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The Seventh Partial Report on Oxygen Source Material

Absorption of Carbon Diozide and Liberation of Oxygen from Alkali Oxides on the Submarine U.S.S. S-30

> NAVAL RESEARCH LABORATORY ANACOSTIA STATION WASHINGTON, D. C.

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Date of Tests:	December 14 and 15, 1940
Prepared by:	
Inspared by:	R. R. Miller, Chemist
	W. C. Lanning, Associate Chemist
Reviewed by:	
	P. Borgstrom, Senior Chemist
Approved by:	

H. G. Bowen, Rear Admiral, U.S.N., Director

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ABSTRACT

Carbon dioxide and water vapor were absorbed by alkali oxides and oxygen liberated for thirty-five men for twenty-four hours with the submarine U.S.S. S-30 closed from outside ventilation.

The percentage of carbon dioxide and oxygen in the ship's atmosphere was followed by gas analysis on board. The reaction of the oxides was determined from samples taken at intervals and analyzed later at the Laboratory.

The data in this report shows that the carbon dioxide was removed and oxygen liberated at satisfactory rates under the conditions of this test.

The protection for oxygen generation and carbon dioxide removal is roughly 4.5 man hours per pound. For the protection of one man hour 0.22 pound is required. Compare this with the oxygen cylinder which requires about 0.6 pound per man hour (to which may be added 0.1 pound for the present carbon dioxide absorber used in the submarines if it is removed).

Observers on the test were: R. W. Dole, Lt. Condr., U.S.N. (N.R.L.); J. O'Shea, Lt., U.S.N.R.; A. R. Behnke, Lt., U.S.N. Medical Corps; Mr. Frank Hobson, Navy Department; Mr. Edward Sanner, Mine Safety Appliances Co.; Mr. E. W. Gilliland, Mine Safety Appliances Co.; Dr. P. Bergstrom, Naval Research Laboratory; Dr. W. C. Lanning, Naval Research Laboratory; Dr. E. A. Ramskill, Naval Research Laboratory; Dr. D. Burgess, Naval Research Laboratory; Mr. C. Lamb, Naval Research Laboratory; Mr. R. R. Niller, Naval Research Laboratory.



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AUTHORIZATION

BuC & R let. EN7(269) ME of 7-5-35 Other references: (a) BuAero Secret let. Aer-E-2-EP, F49-1, JC 10 of 2-23-40 (b) Bu C & R Secret let 3-SS-38(Mc) Ser. 1022 of 2-27-40.

STATEMENT OF THE PROBLEM

1. This is the seventh partial report on the development of an oxygen source material. This report gives the results of a test using the alkali oxides as an oxygen source and absorber CO_2 in the purification of the atmosphere in a submarine.

FACTS EEARING ON THE PROBLET.

2. The success of an oxygen source such as potassium tetroxide and socium peroxide depends on the rates of the reactions which take place. The oxides react with both moisture and CO_2 to liberate oxygen. Where the reactions take place at suitable rates, the water vapor, CO_2 and O_2 in the atmosphere, should remain essentially constant with the CO_2 at a suitably low percentage.

THEORETICAL CONSIDERATIONS

3. The reactions of K_2O_4 and Na_2O_2 have been discussed in previous partial reports and are briefly reviewed here. Carbon dioxide in the total absence of moisture at normal temperatures will not react with either K_2O_4 or Na_2O_2 . In the presence of moisture, the following reactions take place:

> (1) $K_2O_4 + CO_2^{+}(H_2O) \rightarrow K_2CO_3 + 3/2 O_2^{+}(H_2O) \xrightarrow{\text{Ratio } O_2/CO_2} \frac{1.5}{1.5}$ (2) $Na_2O_2 + CO^{+}(H_2O) \rightarrow Na_2CO_3 + 1/2 O_2^{+}(H_2O) \cdot 5$ (3) $K_2O_4 + H_2O + 2 CO_2 \rightarrow 2KH CO_3 + 3/2 O_2 \cdot 75$ (4) $Na_2O_2 + H_2O + 2 CO_2 \rightarrow 2 N_2H CO_3 + 1/2 O_2 \cdot 25$

With a mixture of 50 mol. per cent of K204 and Na202, and assuming equal reaction for both, the following reactions take place:

> $K_{2}O_{4} + Na_{2}O_{2} + 2 CO_{2} + K_{2}CO_{3} + Na_{2}CO_{3} + 2O_{2} + \frac{Ratio O_{2}/CC_{2}}{1.0}$ $K_{2}O_{4} + Na_{2}O_{2} + 2H_{2}O + 4 CO_{2} + 2 KH CO_{3} + NaHCO_{3} + 2O_{2}.50$

Reactions with water alone may take place as

 $K_2O_4 + H_2O \Rightarrow 2 \text{ KOH} + 3/2 O_2$ Na₂O₂ + H₂O \Rightarrow NaOH + 1/2 O₂



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4. The respiratory quotient (the ratio of CO_2 exhaled to oxygen consumed) for men with light work, as was the case in this test, is usually given as 0.83. The O_2 to CO_2 ratio required then is 1.0/0.83 = 1.20. The reactions in the material, to be satisfactory, must be a combination of the reactions above such that the ratio of O_2 liberated to CO_2 absorbed will fall near the 1.2 requirement. Then the atmosphere will stay essentially constant, with respect to percentage of O_2 present.

PREVIOUS WORK DONE AT THIS LABORATORY

Tests (to be reported) were carried out on absorption of CO2 5. and liberation of 02 by thin layers of both K204 and the mixed oxide (K204 - Ma202). From the rates of reaction of these samples, calculations were made to determine the required area of exposed material per man to absorb the CO2 and liberate sufficient oxygen. The time required for complete reaction of the material was determined for the thin unstirred layers. An adjustment was made for the fact that the samples were not stirred, while in an actual case the stirring-up of new material to the surface would be feasible and practical. These tests indicated that material could be spread approximately one pound per square foot and that it would react completely in circulated air with about 2% CO2 in six to eight hours. For material having an apparent density of approximately 60 pounds per cubic foot the thickness of the layers would average 1/4 inch and stirring would be required to bring new material to the surface. Pure K204 gives 3.8 cubic feet of 02/1b, and to last a man using one cubic foot of 02 per hour for eight hours would require 8/3.8 x 1, or 2.1 sq. ft. of material per man for pure K204. For the use of the mixed oxide with approximately 3.1 cubic feet of 02/1b., 2.6 square feet of surface was the indicated requirement per man for eight hours.

TESTS CONDUCTED ON U.S.S. S-30

6. Log of Test

December 14 - 10:30 to 11:00 - Placed materials and equipment aboard 11:15 - Closed part of the boat, one hatch remained

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- open. Apparatus was set up. 11:30 - Solid CO was pulverized and scattered. 11:50 - Boat was completely closed and ventilation
 - set at full speed. 2:00 - CO₂ had reached a fairly constant value throughout the boat. Gas analyses were being run at 4 points in the boat; the torpedo room, the forward battery room, the conning tower control room, and the engine room. K₂O₄ was spread.

2:20 - All of the K_2O_4 was out. Used 85.25 lbs.

2:20 P.M. December 14 to 12:45A.M., December 15 - Each hour the atmosphere was analyzed for 02 and CO2 (Table IX, App.), the material stirred and samples of the material gathered for analysis later (Table).

December 15-12:45 A.M. to 1:00 A.M. - Used K204 gathered and replaced in the tin cans.

1:00 A.M. to 1:30 - Mixed oxide spread. Used 85.75 lbs. 1:30 A.H. to 11:00 A.M. - Each hour the atmosphere was analyzed for 02 and CO2 and samples of the materials taken to be analyzed later. The material was stirred every 3/4 hour.

11:15 A.M. - Boat was opened.

11:15 A.M. to 12:00 M. - All material and equipment gathered and returned Naval Research Laboratory.

7. To hasten the test, 41.06 lbs. of solid CO2 was taken into the boat. This on evaporation amounted to 362 cubic feet or 1.67% CO2 in the atmosphere of the boat. This was evaporated before the last hatch was closed and, therefore, forced that volume of air from the boat. The K201 used was heated and ground material. The mesh size ranged from dust to 4 mesh, and the apparent density was about 40 lbs/cu ft. which was somewhat less than the average for this material. The apparent density depends on the temperature to which the material has been heated before grinding. The material was spread with a minimum of dusting by turning the open cans open end down on the surface and allowing the materials to flow out by lifting the can slowly away from the surface. The surfaces on which the material was spread were thin black iron sheets which had a 2 inch edge turned up to form open end trays, 30 inches wide and 6 feet long. Two rows of three trays each were placed end to end under the bunks in the forward battery compartment. The total area was 96 square feet, but since the ends were open the material was scattered to within 3 inches of each end. This gave an area for the material of 82-1/2 sq. ft. With a weight of 85.25 pounds of K_20_4 , this gave 1.03 pounds/sq. ft. of area. The thickness of the material varied from 1/4 to 1/2 inch, but with stirring this thickness varied over the surface from time to time. Temperatures taken in the material are given in Table II. A brief table of data follows and more complete data will be found in the Appendix.

