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The Eighth Partial Report on Oxygen Source Compounds

Laboratory and Canister Tests on Alkali Oxides



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NAVAL RESEARCH LABORATORY

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February 28, 1941

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NAVY DEPARTMENT

The Eighth Partial Report on Oxygen Source Compounds
Laboratory and Canister Tests on Alkali Oxides

NAVAL RESEARCH LABORATORY
ANACOSTIA STATION
WASHINGTON, D. C.

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ABSTRACT

Oxygen liberation from the higher oxides of potassium and sodium has been studied in the presence of inorganic compounds as catalysts. Laboratory tests have indicated an enhanced rate of liberation by water vapor with some of the catalysts.

Sodium peroxide has been hydrated in the presence of K_2O_4 and the resulting material showed somewhat greater activity in the absorption of CO_2 .

Breathing tests on canisters of potassium tetroxide show that oxygen liberation and CO_2 absorption take place at rates suitable for breathing purposes. The lowest temperature at which canisters can be used and the life of the canister depend on the size and construction of the apparatus and the amount of activity of the breather.

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Preparations of the Hydrates of the Mixed Oxide	Appendix, Page 1
Effect of Catalysts upon the Rate of Reaction Between Water Vapor and Oxygen Source Materials	" Page 1
Table III - Results of Laboratory Tests on Catalyzed Samples	" Page 2

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AUTHORIZATION

This problem was authorized by Bureau of Aeronautics letters as follows:

- (a) Confidential letter Aer. E-251 SG F49-1 of October 19, 1937.
- (b) BuAero. Secret letter Aer-E-2 EP, F49-1 JC 10 of February 23, 1940.
- (c) BuC&R Secret letter S-SS-38 (Mc) Ser. 1022 of February 27, 1940.

STATEMENT OF PROBLEM

1. The general problem is a study of chemical compounds which may be caused to react under practical conditions to produce oxygen gas suitable for breathing purposes. This report gives the results of tests on three phases of the problem:

- (a) Laboratory tests on catalyzed alkali oxides.
- (b) Hydration and reaction of the mixed oxides.
- (c) Breathing tests showing comparison of canisters.

KNOWN FACTS BEARING ON THE PROBLEM

2. The reactions of alkali oxides have been reported and discussed in NRL Report P-1612. The present report gives results on the tests which were outlined in the above-mentioned report. The results of these tests indicate that the nature of air flow through the material and nature of the breathing are both factors which influence the efficiency of breathing canisters. In Report P-1612 GOX, sodium peroxide (Na_2O_2) and potassium tetroxide (K_2O_4) were discussed, and in Report No. P-1619, the preparation of a mixed alkali oxide was described. This oxide was made from a mixture of sodium and potassium metal and contained 80-85% as much available oxygen as pure K_2O_4 . As far as tests have progressed, the weight efficiency and the reaction efficiency both favor pure K_2O_4 over the other three preparations.

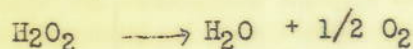
HYDRATES OF OXYGEN SOURCE MATERIALS

3. No hydrate of K_2O_4 is known, and since water in the liquid or vapor form added to K_2O_4 always reacts to set free oxygen, no hydrate is believed to exist. The sodium peroxide hydrates are known to exist; two are given in the literature: the dihydrate ($\text{Na}_2\text{O}_2 \cdot 2 \text{H}_2\text{O}$), and the octahydrate ($\text{Na}_2\text{O}_2 \cdot 8 \text{H}_2\text{O}$). The octahydrate decomposes near room temperature while the dihydrate is stable to considerably above room temperature. The dihydrate has 46% water on the dry basis, and the octahydrate has 185% of its dry weight as water. The dihydrate has

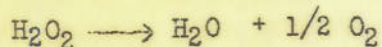
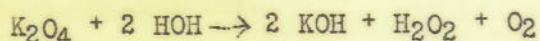
twice the amount of water required to react with the peroxide to set free the available oxygen and the octohydrate has eight times the required amount. The reaction of Na_2O_2 with its water of hydration can be brought about in either case by an increase in temperature. The hydrates under breathing conditions should absorb more water and CO_2 which should result in an increase in temperature and, in turn, would cause the water of hydration to react. In this way, it seems reasonable to expect that a greater amount of oxygen could be liberated than by the reaction of the exhaled moisture and CO_2 alone. The hydrate might readily combine with dry CO_2 , while dry CO_2 and dry unhydrated sodium peroxide do not react.

THEORETICAL CONSIDERATIONS

4. The decompositions of the alkali oxides in liquid water take place with the formation of hydrogen peroxide. With sodium peroxide, most of the oxygen probably forms hydrogen peroxide and the oxygen is evolved by the decomposition of the hydrogen peroxide.



With K_2O_4 only $1/3$ of the O_2 will form H_2O_2



While no H_2O_2 has been found in the oxygen liberated by the oxides with water vapor, it is possible that the above reactions may take place in a very thin layer at the boundary of the reacted and unreacted material. If such is the case, a catalyst which favors the decomposition of hydrogen peroxide in water may enhance the rate of reaction.

EXPERIMENTAL WORK

5. A number of compounds were tested as catalysts for the above reactions while passing water vapor through the material. The materials were placed in glass tubes held in a constant temperature bath while air, saturated with water vapor, was circulated through the samples in a closed system. The increase in total volume of gas was liberated oxygen. From this volume increase, the rate of liberation of oxygen per minute was determined. Details of the method and complete results of the tests are given in Appendix, Table III, and Plate 27. A few typical results are given in Table I. The catalytic materials were added by two methods, one by mixing the catalyst with the fine unheated oxide, then heating to sinter. This gives an intimate mixture of catalyst and oxide. The other method of addition was dusting the dry, finely powdered catalyst on the particles when they were ready for use; in this case the catalyst was only on the surface.

TABLE I

Rate of Liberation of Oxygen

Material	Catalyst	O ₂ liberated in cc/minute at the times given after start					
		1 min	2 min	3 min	5 min	8 min	10 min
K ₂ O ₄	None	3.0	4.0	4.5	4.8	5.1	5.2
K ₂ O ₄	Zn (pwd.)	2.7	3.4	3.9	4.4	4.9	5.1
K ₂ O ₄	Copper (pwd)	4.6	6.2	7.0	7.8	8.2	8.1
K ₂ O ₄	Copper sulphate(anhy.)	5.9	7.8	8.4	8.9	9.0	9.1
Mixed Oxide	None	1.4	1.7	2.0	2.3	2.3	2.2

The table gives the rate at which oxygen was being liberated in cubic centimeters per minute at the times noted. Air saturated with water vapor at 23° C. was passed through the samples at the rate of one liter per minute.

6. A few of the catalysts which increased the rate of reaction were added to larger samples for use in canisters. Preliminary tests have not shown any great increase in the rate of evolution of oxygen under breathing conditions. Further tests are to be made on canisters to which catalytic materials have been added.

7. Hydrates of a Mixed Oxide. Samples of the mixed oxide were held in contact with water vapor at approximately 5 mm partial pressure for different lengths of time. The samples showed different weights of absorbed water and on analysis appeared to be hydrated to approximately the extent calculated from the weight increase. A description of the method of forming hydrates is given in the Appendix, page 1. The data on the hydrates are given in Table II.

TABLE II

Sample #	% O ₂ on the basis of pure K ₂ O ₄	% water absorbed	%O ₂ of hydrate on K ₂ O ₄ (calc.)	%O ₂ of hydrate on K ₂ O ₄ (analysis)
1	80.9	3.9	77.7	76.9
2	80.9	8.9	74.2	72.2
3	80.9	2.76	78.8	--
4	80.9	6.2	76.2	--
5	80.9	8.2	74.6	--
6	80.9	4.65	77.2	77.3

Samples number 2 and 4 were heated in a closed system to determine the temperature at which oxygen would be evolved. In both cases O₂ in measurable quantities was evolved at 62°C. (144°F.) Sample number 4 was held between 50° and 55°C. for two hours without measurable evolution of oxygen. Samples heated to higher temperatures until the reaction was complete evolved more oxygen than the amount which would be calculated from the reaction of the water present and sodium peroxide. During the heating process, a part of the moisture had reacted with K₂O₄.

8. Carbon Dioxide and Hydrates. Hydrates containing 6 to 15% water removed CO₂ from dried nitrogen-carbon dioxide mixtures to -10°C.; below this temperature no CO₂ was removed. When the nitrogen-CO₂ mixture passed through diluted sulphuric acid solution to give a partial pressure of water equal to 5 mm., the CO₂ was removed by the hydrated material at -25°C. and partially removed at -50°C. The same gas mixture with water vapor passed through a cold coil at -30°C. and then on to the sample at -30°C. showed only partial removal. The partial absorption of CO₂ at the cold temperatures and removal from dry gases at higher temperatures indicate a greater activity towards CO₂ than the unhydrated mixed oxide. Hydrates tested in canisters are described below.

