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REPORT ON THE ABSORPTION OF CHLORINE BY LITHIUM HYDROXIDE

Clyde W. Balch

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NAVY DEPARTMENT

Report

on

The Absorption of Chlorine by

Lithium Hydroxide

NAVAL RESEARCH LABORATORY ANACOSTIA STATION WASHINGTON, D. C.

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ABSTRACT

The results of this report have shown that lithium hydroxide, the present carbon dioxide absorber on submarines, can be used effectively for removing chlorine gas resulting from salting of the storage batteries.

The simplest method for removing the chlorine, although not the most effective, is to follow the instructions issued by the Bureau of Construction and Repair for removing carbon dioxide with the above material. Slight modifications are necessary; the most important is that moisture in some form must be present. This is true of its use by any method since it is the monohydrate form that is the most reactive. While this method will remove the greater portion of the chlorine, it will not in any reasonable time reduce the concentration to a point where it is safe for personnel. This is about 1 part per million.

In order to reduce the concentration to such a value, all of the contaminated air must make direct contact with the absorbing material in some manner. This can be accomplished by passing the contaminated air through some form of vessel containing the material. Very efficient absorption can be obtained in this manner.

It is estimated that about 75 pounds of this material would absorb the chlorine that could be evolved if the air space above the electrolyte in each of the 120 Exide UL-53 type cells in a submarine such as the S-21 were completely flooded with salt water.



INTRODUCTION

(a) Authorization

1. This problem was authorized by Bureau of Engineering letter S62-4/L5 (6-16-Ds) of 20 June 1939.

(b) Statement of Problem

2. The purpose of this study was to determine the possibility and the methods of using anhydrous lithium hydroxide, the present carbon dioxide absorbent for submarine air purification, as a means of removing chlorine during an emergency.

(c) Theoretical Considerations and Known Facts Bearing on the Problem

3. When salt water enters a lead-acid storage battery chlorine gas is evolved in an amount depending upon the following factors: the quantity entering, the concentration of the acid in the electrolyte, the extent of mixing, the condition of the charge, and the temperature. If a battery in this condition is charged, chlorine would be generated as long as any of the salt remains in the electrolyte.

4. At the present time the mechanism of these reactions is not completely known. It is hoped that a more complete picture can be given in a future report. Briefly the reactions which are believed to take place are as follows. The sodium chloride which enters the cell reacts with sulfuric acid with the formation of hydrochloric acid as shown by the equation -

NaCl +
$$H_2SO_4 \longrightarrow HCl + NaHSO_4$$

The hydrochloric acid formed will then react with the active material of the positive plate, lead dioxide, in the presence of sulfuric acid to form free chlorine and lead sulfate according to the following equation -

2 HCl + PbO₂ + $H_2SO_4 \rightarrow PbSO_4 + Cl_2 + 2H_2O$

This free chlorine will escape to the atmosphere, or if it comes in contact with the active material of the negative plate, spongy lead, in the presence of sulfuric acid, will react to form lead sulfate and hydrochloric acid, as follows:

$$Cl_2 + Pb + H_2SO_4 \rightarrow PbSO_4 + 2HCL$$

Thus, unless the chlorine liberated escapes to the atmosphere, this cycle of reactions will continue until the battery is completely discharged. When such a battery is charged, chlorine is liberated at the positive plate as long as there are any chloride ions remaining in the electrolyte. During this operation some of the liberated chlorine may react with the negative plate as described above. If this happens the above cycle will be repeated and therefore require a longer time to eliminate all of the chloride.

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5. In order to obtain some idea as to the amount of chlorine which could result from the worst conditions of salting, let us assume that this would be the case if the air space above the electrolyte is completely filled with salt water, If all of the chlorine in this amount of salt water was liberated in a submarine such as the S-21 with 120 cells of the Exide UL-53 type, the resulting concentration would be about 3 per cent at 1 atmosphere pressure and 80°F. For each 100 cubic inches of a 4 per cent salt solution there is available a maximum of about 0.09 pound of chlorine. For 120 of the above cells this would give a total of about 110 pounds or 620 cubic feet of chlorine at 1 atmosphere pressure and 80°F.

