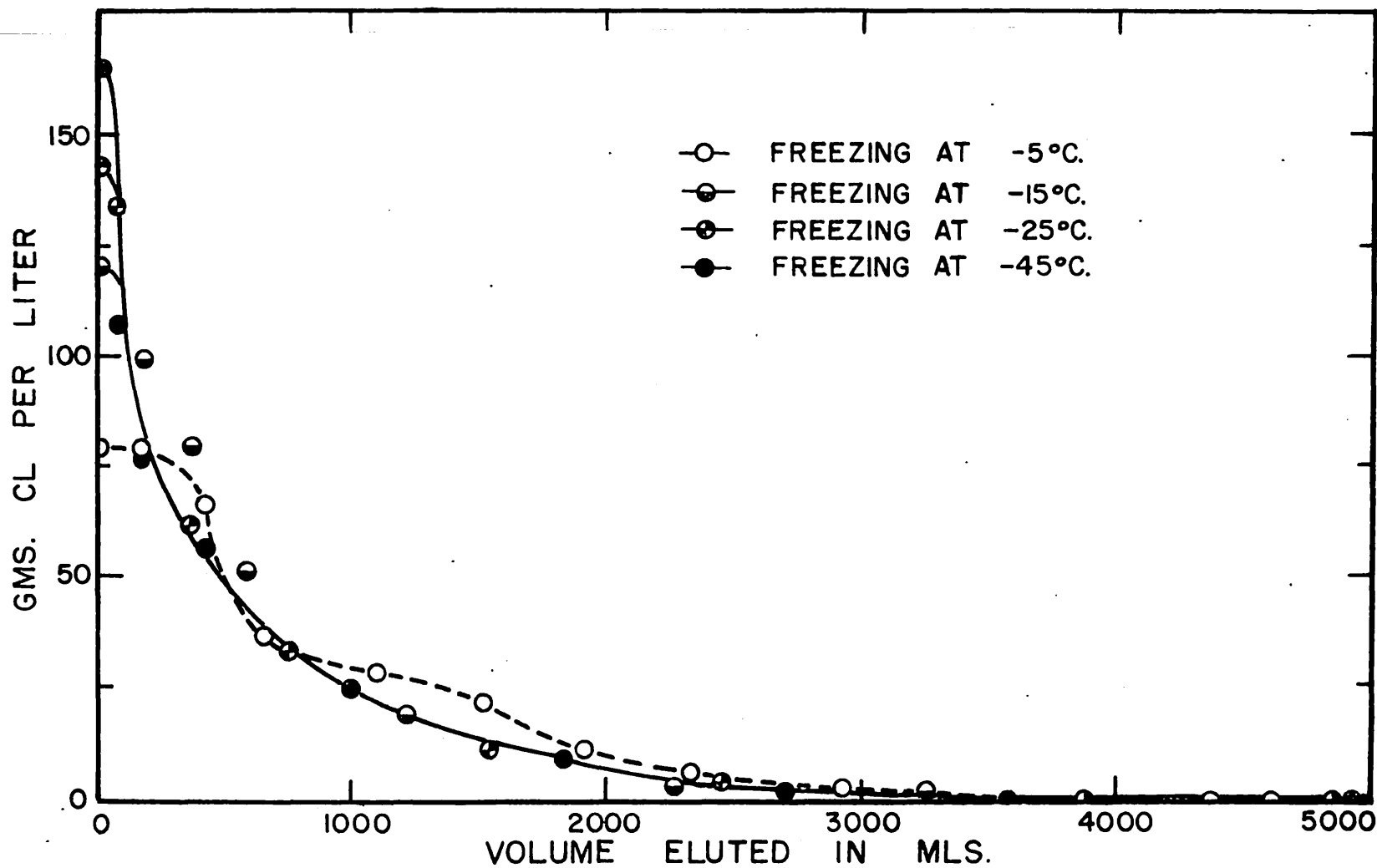
Chlorosity of the water in every tenth test tube was determined. The total volume of liquid collected up to each chloride determination was obtained by summation of the individual measured volumes of water in each test tube. The data are tabulated in Table III and shown graphically in Figure 4.