9. Breathing Tests. The apparatus used is shown diagrammatically on plates 28 and 29. The "flat canisters" used on the number one type apparatus were old GOX canisters from Mine Safety Appliance Co; they were made as shown on plate 28. Apparatus number two, using "round canisters," was constructed at the Laboratory from odd parts made by the above concern. The valves were from a Navy submarine escape lung. The air supply bag was of the type used on the older Bureau of Aeronautics flying apparatus. The mouthpiece was a Hydebrink transparent cup with inflated rubber edging.

10. The canisters were loaded in layers approximately 3/4" thick, numbered in the direction of air passage through the material; in each case the air started into the material at the bottom of the canister and moved upward so that the number one layer was at the bottom. Temperature was measured with a thermocouple inserted between the second and third layers and a permanent record kept by use of a recording pyrometer. Gas samples were drawn from the air supply bag and analyzed in an Orsat or Haldane gas absorption apparatus. A measure of the rate of breathing and an indication of the resistance to breathing were obtained with a respiration recorder which was constructed at the Laboratory.

11. In starting a breathing test, one breath was exhaled into the bag through the canister, a second breath of air was taken and the mask put on. This method was followed in all cases except where otherwise noted. An artificially rapid breathing rate was usually maintained for a few minutes, until the canister reaction had well started; thereafter the observer attempted to breath normally.

12. Oxygen and carbon dioxide percentages are shown plotted against time in the plates below. In the paragraphs on the pages opposite are contained notes relevant to the special conditions under which the particular canisters were studied. By "bath temperature" is meant the temperature of the air bath in which the canister and air supply bag were suspended; in the walking tests at the end of the series, this temperature was that of the outside air. By "by-pass" is meant the small tubing used to conduct exhaled air through a given lower layer of material without its contacting that material. (See Discussion of Results and Plates 28 and 29). The heading "canister type" denotes not only the canister used but the apparatus with which it was studied (i.e., the "flat canister" could be used only with apparatus number one and the "round canister" only with apparatus number two.)

Graph 1

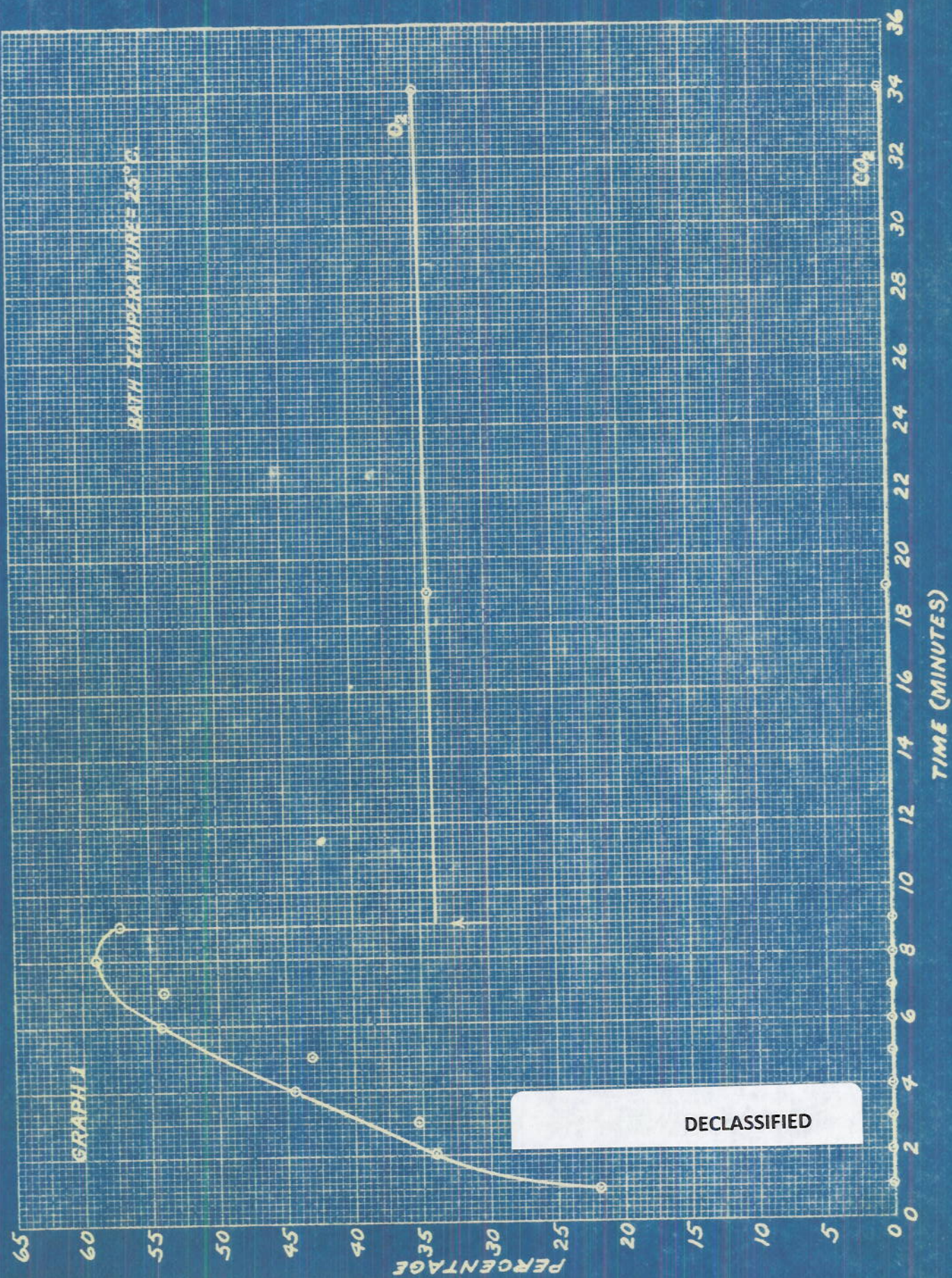
Canister type: Flat.
Date packed: August 14, 1940.
Date used: October 22, 1940.
Initial weight of material: 279.4 gms.
Bath temperature: 25°C.
Insulation: None

<u>Layer</u>	<u>Material</u>
1	K_2O_4 + 1 gm. LiOH
2	K_2O_4
3	K_2O_4
4	K_2O_4

Breathing was discontinued after 9 minutes and then resumed after a pause of 26 minutes. The air supply was practically depleted 25 minutes later.

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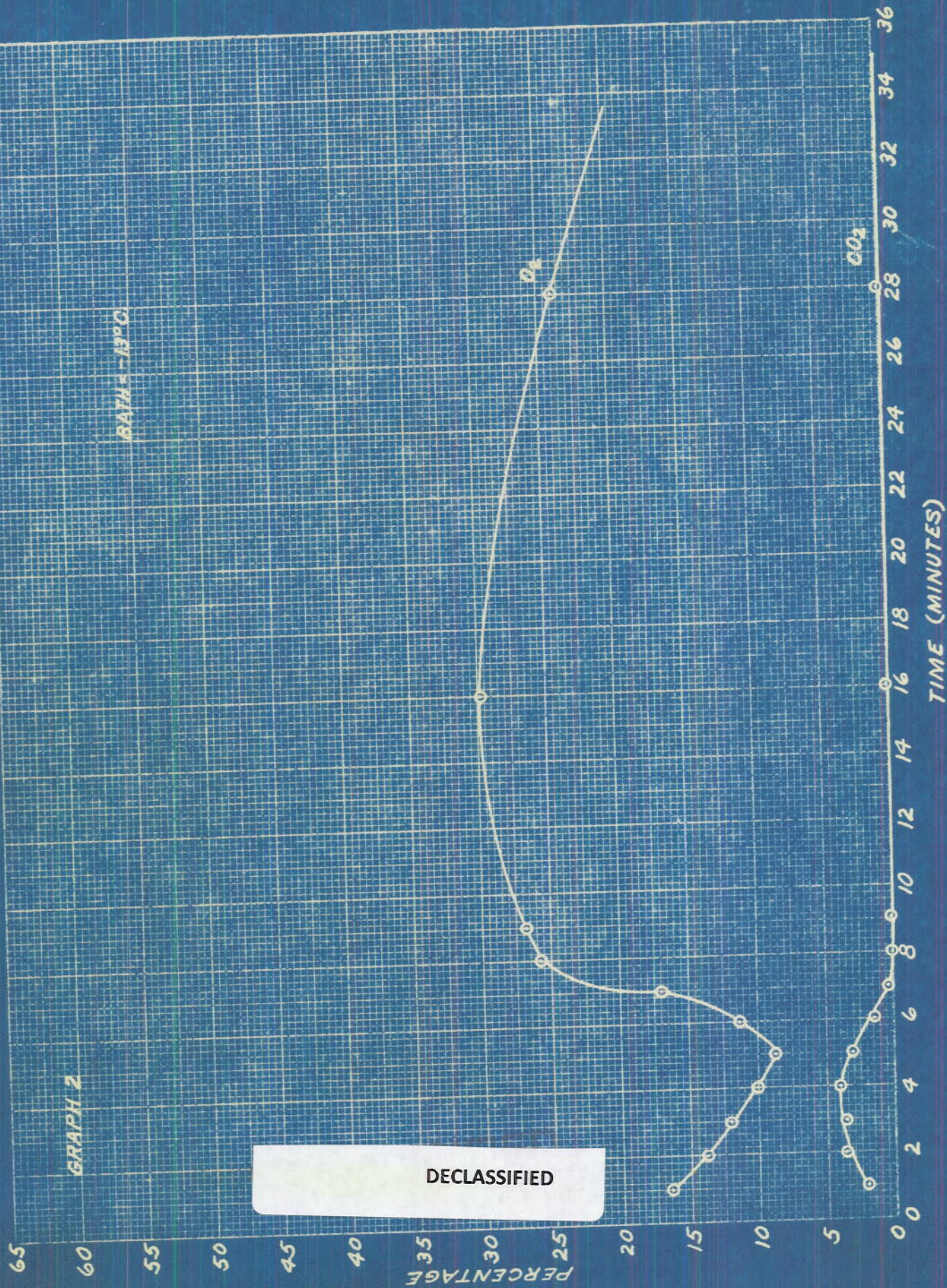
Graph 2