> Material Per cent Purity Oxygen per pound Weight of material used Oxygen available (Total) Oxygen liberated/lb. when spread out Oxygen liberated (Total) while spread 241 cu. ft. n.t.p.

K204 (potassium tetroxide) 94% 3.55 cu.ft. at n.t.p. 85.25 lbs. 302.5 cu. ft. at n.t.p. 2.83 cu. ft. at n.t.p.

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Oxygen liberated (Total) including that from canned material liberated from cans	
after material was gathered up	297 cu. ft. n.t.p.
Per cent available Oxygen liberated while	5.00
spread	80%
Per cent available Oxygen liberated includ- ing that liberated from cans after material	
was gathered up	98%
CO2 absorbed/1b.	2.90 cu. ft. at n.t.p.
CO2 absorbed (Total)	247.2 cu. ft.
Oxygen in the atmosphere of the torpedo	1210103 • EP • U.S. 2012000 - Sector-Graver - C. 20209 January
compartment at the start of the K20, tests	19.4%
Oxygen in the atmosphere of the torpedo com-	-2 4
partment at the end of the K204 tests	19.35%
CO2 in the atmosphere of the torpedo com-	
partment at the start of the K201 tests	2.13%
CO2 in the atmosphere of the torpedo com-	
partment at the end of the K201 tests	1.91%
Air temperature	22°C (71°F)
Temperature range in the material	35°C to 50°C
Area of material	82.5 sq. ft.
Weight of material/sq. ft.	1.03 lb.
Number of men	35*
	2.47 sq. ft.
Length of test	10-1/2 hrs.

* Except for two and one-half hours when 34 men were present.

8. The mixed oxide used was heated and ground to the same mesh size at K_2O_4 (dust to 4 mesh). It was spread on the same sheets with in additional sheet, two feet by six feet, placed in the torpedo compartment. The total area of the mixed oxide was 94-1/2 sq. ft. The weight of material spread was 87.75 lbs. or 0.93 lb. sq. ft. It was stirred at intervals and the temperature in the material noted. A brief table follows and more complete data will be found in the appendix.

Material Per cent Purity

> O2/pound Weight of material used Oxygen available (Total) Oxygen liberated/lb. Oxygen liberated (Total) Per cent of available O2 liberated CO2 absorbed/lb. CO2 absorbed (Total)

Mixed Oxide 80.5% of the O2 value for pure K2O4 3.06 cu. ft. at n.t.p. 87.75 lbs. 268 cu. ft. n.t.p. 2.88 cu. ft. n.t.p. 252 cu. ft. n.t.p. 94% 2.73 cu. ft. n.t.p. 239.5 cu. ft. n.t.p.

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O ₂ in the atmosphere of the torpedo compartment at start of mixed oxide	
test.	19.35%
02 in the atmosphere of the torpedo	
compartment at end of mixed oxide test	19.60%
CO2 in the atmosphere of the torpedo com-	
partment at start of mixed oxide test	1.91%
CO2 in the atmosphere of the torpedo com-	
partment at end of mixed oxide test	1.95%
Air temperature	21° C (70°F)
Temperature range of the material	42.°C to 55°C
Number of Men	35 thru 40 (see Table I)
Area of Material	94.5 sq. ft.
Weight of material/sq. ft.	0.93 lbs.
Area per man (based on av. no. of men)	2.52
Length of test	9.5 hrs.

DISCUSSION OF RESULTS

9. From analyses of samples taken from the trays at intervals and at the end of the test it was found that the K_2O_4 had liberated 2.83 cu. ft. of oxygen per pound of dry material or 80% of its total oxygen (a total of 241 cu. ft. of oxygen at n.t.p.). The carbon dioxide absorbed during the same time was 2.9 cu. ft. per pound, a total of 248 cu. ft. of CO₂ at n.t.p. The material was then put into cans for return to the Naval Research Laboratory. During this storate period the material reacted further, liberating 56.3 cu. ft. of the 61.5 cu. ft. of oxygen still available. In all, the K_2O_4 liberated 297.3 cu. ft. of oxygen at n.t.p.