6. The above calculations are for the worst possible conditions as well as for the maximum amount of chlorine that could be evolved if the battery is completely flooded. The actual rate and amount of chlorine evolved under such conditions are not known, but are expected to be made the object of a report in the near future. This study would determine the rate at which the chlorine is evolved, as well as the amount, when the battery is completely filled with salt water in a completely charged condition and at various rates of charging and discharging.

7. It is evident that chlorine would be evolved in relatively large amounts in very dangerous proportions. The following table which was taken from reference (1) gives the only available data on the physiological effects of chlorine:

Rapidly fatal for short exposure. . . . 1000 ppm

It can be seen from these data that regardless of the method to be used in removing chlorine during an emergency, it must be capable of reducing the concentration to about 1 part per million. This means, of course, that practically all of the contaminated air has to come into direct contact with the absorbing material.

8. The possibility of using lithium hydroxide for the removal of "acid" gas was suggested in reference (2) in addition to its use as a carbon dioxide absorbent in submarine air purification. The reaction between chlorine and lithium hydroxide should go to completion providing

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contact can be made between them. The reaction is as follows:

 $2\text{LiOH} + \text{Cl}_2 \longrightarrow \text{LiCl} + \text{LiClO} + \text{H}_20$

The lithium hypochlorite under certain conditions decomposes to form the chloride and oxygen.

These reactions are characteristic of those between any of the alkalies and the halogens. Thus any alkali could be used for the removal of chlorine if direct physical contact could be made between the two.

9. Since anhydrous lithium hydroxide is already available aboard the submarine it would be the most logical first choice as an absorbent for chlorine. Since the anhydrous form tends to dust in an air stream and thereby causes an irritation in the nose and throat, silicated and hydrated lithium hydroxide were also used in this study. The silicated material prepared, as will be described later, reduces the dusting and acts as a binder and holds the material together. The hydrated material does not dust since it is slightly hydroscopic. This latter form was also used because preliminary tests indicated that moisture in some form or other was required for efficient absorption.

10. Theoretically, it requires 0.676 gram of anhydrous lithium hydroxide to absorb 1.0 gram of chlorine. It would therefore require about 0.28 gram of the anhydrous material to absorb the chlorine that could be liberated from 1 cubic inch of sea water on the basis of a 4 per cent sodium chloride solution. Since the surface of any absorbent is limited both by the compounds formed in the reaction and the size of the particles, there is necessarily a considerable portion of the material which is not available for the reaction and therefore its usefulness will be considerably below that predicted from the theory. Nevertheless, since it is highly improbable that all of the chlorine in the entering salt water would be liberated, we can make an approximate calculation as to the amount of lithium hydroxide that would be required to absorb the chlorine that would be liberated from 120 UL-53 type cells if they were completely flooded by assuming that complete reaction takes place in both cases. Under such conditions it would take about 75 pounds of lithium hydroxide, or about 2/3 of a pound of lithium hydroxide for each pound of chlorine.

EXPERIMENTAL PROCEDURE

(a) Material Used

11. Anhydrous, monohydrate and silicated lithium hydroxide were used in these experiments. In addition, two runs were made with lithium carbonate. The anhydrous and the monohydrate form were obtained from the Maywood Chemical Works. The silicated material was prepared by mixing 2.1 cc of 0.5 per cent sodium silicate solution with each 10 grams of the lithium hydroxide monohydrate used and then drying in an oven either at 65°C. or 100°C. to constant weight.



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(b) Description of Experiments

12. Three methods of absorbing chlorine by lithium hydroxide were studied, the first in which a chlorine in air mixture was continuously passed through a vessel with the absorbent at the bottom; second, in which a 0.5 per cent mixture of chlorine in air was introduced into a large box with the absorbent placed at the bottom and the concentration of the chlorine determined at various intervals of time; and third, in which a chlorine in air mixture was passed through the absorbent contained in glass tubes.