Canister type: Flat.
Date packed: August, 1940.
Date used: October 23, 1940.
Initial weight of materials: 270.8 gms.
Bath temperature: -13°C.
Insulation: Wrapping of asbestos paper.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	None
2	K ₂ O ₄	"
3	K ₂ O ₄	"
4	K ₂ O ₄	"

After 28 minutes of breathing the air supply was practically depleted. Canister reached a maximum temperature after 12 minutes.

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Graph 3

Canister type: Flat.
Date packed: August 8, 1940.
Date used: October 24, 1940.
Initial weight of material: 286 gms.
Bath temperature: -13°C.
Insulation: Wrapping of asbestos paper.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄ + 1 gm. LiOH	None
2	K ₂ O ₄	"
3	K ₂ O ₄	"
4	K ₂ O ₄	"

No particular difficulty was observed during the first few minutes on this canister. Carbon dioxide adsorption was good and the air supply was adequate up to 69 minutes. Canister reached a maximum temperature after 20 minutes.

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GRAPH 3

WATER = -13°C.

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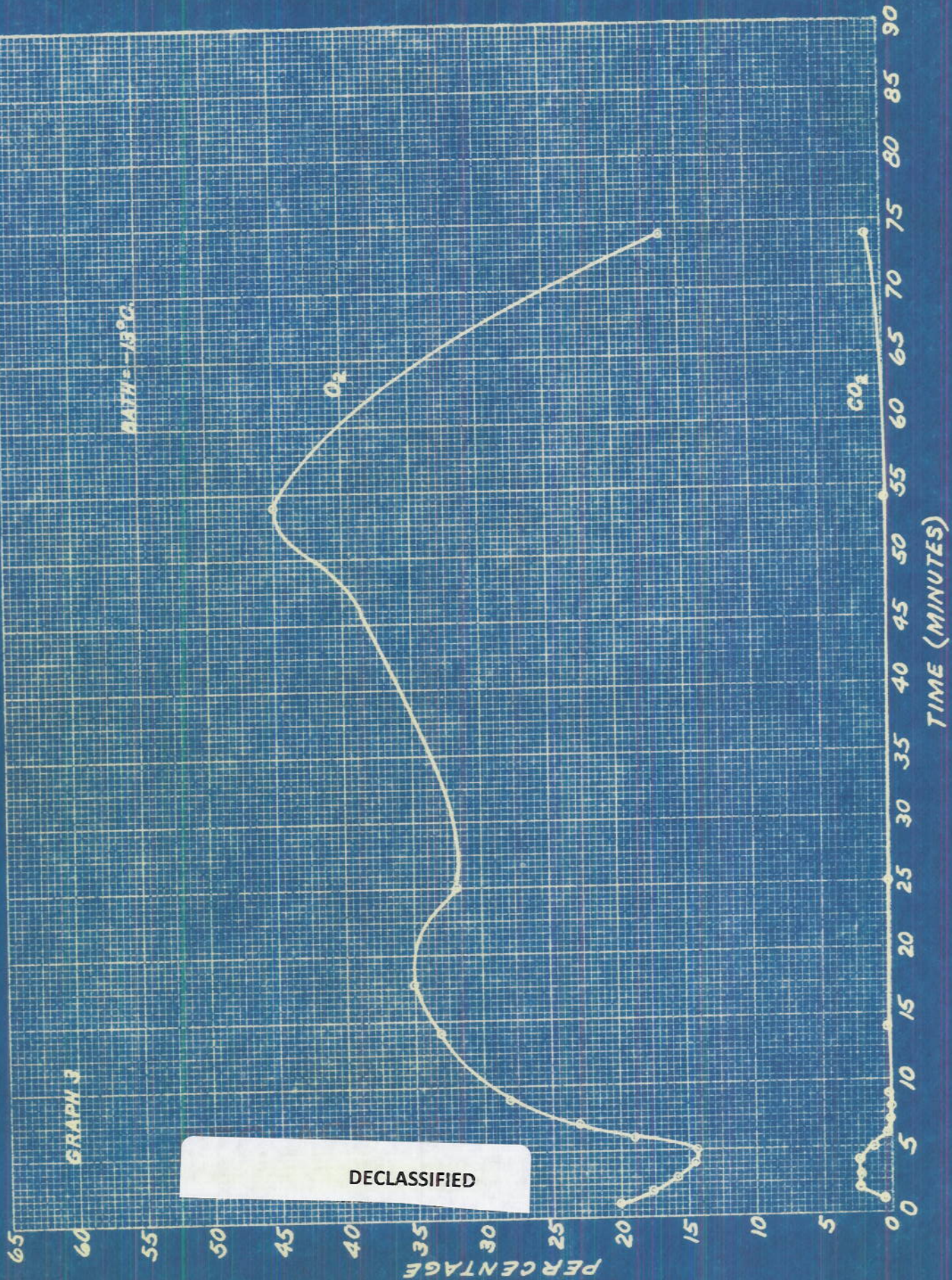


PLATE #3

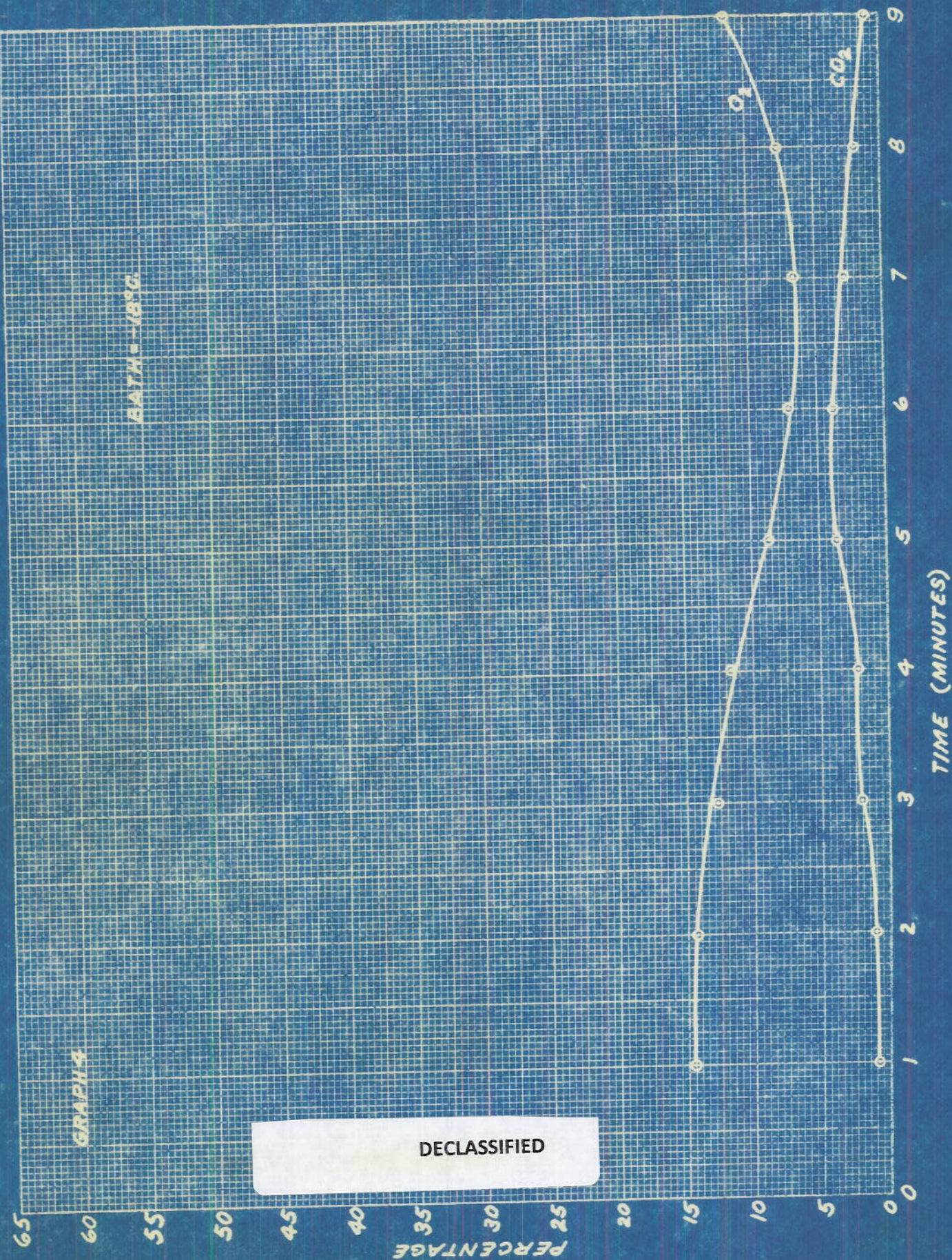
Graph 4

Canister type: Flat.
Date packed: August 8, 1940.
Date used: October 24, 1940.
Initial weight of material: 282.1 gms.
Bath temperature: -18°C.
Insulation: Wrapping of asbestos paper.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄ + 1 gm. LiOH	None
2	K ₂ O ₄	"
3	K ₂ O ₄	"
4	K ₂ O ₄	"

This canister was beginning to react more rapidly after 9 minutes, but by that time, the observer was utterly exhausted.