The water from the later portions of the melting ice had a very low chloride content and contained less than the U. S. Public Health Service maximum of 250 ppm. chloride for potable water. The chloride concentrations in grams per liter, as obtained from graphs of the data in Table III by means of a planimeter, for different fractional volumes of water from the melting ice and the residual brine are given in Table IV. These results represent the concentrations that would exist if the waters of the fractions indicated were

TABLE III

VOLUMES AND CHLORIDE CONCENTRATIONS OF BRINES AND  
WATERS OBTAINED BY MELTING AND ELUTING OF SEA WATER  
SUBJECTED TO FREEZING AT DIFFERENT TEMPERATURES

-5° C.		-15° C.		-25° C.		-45° C.	
Volume Eluted ml	Cl gm/l	Volume Eluted ml	Cl gm/l	Volume Eluted ml	Cl gm/l	Volume Eluted ml	Cl gm/l
21.8	79.49	18.6	120.4	19.0	143.4	21.6	166.0
108.6	79.38	95.8	118.8	91.2	134.4	97.0	107.7
218.7	78.66	197.6	99.70	194.4	79.14	200.0	76.24
437.3	66.48	392.1	79.50	378.6	61.44	425.1	57.09
664.3	36.48	596.4	51.01	563.4	42.73	630.9	45.77
889.8	31.86	794.9	35.52	763.0	32.31	821.8	31.90
1115	28.40	997.9	26.70	955.1	23.86	994.2	25.29
1230	25.24	1233	18.79	1151	18.44	1222	17.46
1527	21.16	1438	14.48	1349	13.51	1422	13.06
1729	14.01	1609	12.03	1547	10.51	1629	10.79
1926	10.49	1818	8.91	1749	8.52	1850	9.13
2131	7.54	2046	4.16	1952	8.09	2066	6.13
2334	5.73	2280	2.75	2156	5.23	2275	4.98
2522	4.08	2516	1.72	2462	3.36	2487	2.84
2734	3.37	2776	1.19	2650	2.46	2710	1.97
2927	2.29	3074	0.74	2857	1.61	2921	1.65
3143	1.73	3264	0.43	3066	0.87	3133	0.81
3345	1.05	3496	0.28	3286	0.48	3352	0.57
3550	0.58	3708	0.17	3493	0.37	3592	0.18
3756	0.26	3938	0.08	3695	0.18	3828	0.11
3958	0.16	4186	0.02	3890	0.10	4054	0.04
4157	0.07	4447	0.01	4085	0.04	4277	0.02
4377	0.03	4633	0.01	4278	0.02	4497	0.006
4585	0.01	4833	0.002	4470	0.01	4716	0.003
4799	0.003	4930	0.002	4661	0.01	4932	0.006
				4849	0.02		



**FIGURE 4.** Chloride Concentrations and Volumes of Brines and Waters Obtained by Melting and Eluting Sea Water Subjected to Freezing at Different Temperatures.

TABLE IV

CHLORIDE CONCENTRATION OF FRACTIONAL VOLUMES OF WATER FROM  
MELTED ICE AND RESIDUAL BRINE BY PLANIMETER DETERMINATION  
AND FROM DATA ILLUSTRATED IN FIGURE 4

Temperature of Freezing	-5° C.	-15° C.	-25° C.	-45° C.
Fractional Volume liters	Cl gm/l	Cl gm/l	Cl gm/l	Cl gm/l
0 to 1.0	55.06	65.40	57.65	59.74
1.0 to 3.5	10.70	6.85	6.44	6.97
1.0 to 5.0	7.14	4.05	4.00	4.20
1.5 to 5.0	4.00	1.99	2.38	2.42
2.0 to 5.0	2.37	0.84	1.31	1.32
3.0 to 5.0	0.44	0.17	0.21	0.23
3.5 to 5.0	0.13	0.07	0.07	0.06

homogeneous. From an examination of these data the following conclusions may be drawn: (1) eighty percent of the total salts originally present in the five liters are confined to a volume of one liter, (2) fifty percent of the water was partially desalted and contained only twenty percent of the salts originally present in the sea water, and (3) thirty percent of the sea water, or 1.5 liters, has been sufficiently desalinified to yield a potable water containing from 70 to 130 ppm. chloride.