(1) First Method

13. The first method was used in order to determine the possibility of absorbing chlorine which is being continuously formed by placing the lithium hydroxide at the bottom of the compartment. Since chlorine gas is about three times as heavy as air, the best position for the absorbent would be at or near the bottom.

14. These runs were carried out by passing 1.6 per cent chlorine in air mixture through a desiccator with the absorbent placed on a glass plate at the bottom. The desiccator was about 12 inches in diameter, 12 inches high and had a volume of about 8 liters. The chlorine was generated electrically from a solution of zinc chloride using a carbon rod anode and a mercury cathode. The air with which this chlorine was mixed was either saturated with water vapor or dried as required. The chlorine in air mixture was released through a funnel about midway in the desiccator, while the exit mixture was removed at the top. The chlorine which was not absorbed by the material was determined by passing this exit gas through a solution of potassium iodide and titrating the liberated iodine.

(2) Second Method

15. The second method was carried out in order to determine the rate at which chlorine could be absorbed and to what extent by placing the absorbent in the bottom of a closed compartment. This was accomplished by placing the lithium hydroxide in shallow pans on the bottom of a rectangular box of about 100 liters capacity $(32 \times 44 \times 72 \text{ cm})$ and then introducing enough chlorine from a cylinder to bring the concentration up to about 0.5 per cent. Samples were removed at various time intervals and at different heights by means of a glass tube whose position could be raised or lowered. This enabled the concentration of chlorine to be followed at various heights during a run.

(3) Third Method

16. The third method was studied since the results of the first two indicated that while the concentration of chlorine could be reduced at a fairly high rate by both, it was impossible to reduce the concentration to a point where it was safe for personnel. Thus, in order to reduce the concentration of chlorine to where it is safe for personnel, all of the contaminated air must come into direct contact with the absorbing material. The conditions under which this could be done most efficiently were deter-

mined by passing various percentages of chlorine in air through absorption tubes containing the different forms of lithium hydroxide. The air with which the chlorine was mixed could either be dried or saturated with water vapor. The exit gas was passed through a solution containing potassium iodide and starch in order to determine the time at which chlorine was first passed by the absorbent. In one run after the break occurred, the exit gas was analyzed at various time intervals in the same way as used in the first method. The absorption cells were about 1.7 centimeters in diameter and were usually filled to a depth of about 4 centimeters with the absorbent.

DISCUSSION OF DATA AND RESULTS

(a) First Method

17. The first run was made in order to obtain some idea as to what could be expected of lithium hydroxide as an absorbent for chlorine gas. The results are shown by the curve of run number 1, Plate 1. The percentage of chlorine in the original mixture that is not absorbed is plotted against time.

18. The type of material, the amount and the number of equivalents of alkali for each run are given in the table below. A flow of 425 cc of a 1.6 per cent chlorine in air mixture was used in all runs except number 1. In this case the per cent of chlorine was 1.5. All runs except number 9 were made with the gas mixture saturated with water vapor, in which case the gas was dried.

Bun No	Material Used	Wt. in Grams	No. of Equivalents of Alkali
uuii no.	THE OUT THE OPON	and the second distance of the second distance of	weitiget weitige general weitige general weitige general weitige general weitige general weitige general weitig
1	Lithium hydroxide, monohydrate	30.0	0.71
3	Lithium hydroxide, monohydrate	4.2	0.10
5	Lithium hydroxide, monohydrate	9.0.	0,21
4	Lithium hydroxide, monohydrate	7.0	0.17
9	Lithium hydroxide, anhydrous, dry air	4.0	0.17
10	Lithium hydroxide, anhydrous, wet air	4.0	0.17
6	Lithium carbonate, anhydrous	11.0	0.30
7	Lithium carbonate, anhydrous,	11.0	0.30

19. Runs number 3 and 5 were made in addition to number 1 to show how the amount of material used effects the rate of absorption of chlorine. For comparison the results of all three runs are shown on Plate 1.