10. From the mixed oxides 2.88 cu. ft. of oxygen was liberated per pound, or a total of 252 cu. ft. at n.t.p. At the same time this oxide absorbed 2.73 cu. ft. of CO₂ per pound or 172.8 cu. ft. total.

11. From the data formed in Table V a balance can be made on the test from 1:00 P. M. December 14th to 11:00 A.M. December 15th, or 22 hours. From Table I this period of time will represent 794 man hours. Using the following data obtained in the torpedo compartment:

	K204	Mixed oxide
% Og begin	19.4	19.35
% O2 begin end	19.35	19.60
% CO2 begin	2.13	1.91
% CO ₂ begin end	1.91	1.95
Change in oxygen	-0.05%	+0.25%
" " CO2	-0.22%	+0.04%
Change in complete test	- 8 -	

02	+0.2%
02 C02	-0.18%
P	P

As the gas analyses may be open to a fluctuation of as much as $\pm 0.1\%$ due to a variation in samples taken from the circulating air, it is probable that the man hours protection per pound can be approximately calculated from total man hours divided by weight of material, or

$$\frac{794}{173}$$
 or 4.6 man hours per pound.

If the gain in oxygen is considered the value is $\frac{794 + 55}{173} = 4.9$ man hours per pound for oxygen, and

 $\frac{794+60}{173} = 5.0 \text{ man hours per pound for}$

carbon dioxide protection.

Description of

12. From the analyses it is interesting to note the following:

Weight of material (Total)	173 lbs.
CO2 absorbed	
Cu. ft. n.t.p.	487
lbs.	59.6
02 liberated	
cu. ft. n.t.p.	549
lbs.	48.9
Ratio	ing milt and lite
Q2	1 10
COn	1.13
6	

This value of 1.13 is very interesting for it shows that the ratio of oxygen liberated to CO_2 absorbed is nearly the desired value of 1.2 as discussed in paragraph 4.



CONCLUSIONS

13. The data obtained in this test shows that oxygen can be liberated and carbon dioxide absorbed from the atmosphere of a submarine (relative humidity about 50% and carbon dioxide 2%) is nearly the correct ratio for respiration. To maintain the carbon dioxide at a lower concentration and to supply the necessary oxygen, it will probably require a larger area of material per man if the experiment is conducted under conditions similar to this test. More detailed data on the ratio of liberation of oxygen and the absorption of carbon dioxide as determined from the analysis of samples of the oxides will be found in the Appendix. It should be noted that the nearly expended material (oxides) can be put into a can and more oxygen will be liberated. This is probably due to the moisture absorbed by the carbonate or by dioxide before it was collected and put into the container where it can react with the remainder of the unreacted oxides.

14. The man hours protection per pound is over four which is very good under these conditions.

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RECOMMENDATIONS

15. Two further tests are recommended for these materials. One test should be made at the lowest temperature encountered in service, about $35^{\circ} - 45^{\circ}$ F. The other test should be made under tropical conditions with a temperature of 80° to 90° F and high humidity.

APPENDIX

The tables and plates below give the data obtained from analyses of the boat's atmosphere and of samples of materials removed during the test. Table III gives the results of analyses of the K_2O_4 and also the calculated values determined from these analyses. Plate I gives some of the values from Table III in graphic form. Plate II gives the calculated values for the rate at which oxygen was liberated in the K204. Table IV for the mixed oxide gives the results of the analyses and other values calculated from the analyses. These values are plotted on Plate III. The calculated rate of evolution of oxygen from the mixed oxide is shown graphically on Plate IV. Values have also been calculated from the analyses for the ratio of oxygen liberated to CO2 absorbed during the test. The curves for these values for both $\tilde{K}_{2}O_{4}$ and the mixed oxide are given on Plate V. On Plate VI is a graphic representation of all the analyses taken on the boat's atmosphere. These are also given in tabular form in Table V. Data have been placed in Tables III and IV showing the analyses on the used material taken from the cans after standing three days at the Laboratory. The available oxygen for both materials was almost completely liberated.