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Graph 5

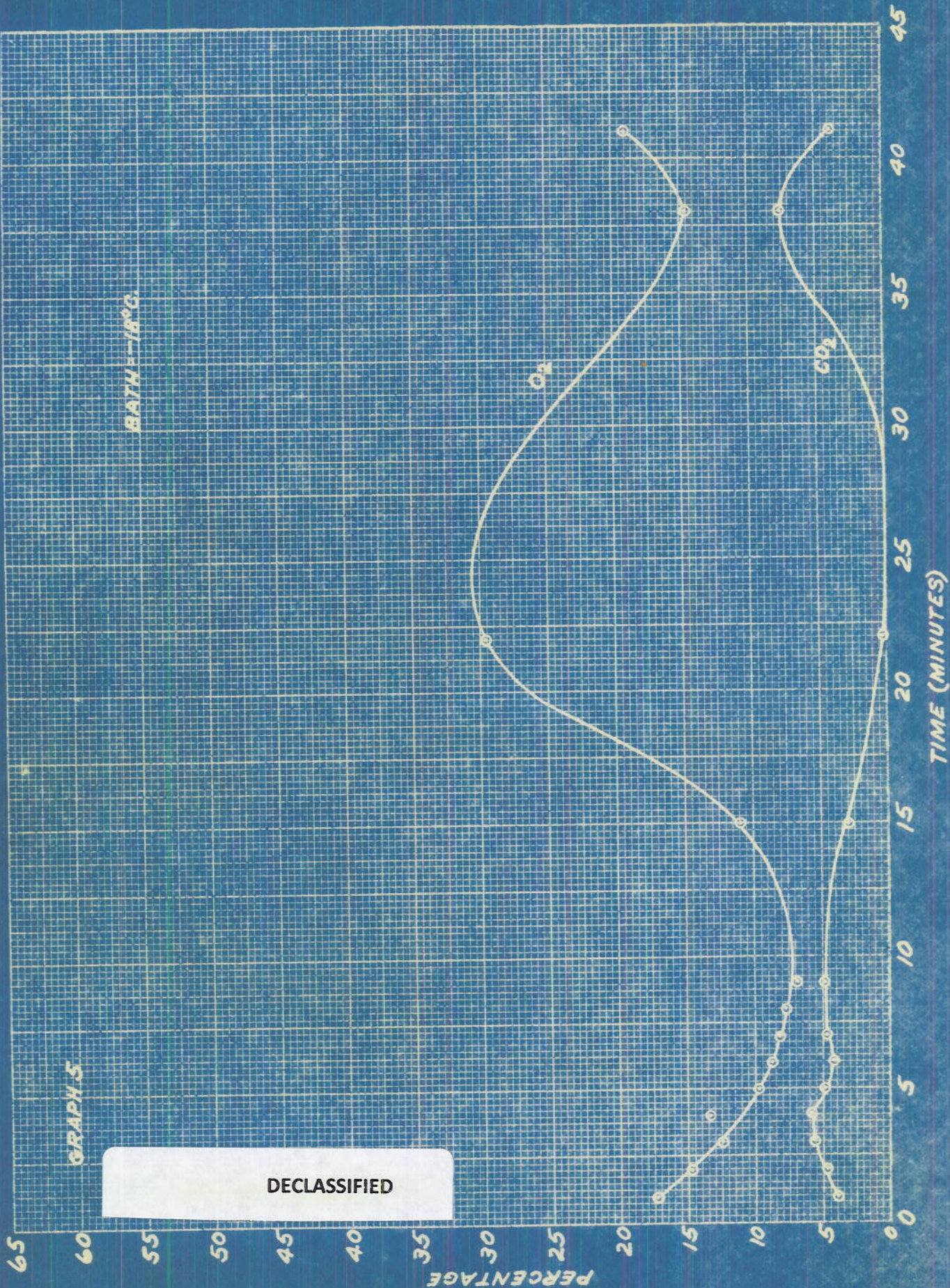
Canister type: Flat.
Date packed: October 25, 1940.
Date used: October 25, 1940.
Initial weight of material: 233.5 gms.
Final weight of material: 241.0 gms.
Weight gain of material: 7.5 gms.
Bath temperature: -18°C.
Insulation: Wrapping of asbestos paper.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	None
2	K ₂ O ₄	"
3	K ₂ O ₄	"

Throughout the first 15 minutes the observer was in difficulty; thereafter, the air supply was satisfactory until 38 minutes. After 42 minutes, resistance to breathing through the canister became excessive.

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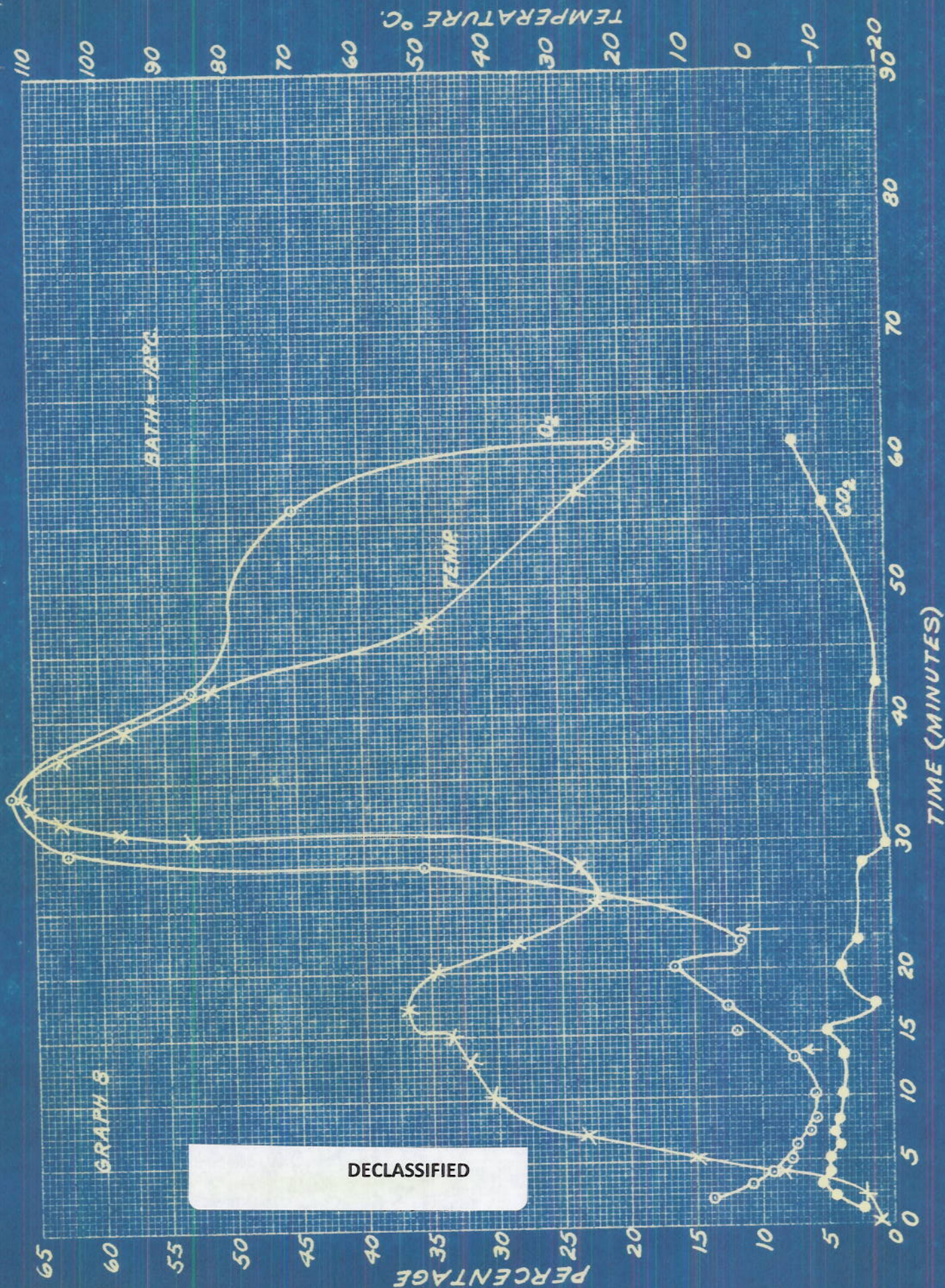
Graph 8

Canister type:	Flat.
Packed and used:	October 30, 1940.
Initial weight of material:	297.5 gms.
Final weight:	313.5
Weight gain:	16.0
Bath temperature:	-18°C.
Insulation:	None.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	None
2	K ₂ O ₄	"
3	K ₂ O ₄	"
4	K ₂ O ₄	"

The canister reaction was unsatisfactory throughout the first 22 minutes; extra breaths had to be taken in at 13 and 22 minutes. The air supply bag was insufficient to hold the oxygen generated at 32 minutes and some air was released. Satisfactory breathing continued until 61 minutes.