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20. Runs number 4, 9 and 10 can be used to compare the absorption ability of the anhydrous and hydrated material. In addition, the effect of a wet and dry gas on the absorption of chlorine by the anhydrous material can be shown by the results of runs number 9 and 10. The results of these three runs are given by Flate 2. The same number of equivalents of lithium hydroxide were used in each of these runs.

21. Inasmuch as the product of the reaction between carbon dioxide and lithium hydroxide is an alkali and should be capable of absorbing chlorine, lithium carbonate was used in runs number 6 and 7. In run number 6 the anhydrous material was used and resulted in very little absorption. In run number 7, 10 cc of water was added to the material used in run number 6 and the passage of chlorine continued. The results are shown by Plate 3 and as a comparison the results of run number 5 are also plotted. The number of equivalents of alkali used in run number 5 was about 2/3 of that used in number 6 and 7.

22. The results of the various runs show that lithium hydroxide can be used to remove a considerable portion of the chlorine that is being continuously formed by placing the absorbent at or near the bottom of the compartment, provided it is not necessary to reduce the concentration below about 1000 parts per million. The results also indicate that water must be present in some form in order to obtain the most efficient absorption and that hydrated lithium hydroxide is more efficient than the anhydrous material. Lithium carbonate will absorb chlorine when wet but not to the extent of an equivalent amount of lithium hydroxide.

(b) Second Method

The conditions under which these runs were made are given 23. below. In all runs enough chlorine was added at the top of the box to bring the concentration up to 0.5 per cent, with the exception of run number 9, in which case the chlorine was added at the bottom. All samples for analysis were taken 1/2-inch above the bottom of the box. In run number 9 samples were also taken from a point 12 inches above the bottom. At the start of each run the box was filled with air from the room. In run number 5 water was added to the anhydrous material which was then mixed into a paste. In runs number 6, 7, 8 and 9 water was placed on the bottom of the box in order to make sure that the gas mixture was saturated with water vapor. These runs were carried out at room temperature, which averaged 77°F. In runs number 7 and 8, successive additions of chlorine were made after the concentration had been reduced to a fairly constant level. In each addition the concentration was brought back up to 0.5 per cent. The table gives the weight and the type of material used in each run.

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Run No.	Weight in Grams	Number of Equivalents	Form of Lithium Hydroxide Used
3	20	0,48	Monohydrate
4	4	0.095	Monohydrate
5	10	0.42	Mixture of anhydrous and 7.5 cc water
6	10	0.42	Anhydrous
7	10	0.42	Anhydrous
8	4	0.17	Anhydrous
9	4	0.17	Anhydrous

24. The results of these runs are given by Plates 4 to 10 with the number on each curve indicating the number of the run. In all curves the concentration of chlorine was plotted against time.

25. Plate 4 gives the results of runs number 3 and 4 and shows the effect which the amount of material has on the rate of absorption of chlorine by the hydrated lithium hydroxide.

26. Plate 5 compares the results of runs number 3, 5 and 6 in order to show the relationship between the absorption ability of the hydrated and anhydrous material in which the latter was wetted by two different methods. Approximately equivalent amounts of lithium hydroxide were used in each of these runs.

27. Plate 6 gives the results of run number 6 and part A of run number 8 and illustrates the effect which the amount of material has on the rate of absorption of chlorine by the anhydrous material.

28. Plates 7 and 8 give the results of runs number 7 and 8 respectively. These curves show the effect of successive additions of chlorine to the same batch of material after former additions of chlorine have been reduced to a low and fairly constant value.

29. Plate 9 shows the effect of introducing the chlorine at the top and at the bottom of the box by plotting the results of part A of run number 8 and part A of run number 9, the other conditions of these two runs being the same.

30. The two curves of Plate 10 show how the concentration of chlorine changes at two different heights above the bottom of the box, In curve A the samples were taken as in the previous runs, 1/2-inch above the bottom, and in curve B the samples were taken 12 inches above the bottom.