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TABLE I

Number of Men Aboard the U.S.S. S-30

Time 1	Number o: Men	f
11:10 AM to 5:30 PM	35	
5:30 PM to 8:00 PM	34	
E:00 PM to 12: Nidnite	e 35	
12:00 to 4:00 AM	36	
4:00 Ali to 8:00 Ali	37	
E:00 to 9:00	39	
9:00 AN to 11:15 AM	40	
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TABLE	11

Temperature of the Material While Spread

K204

Mixed Oxide

Time after spreading hrs.	Temperature °C	Room Temp.	Time after spreading hrs.	Temperature °C	Room Temp.
0	21	21° C	0	21	21° C.
1.0	50		1.0	55	
1.7	49	11	1.5	53	11
2:3	50		2.0	55	11
3.2	46		2.7	52	
4.0	43		3.25	55	
5.7	46		4.0	51	
6.2	46	11	4.75	53	11
6.7	44		5.50	52	
7.3	41		6.25	46	
0.9	42		7.2	47	
8.3	40	11	8.0	44	11
9.0	39		8.5	43	
9.7	37	11	9.25	42	11
10.2	35				



TABLE III

Data on the Analyses of Material Samples

 K_2O_4 - Original O_2 3.55 cu.ft./lb. at N. T. P.

Time of Exposure	Sam- ple #	Dry Ut. of Sam- ple		e Sample	Resid- ual O2	ated O2	% of K204	Absorbed CO ₂ cu.	Ratio 02 Liberated
nours	#	(Grams)	N.T.P. 02	(00) CO ₂	cu.ft. per lb.	cu.ft. per 1b.	decom- posed	ft./1b.	CO2 ab- sorbed
	l	9.59	1590	475	2.65	0.90	25.3	0.79	1.14
1-1/2	2 3	13.52	2325	630	2.75	0.80	22.5	0.75	1.07
1.0		10.52	1090	510 .	2.57	0.98	27.6	0.78	1.20
2-1/2	4	14.55	2165	840	2.38	1.17	33.0	0.92	1.27
2.57	5	8.88	1090	700	1.90	1.59	44.8	1.20	1.26
3-1/2	6	14.84	1895	1070	2.04	1.51	42.5	1.15	1.31
2-313	7	10.99	1200	1025	1.83	1.72	48.5	1.49	1.15
4-1/2	8 9	- 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1	- 076	- 0150		-	-	- T	-
0-110	9	11.80	1045	1365	1.42	2.13	60.0	1.85	1.15
5-1/2	10	12.92	1000	1560	1.31	2.24	03.1	1.93	1.10
3-31-33	11	10.42	830	1315	1.28	2.27	64.0	2.02	1.12
6-1/2	12	13.32	930	1820	1.12	2.42	68.5	2.19	1.11
	13	11.76	830	1620	1.13	2.42	68.2	2,20	1.10
7-1/2	14	11.74	770	1790	1.05	2.50	70.4	2.44	1.02
	15	9.31	635	1515	1.09	2.46	09.3	2.60	.95
8-1/2	16	11.50	660	1875	.92	2.63	74.1	2.61	1.01
	17	11.14	610	1870	0.88	2.67	75.2	2.69	0.99
9-1/2	18	10.85	530	1870	0.78	2.77	78.0	2.76	1.00
	19	12.54	615	2280	0.78	2.77	78.1	2.91	.95
10-1/2	20	13.02	560	2380	0.69	2.80	80.5	2.92	.98
	Ar	nalysis on mat	erial from	the cans 3	days late	r at the	Laborato	ory	
	1	13.30	10	2570	.01	3.54	99	3.08	1.15
	2	11.65	260	2370	.30	3.25	91.5	3.25	1.00

TABLE IV



Data on the Analyses of Material Samples

Mixed Oxide Original N.O. Had 3.06 cu. ft. of 02/1b. N.T.P.