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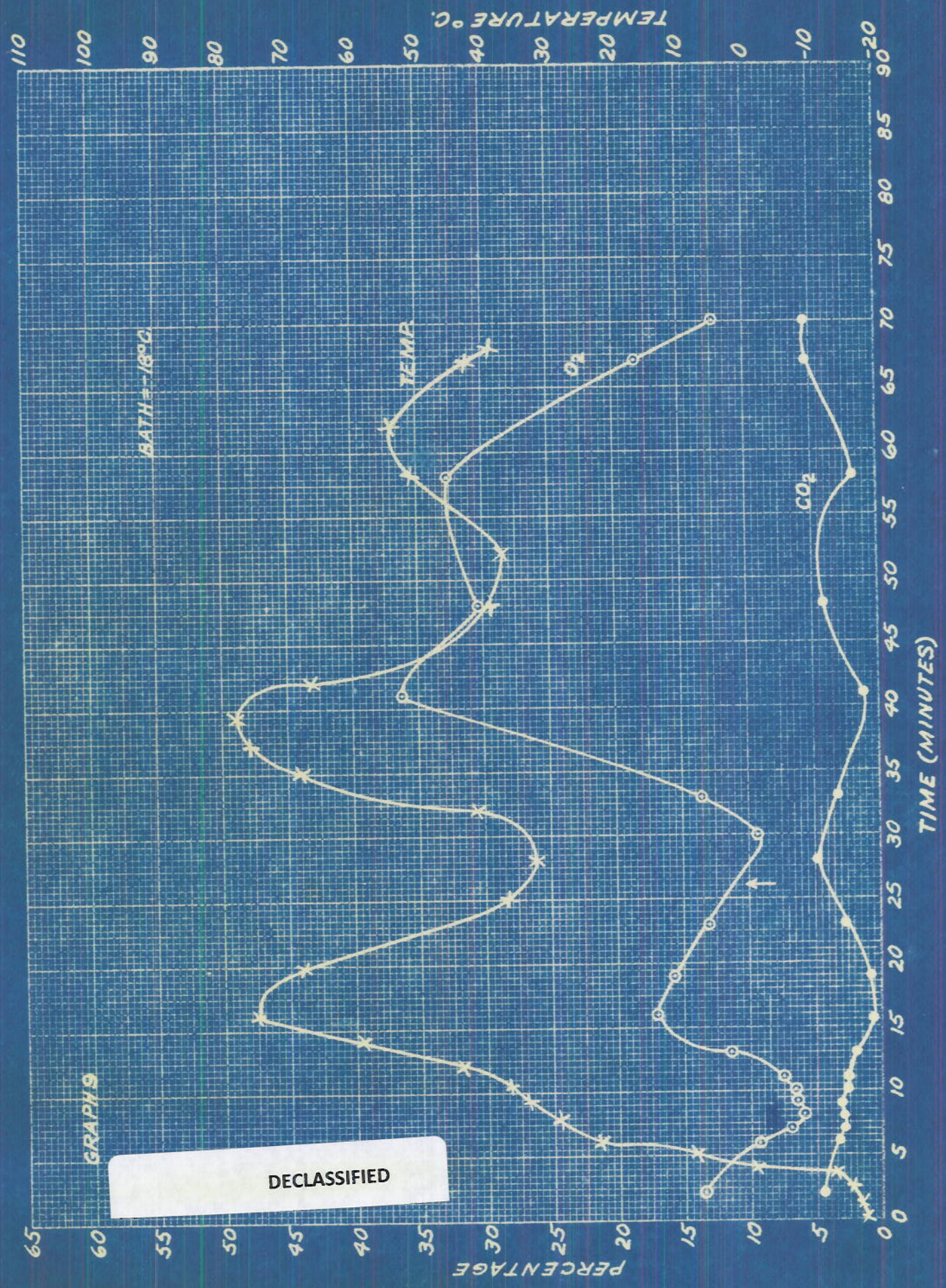
Graph 9

Canister type:	Flat.
Packed and used:	October 31, 1940.
Initial weight of material:	296.4.
Final weight of material:	327.9.
Weight gain:	31.5.
Bath temperature:	-18°C.
Insulation:	None.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	One, 3/16" diameter
2	K ₂ O ₄	One, 1/16" diameter
3	K ₂ O ₄	None.
4	K ₂ O ₄	None.

The observer experienced considerable difficulty throughout the first 12 minutes. The air supply was so low as to require an extra breath at 26 minutes. Breathing was fairly satisfactory despite continual presence of CO₂ for 65 minutes.

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Graph 10

Canister type:

Flat.

Material:

No oxygen source.

Bath temperature:

-18°C.

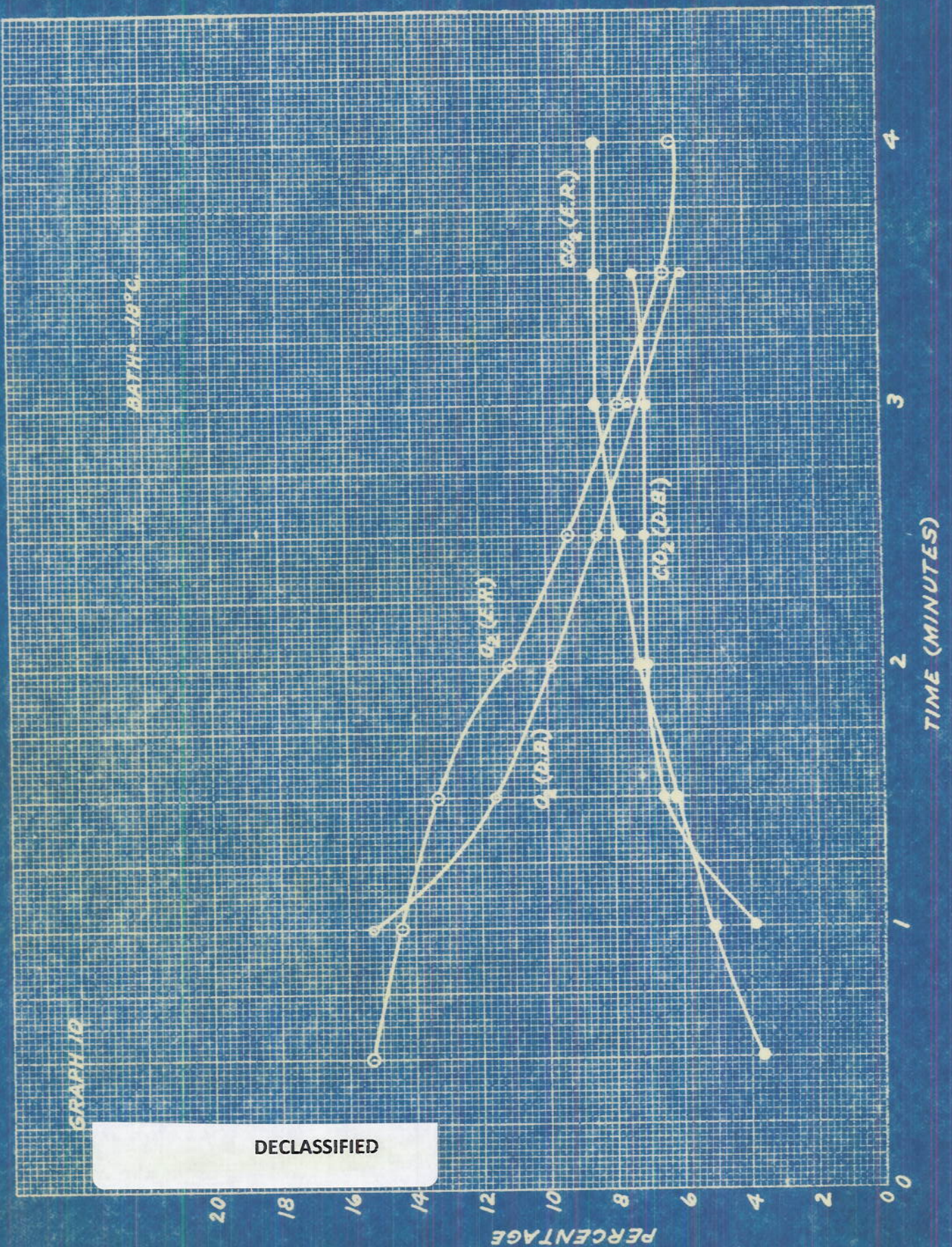
These tests were made with the usual apparatus but with empty canisters; the object of the tests was to learn how rapidly CO₂ was produced and O₂ was consumed by the observer.