31. A blank run was made to determine the distribution of chlorine in the box after having been introduced at the top. The usual amount of chlorine was added and at the end of 6 minutes the concentration of chlorine 1/2-inch from the bottom was 1.29 per cent.

32. The results of the above runs indicate that moisture in some form or other must be present in order for lithium hydroxide to be an efficient absorber for chlorine. This may be either in the form of the hydrate or water vapor in the case of anhydrous material. The anhydrous form under these conditions appears to be slightly more efficient than the hydrated material.

33. The effect of the amount of material was as ordinarily would be predicted in the case of both the hydrated and anhydrous material.

34. Just as in the case of the first method, the chlorine cannot be completely removed or at least lowered to a few parts per million in any practical time limit by this method. The results indicate that about the best that can be expected under the conditions used is to reduce the chlorine to around 100 parts per million in one to two hours. This of course is considerably above a safe limit.

35. While lithium hydroxide under these conditions absorbs chlorine at a very high rate and with a good efficiency, it must not be expected to remove all the chlorine since direct contact must be made between the chlorine and the material in order for the reaction to take place. Therefore, in order to reduce the chlorine concentration to a few parts per million, all of the contaminated air must come to direct contact with the absorbent.

(c) Third Method

36. The efficiency of the absorption of chlorine by the various forms of lithium hydroxide by this method was determined by placing the material in an absorption tube and passing 410 cc of various concentrations of chlorine through it per minute. The gas was saturated with water vapor in each run.

37. The data obtained from these runs are given in the table below. The time of break is considered to be the time at which the first trace of chlorine is passed by the absorbent.

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	Number of cc of Water Added																				2.9	None	12	51	11	11	3.7	3.0	3.7	3*6	8*17	1.5	2.5	2.5	2.5
	Temperature of Drying oc.																					100	100	100	100	100	65	100	100	100	100	100	100	65	65
	Per Cent of Chlorine in Air Used	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	0*42	0.42	0.42	0*42	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	1.46	2.46	1.46	1.46	1.46	0.42	0.42	0*42	0.42
	Time of Break in Minutes	84	63	69	96	55	19	59	69	48	248	370	163	299	12	10	16	12	32	11	12	29	ET	17	16	69	40	더	ŝ	30	12	241	60	304	41.5
Per Cent of	Theoretical Amount Absorbed	30.4	25.2	27.8	24.5	25.6	29.1	30.2	30.3	27.0	23.2	31.2	16.5	29.5	4.4	3,8	5.3	4.1	5.1	4.6	3.7	10.5	5.3	9.2	2*2	8.4	10*3	10.1	1.05	1.7	3*1	12.4	6*4	15.1	21.4
	Weight in Grams	2.75	5.60	5.72	8.20	4.58	4.37	4.08	4.72	4.17	6.50	7.27	5.98	61.9	3.25	3.09	3.56	3*54	4.63	3*34	3.89	2.75	3.19	2.74	3.15	10,38	4.87	6.07	6.25	4.89	4.84	6.78	6.71	7.02	2.00
Form of	Li thium Hydroxide Used	onohydrate		-	u	11	11	11	11	tt.	11	11	11	11	Anhydrous	II.	11	11	11		11	Silicated	11	11	=	11	Ħ	II	11	11	H	ĝer Ban	11	11	11
	Run No.	T	~	en	4	5	9	4	8	6	IO	TT	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34

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38. The results of runs number 1 to 13 show the efficiency of absorption of chlorine by the hydrated material. The effect of chlorine concentration in the gas can be shown by comparing the average of the efficiencies in the first nine runs with that of the next four runs; they are 28 and 25.6 per cent, respectively.

39. Runs number 14 to 20, in which the anhydrous form was used, show an average efficiency of 4.4 per cent. The addition of 2.9 cc of water to run number 20 apparently had no appreciable effect. In runs number 21 to 25, in which the silicated material was used with the addition of water, fairly consistent but low results were obtained with an average efficiency of about 8.1 per cent.