The yield of potable water could be increased considerably by reprocessing the partially desalted water. To simulate this partially desalted water, sea water was diluted to a chlorosity of 3.32 grams per liter. This diluted sea water was frozen at  $-15^{\circ}$  C., the resulting ice was melted and analyzed in the same manner as above. The data obtained are given in Table V and are graphically presented in Figure 5.

TABLE V

CHLORIDE CONCENTRATIONS AND VOLUMES OF BRINE  
AND WATER OBTAINED BY MELTING AND ELUTING OF  
PARTIALLY DESALTED WATER REFROZEN AT  $-15^{\circ}$  C.

<u>Volume Eluted ml</u>	<u>Cl gm/l</u>
10.4	100.3
76.4	44.52
179.1	27.08
368.2	12.22
574.6	8.61
788.8	7.49
998.4	4.53
1207	3.51
1427	2.60
1638	2.27
1851	2.33
2062	1.39
2272	1.23
2481	1.03
2688	0.94
2898	0.59
3114	0.42
3328	0.23
3541	0.14
3752	0.08
3963	0.04
4174	0.02
4383	0.01
4591	0.008
4797	0.002

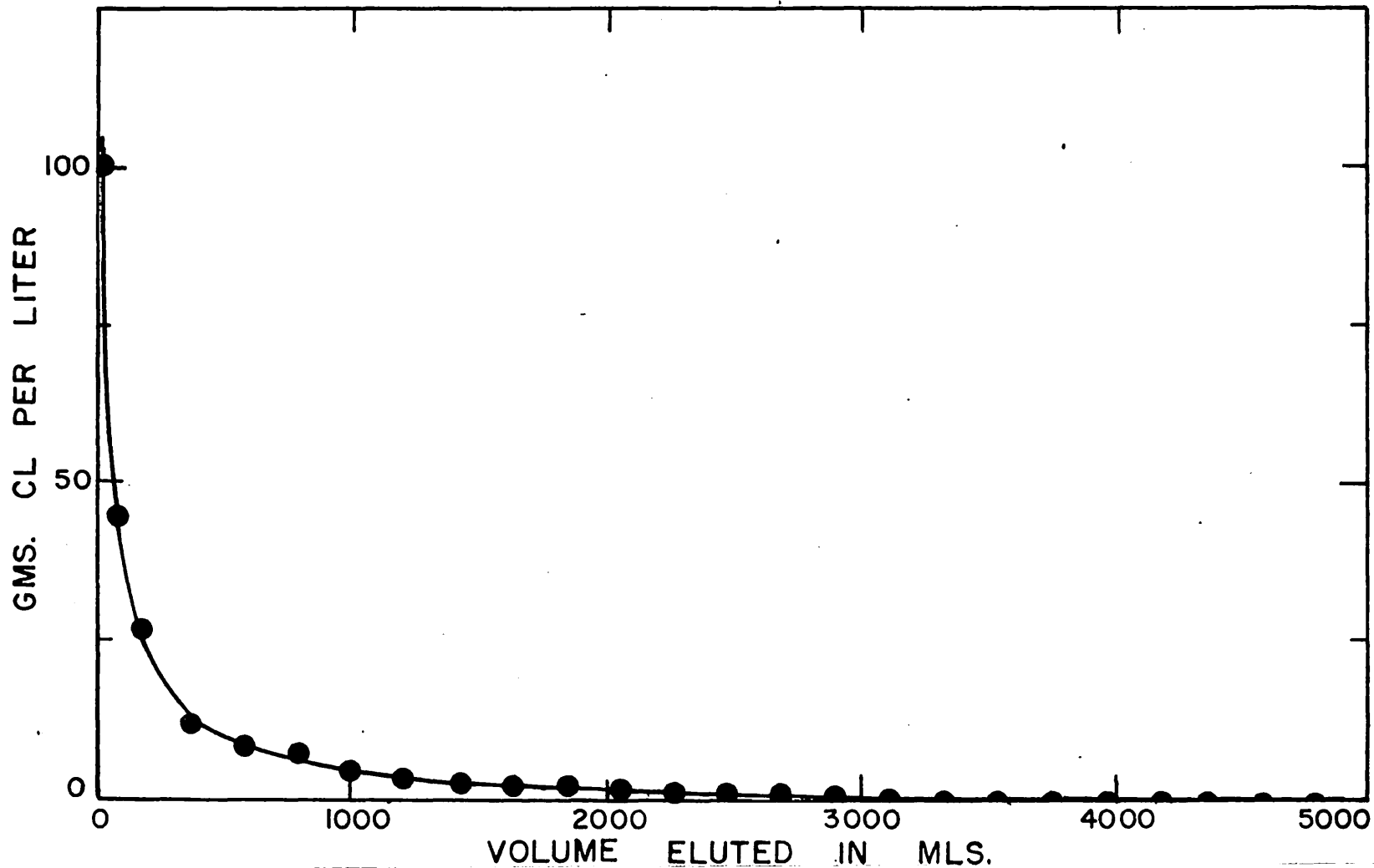


FIGURE 5. Chloride Concentration of Brine and Water Obtained by Reprocessing Partially Desalted Water at  $-15^{\circ}$  C.

## DISCUSSION

Although the salts concentrate in the brine at the bottom of the vessel when sea water is frozen in the laboratory, twenty to twenty-five percent of the salts are retained in the ice either as brine or deposited salts. The concentration of salts in the ice increases slightly with depth below the surface when the ice is formed at air temperatures below  $-5^{\circ}$  C. The expected increase in salt content for the porous ice in contact with the bottom brine is found for all temperatures. In addition, a small amount of brine appears on the surface of the ice at lower temperatures.

When the ice melts in contact with the brine, the resulting water shows decided stratification. Although the bulk of the salts are in the brine at the bottom, the upper waters contain a sizeable amount of the total salts due to convection currents and mixing during melting of the ice. The surface water is relatively fresh since it is formed from the last of the salt free ice.

If the brine is removed from the ice before it melts, the first water drained from the melting ice possesses a high salinity due to the natural washing out of occluded salts and brine. The concentration of salts in the outflowing water constantly diminishes until the last thirty percent of the water is potable. The yield of fresh water can be increased to at least 50 percent by re-freezing the partially desalted water.

The fresh water obtained by the melting of ice formed by freezing sea water in the laboratory will not be entirely free of