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GRAPH 10

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Graph 11

Canister type: Round.
Date packed: August 1, 1940.
Date used: November 5, 1940.
Bath temperature: -18°C.
Insulation: None.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	None
2	K ₂ O ₄	"
3	K ₂ O ₄	"
4	K ₂ O ₄	"

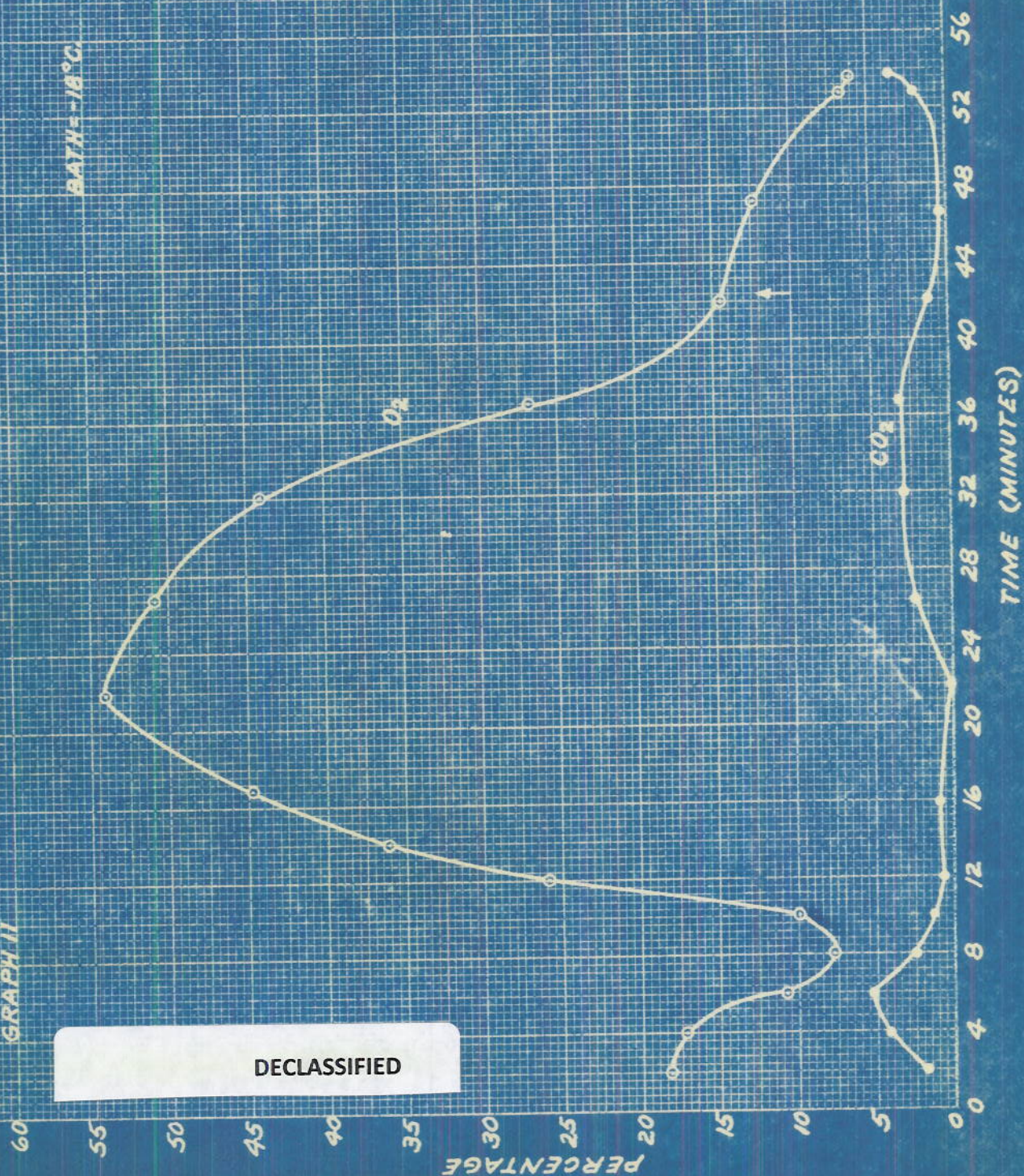
After starting slowly, the canister produced excess oxygen and required venting four times between 12 and 22 minutes. An extra breath was required at 42 minutes.

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GRAPH II

BATH = -18°C.

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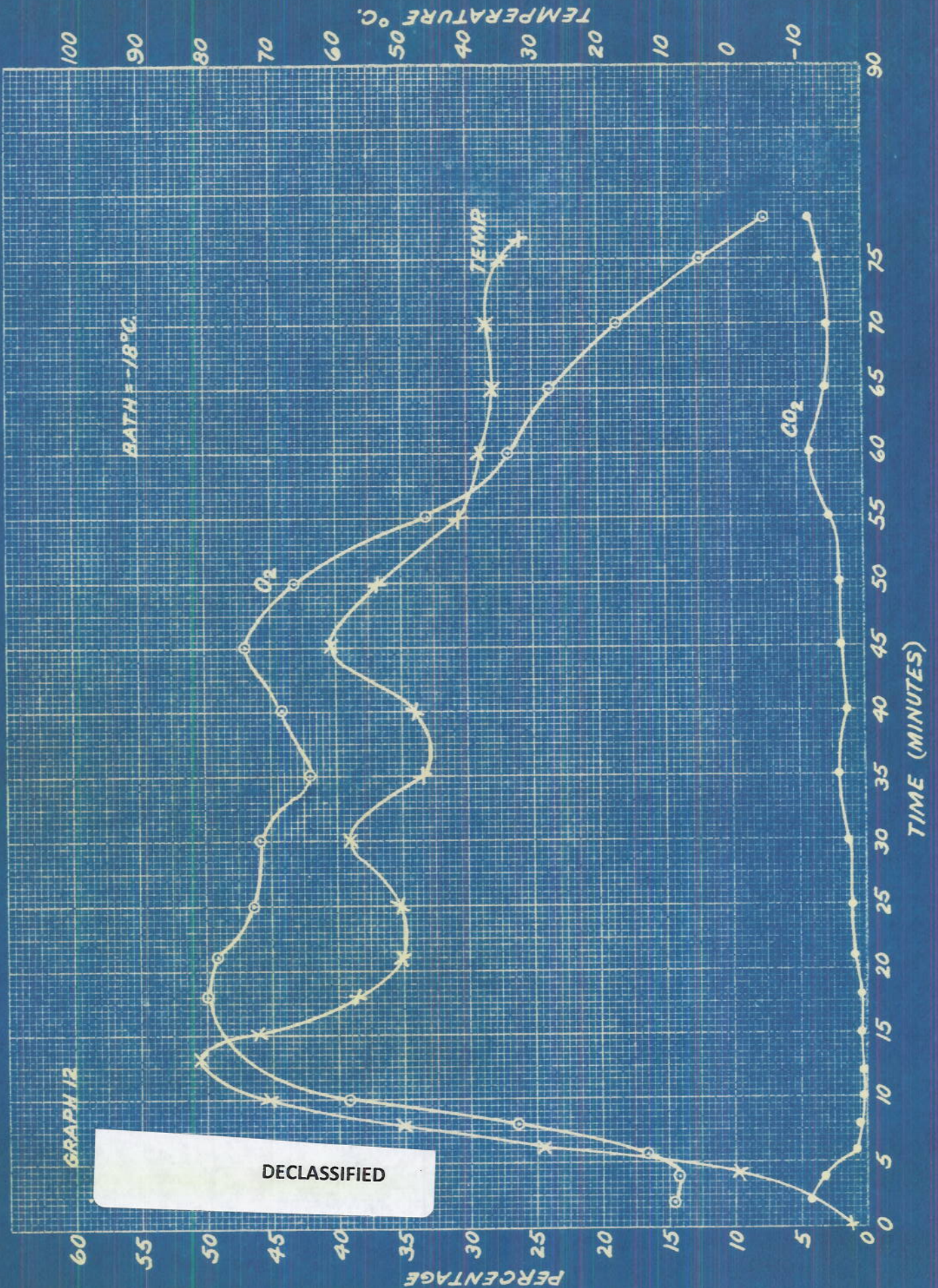
Graph 12

Canister type: Flat.
Date packed and used: November 7, 1940.
Initial weight of material: 294.5.
Final weight of material: 298.5.
Weight gained: 4.0.
Insulation: None.
Bath temperature: -18°C.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	One, 1/4" diameter.
2	K ₂ O ₄	One, 3/16" diameter.
3	K ₂ O ₄	--
4	hydrated mixed oxide	--

This canister started more quickly than previous ones at this temperature. Breathing was comfortable for 70 minutes despite the increasing concentration of carbon dioxide. During the run about 20 cc of water collected in the inlet tube below the canister. The layers nearest the bottom of the canister were most completely reacted.