40. In runs number 26 to 34 the silicated material was dried at two different temperatures and then various amounts of water added. The results were quite inconsistent, but it can be concluded in a general way that the material which was dried at 65°C. had a higher efficiency than that which was dried at 100°C. Roughly, the efficiency of the material dried at 65°C. was between 10 and 20 per cent, while that dried at 100°C. was less than 10 per cent.

41. The inconsistent results obtained when water is added to the material are probably due to the ununiform way in which the material is wetted. The water would dissolve out certain portions and thereby leave a channel through which the chlorine can pass. In addition, the material dried at 100° C. appears to have a glaze on the surface which is not present on that dried at 65° C. The glaze would make it difficult to wet the surface uniformly and thus probably explain why the material dried at 65° C. is more efficient.

42. It was discovered during the runs in which the hydrated material was used that if the tubes were filled and allowed to stand over-night before using, the efficiency was considerably decreased. For example, two samples, 5.4 and 5.3 grams of hydrated material, were placed in absorption tubes and allowed to sit over-night. The next day 0.42 per cent chlorine was passed through them and the break came at 28 and 24 minutes, respectively, as compared with about 200 minutes for the same amount of material which was not allowed to stand. The results were the same when the 1.46 per cent chlorine mixture was used.

43. In order to show how much additional absorption takes place after the break occurs, run number 9 was continued for some time. The exit gas was analyzed for chlorine and results are given below and plotted on Plate 11.

Time in Minutes	48	51	60	70	75	80	90	100	110	120
% of 1.46% chlorine mixture passing	0	1.34	12.2	34	52.5	65.5	79	85	87	88

"The larger the area of the exposed surface of the absorbent, the more efficient will be the result. When the length of submergence is such as to necessitate carbon dioxide elimination the following steps should be taken:

- (a) Remove the mattress covers from the mattresses of four lower bunks in the most convenient compartment provided with outboard ventilation when surfaced.
- (b) Remove mattresses, slit mattress covers and spread the covers, single thickness, as smoothly and taut as possible over bunk springs. Lash the edges to the bunk spring if necessary to keep it taut. Remove cover from one of the carbon dioxide absorbent containers and pour about one-fourth of the contents (approximately 3-1/2 pounds) on each cover. Spread the chemical as evenly as possible over the mattress covers. In pouring the chemical from the container and in spreading it on the mattress covers, care should be taken not to agitate it any more than necessary. It is caustic and the dust will cause throat irritation. The irritation, however, is only temporary and while in many instances, coughing and sneezing may be induced, the effects are not harmful and will soon be alleviated. When spreading the absorbent care should be taken to prevent its contact with any cuts or abrasions that may be on the hands. Do not rub the eyes after handling before the hands have been thoroughly washed. If it is accidentally gotten in the eyes painful, not dangerous, symptoms will occur. Such may be relieved by washing the eyes with a solution of 1 part vinegar or lemon juice and 6 parts of water or by careful washing with a quantity of fresh or salt water. Do not spread the chemical with the hands. Use a stick or other means. After spreading stir gently once each hour."

50. This method of course would not reduce the chlorine to a concentration that is safe for personnel, as can be seen from the results of the first two methods of this report. Any means of producing additional circulation of the contaminated air in a closed compartment in which the material is scattered would increase the rate of absorption as well as lower the final concentration. If power was available, this could be done by a blower or a fan. In the case where power was not available, consideration should be given to providing a hand-powered blower similar to that suggested by reference (2).

51. While the methods suggested above would certainly reduce the danger of chlorine to a large extent, consideration should be given to methods by which the danger could be almost completely removed. Such methods of course would depend upon whether or not the possible chances of such a danger would justify additional equipment and expense required.





No. 321. Co.



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HIS MUST BE OP. IF BHEET IS READ THE OTHER WAY (VERT 74)

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N. 16. 1



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PLATE 10