salts as would be the case if the water had been procured by distillation. However, if a comparison is made of the calculated energy requirements for getting equal quantities of fresh water by distillation and by freezing from the same amounts of sea water of similar salinities, it will be found that a very considerable energy difference exists. While no data are available for the heat of vaporization or the specific heat of sea water at higher temperatures, it may be assumed that these are about the same as for pure water. Thus, according to calculations, approximately six times as much energy is required for distilling sea water as is needed to freeze it. This difference in the required energy will vary slightly depending upon the original temperature of the sea water being processed and the amount of fresh water to be recovered from a given quantity of sea water.

The use of sea ice as a source of fresh water has been known to inhabitants of the Arctic regions since prehistoric times. Vessels plying Arctic waters in the summer months have often watered ship from ponds formed on ice floes by the melting ice.

An annotated bibliography (1) on producing fresh water from saline water has been compiled. Methods for the precipitation of the dissolved salts in sea water were developed during World War II in order to secure small quantities of potable water during emergencies (5, 6). The use of solar energy as a means of producing fresh water has been investigated recently (7). A historical summary of the development of distillation, the usual method for the recovery of fresh water from sea water, has been published by Hampel (8). Howe (9) has presented a survey of several methods for refining sea water, in-

cluding compression distillation, multiple-effect distillation, solar distillation, the electrolytic process, ion exchange, freezing, and the thermal-difference power plant. Cost data, fuel requirements and other factors for the various methods of sea water reclamation except by freezing has been given by Aultman (10). The cost estimates published by different writers vary depending upon the fuel costs and unit efficiencies quoted. The cost of obtaining fresh water by freezing sea water has been estimated to compare favorably with compression distillation which has the best economy of any scheme for distillation. Various technical problems exist in connection with freezing including separation of salts from the ice, design of heat transfer units for application during the melting of the ice, and design of the counterpart of the multiple-effect still.

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