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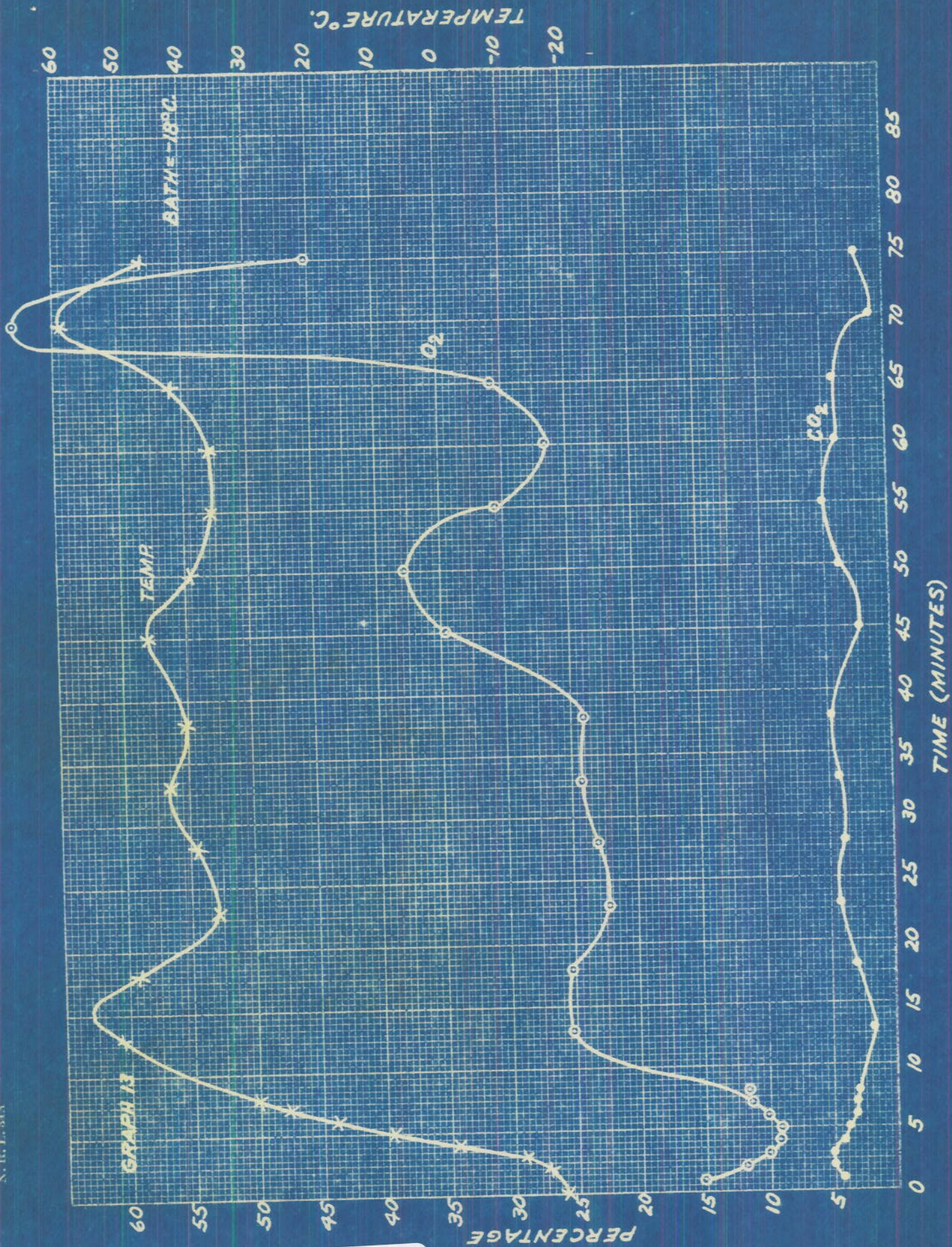
Graph 13

Canister type: Flat.
Date packed and used: November 8, 1940.
Initial weight of material: 304.5 gms.
Insulation: None.
Bath temperature: -18°C.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	$K_2O_4 + 0.1\% CuSO_4$	One, 3/8"
2	K_2O_4	One, 5/16"
3	K_2O_4	One, 3/16"
4	K_2O_4	--

The air supply became low after six minutes, but reaction increased sufficiently that no extra breath needed be taken. Sufficient water had collected in the inlet tube at 28 minutes to interfere with breathing; at 75 minutes, resistance to breathing stood at 11 inches of water. Breathing was stopped because of the effort required rather than low percentage of oxygen. Approximately 40 cc of water had collected and the portion of the bottom layer directly above the inlet was dissolved; it is probable that the two peaks in the oxygen composition were caused by splashing of water into the canister.

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PLATE #11

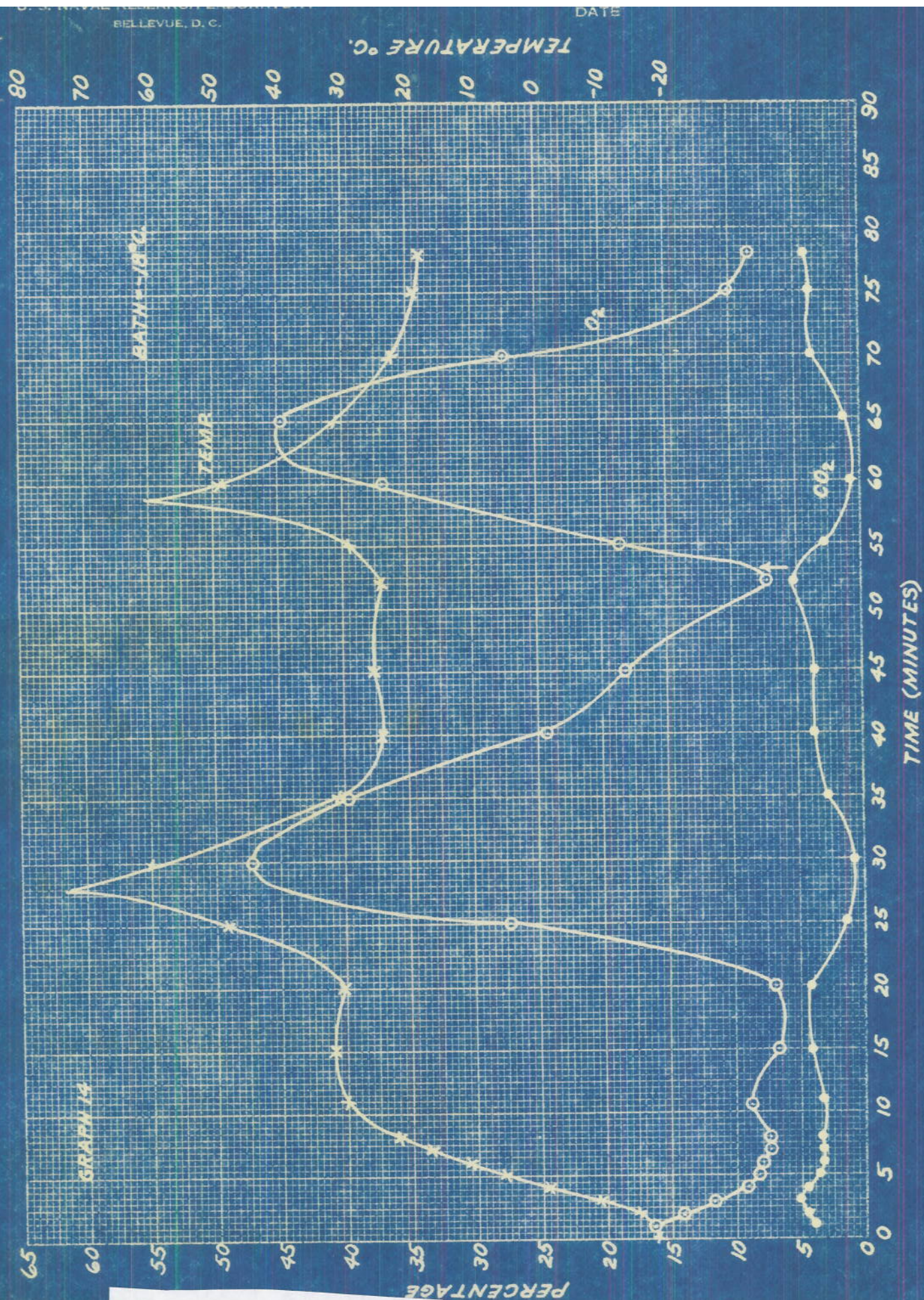
Graph 14

Canister type: Flat.
Packed and used: November 12, 1940.
Initial weight of material: 318.5 gms.
Final weight of material: 330.0 gms.
Weight increase: 11.5 gms.
Bath temperature: -18°C.
Insulation: None.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	$K_2O_4 + 0.1\% CuCO_3Cu(OH)_2$	One, 5/16"
2	K_2O_4	One, 1/4"
3	K_2O_4	One, 3/16"
4	Hydrated mixed oxide	None

The reaction started slowly and decreased again immediately after normal breathing was resumed at 11 minutes. The air supply bag had to be vented after 30 minutes, but an extra breath was required at 52 minutes. It is probable that the peaks in the oxygen composition were caused by splashing of water into the canister; a portion of the bottom layer was dissolved.