Time of Exposure Hours	Sam ple #	Dry Wt. of Sam- ple (Grams)	Volume of Gas from the Sample N.T.P. (CC)		Residual O ₂ cu.ft. per 1b.	Liberat O2 cu.f per lb.	t. 1.0.	Absorbed CO ₂ cu. ft./lb.	Ratio 02 Liberated CO2 ab-
			02	C02	P		posed		sorbed
	21	16.82	2920	425	2.78	0.28	9.16	0.40	0.70
1-1/2	22	11.76	2170	255	2.95	0.10	3.60	0.35	0.31
- 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1	23	14.08	2340	665	2.60	0.40	13.10	0.70	0.53
2-1/2	24	14.64	2535	b50	2.77	0.29	9.5	0.71	0.41
155 15	25	11.30	1620	815	2.28	0.78	25.5	1.15	0.08
3-1/2	26	12.99	1940	870	2.39	0.67	21.9	1.07	0.00
	27	8.42	935	835	1.78	1.28	41.8	1.59	0.81
4-1/2	28	10.88	1305	1000	1.92	1.14	37.3	1.47	0.78
	29	7.86	650	925	1.32	1:74	50.9	1.88	0.93
5-1/2	30	12.72	1250	1340	1.57	1.49	48.7	1.69	0.88
	31	9.52	450	1375	.760	2.30	75.2	2.31	1.00
6-1/2	32	10.74	785	1335	1.17	1.89	01.8	1.99	.95
-11-2	33	8.98	250	1495	0.45	2.01	85.3	2.60	.98
7-1/2	34	11.08	500	1630	0.72	2.34	76.5	2.35	1.00
	35	9.14	85	1775	.15	2.91	95.1	3.11	.94
8-1/2	36	9.72	195	1010	0.32	2.74	89.0	2.05	1.03
	39	12.07	200	2085	0.20	2.79	91.2	2.70	1.01
9-1/2	40	11.26	81	1895	0.115	2.94	90.1	2.69	1.09

Analysis of material removed from the cans 3 days later at the Laboratory

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l	12.90	20	2330	.02	3.04	99	2.47	1.23
2	15.75	30	3150	.03	3.03	99	2.75	1.10

TABLE V

Data on Gas Analysis in the U.S.S. S-30

OXYGEN

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Time	Torpedo	C.O.C.	Forward	Engine	Torpedo	Ç.O.C.	Forward	Engine	
	Room	Room	Battery	Room	Room	Room	Battery	Room	
11:50	21.5	-		-	0.85		-	-	
12:10	19.8	-	-	-	1.54				
12:00		-	19.94	20.1		2.35	1.03	2.10	
1:00	19.4	19.30	19.58	19.3	2.13	2.15	2.24	2.58	
1:30	19.8	-	19.53		2.33		2.28		
1:45	~	19.10	19.32		-	2.52	2.33	2.35	
2:45	19.2	-	19.42	19.38	2.59	2.52	2.21	2.40	
3:30	19.4	-	19.49	19.50	2.29	2.27	2.25	2.35	
4:00		-	19.05	-		2.23		-	
4:30	19.5	19.80	19.60	19.50	2.11	2.10	2.10	2.38	
5:30	_		19.65	-	2.07		2.06	2.39	
0:30	20.2	19.85	19.79		1.97	2.27	2.06	ent	
7:00		19.68			_	2.32	_	_ 712	
7:30		19.75	19.65	20.0	2.18	2.28	2.08	2.25	
7:50				-	2.15			_	
8:30	20.1	19.95	19.52	19.0	2.14	2.25	2.14	2.13	
9:30	19.5	19.88	19.48	19.4 .	2.25	2.20	2.13	2.10	
10:30	19.35	19.75	19.41	19.5	2.37	2.20	2.14	2.13	
11:00		_	_		2.10		_	_ 5°31	
11:30	19.05	19.05	19.35	13	2.02	2.30	2.17	2.1	
12:30	19.35	19.02	19.25	19.5	1.91	2.33	2.19	2.26	
1:30	-	19.58	19.25	-		2.34	2.29		
2:30	19.50	20.62	19.53	19.55	2.31	2.28	2.21	2.33	
3:00	-		19.41	-		2.22	2.22	-	
3:30	19.50	_	19.40	19.8	2.22	-	2.21	2.10	



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Time	Torpedo Room	C.O.C. Room	Forward Battery	Engine Room	Torpedo Room	C.O.C. Room	Forward Battery	Engine Room
4:30	19.15	19.90	19.48	19.45	2.30	2.30	2.05	2.14
5:30	19.60	19.80	19.55	19.6	2.00	2.15	2.03	1.99
0:30	19.35	19.81	19.61	20.0	2.04	2.15	1.89	1.94
7:30	19.70	19.85	19.55	19.9	1.98	2.10	1.83	1.82
8:30	19.55	19.9	19.71	20.0	1.99	2.03	1.81	1.85
9:30	19.75	19.92	19.08	19.8	1.93	2.05	1.87	1.90
LO:30	19.35	19.95	19.70	19.9	1.99	1.95	1.86	1.99
11:10	19.60	-	19.45	-	1.95	-	1.98	-
750 0.00								
UEC	Lacsifie	0						
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TABLE V (Cont.) Data on Gas Analysis in the U.S.S. S-30

plant on the sharpbarth to all the state of the

Margar A.