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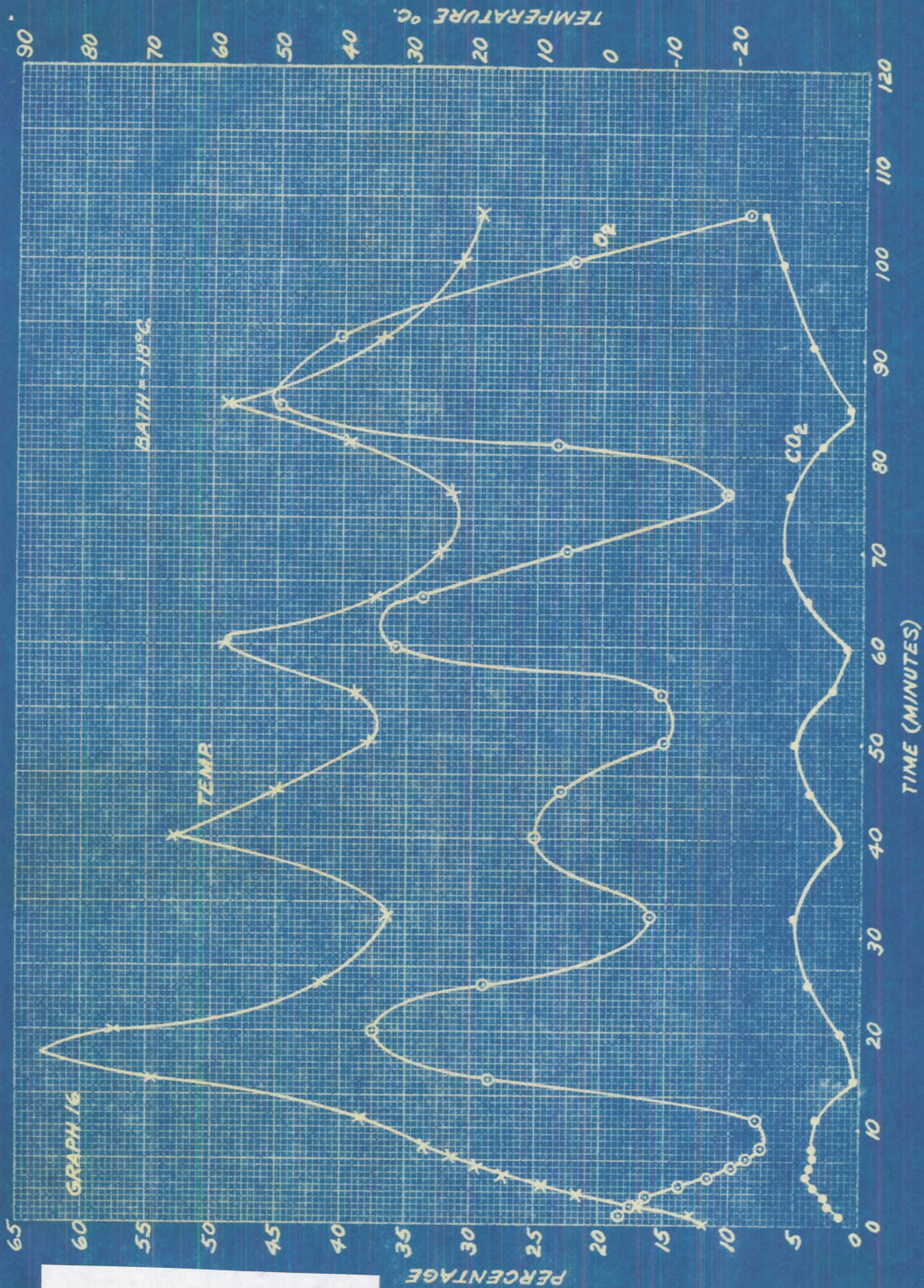
Graph 16

Canister type: Flat.
Packed and used: November 14, 1940.
Initial material weight: 282.0 gms.
Final material weight: 297.5 gms.
Weight gained: 15.5 gms.
Insulation: Wrapping of asbestos paper.
Bath temperature: -18°C.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K_2O_4 + 1 gm. LiOH	One, 3/8"
2	K_2O_4 + 1 gm. LiOH	One, 5/16"
3	K_2O_4 + 1 gm. LiOH	One, 3/16"
4	K_2O_4	--

The peaks in the oxygen composition during the earlier portion of the test conformed with the nature of breathing, i.e., rapid or natural. Rapid breathing during the first 10 minutes accelerated the reaction to the extent that venting was necessary at 15 to 20 minutes; normal breathing was employed at 15 minutes, but at 32 minutes it was necessary to return to rapid breathing. The peaks in the latter portion of the oxygen curve may again be accounted for by the splashing of water condensed in the inlet tube.

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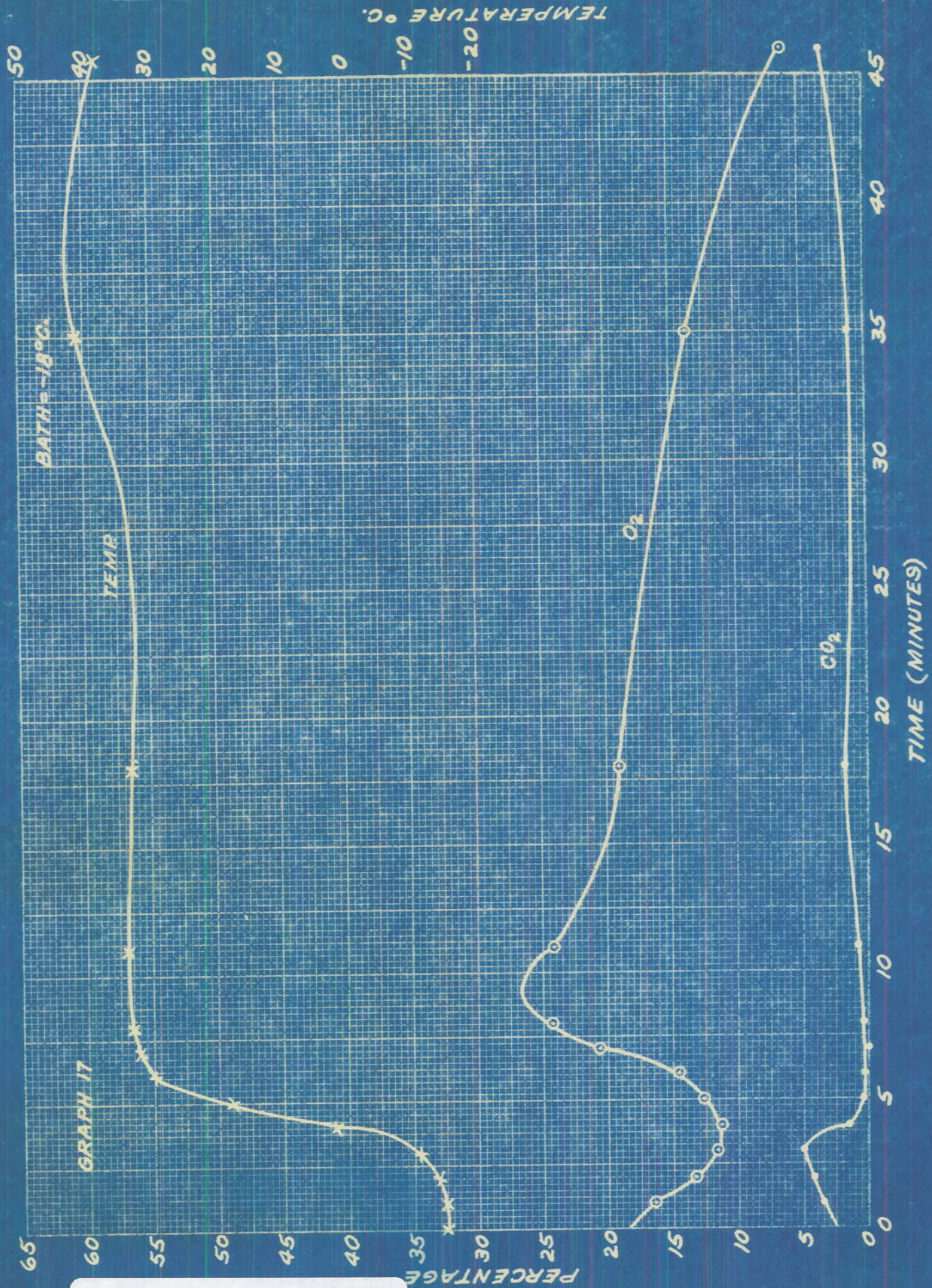
Graph 17

Canister type: Flat.
Packed and used: November 15, 1940.
Initial material weight: 341.7 gms.
Final material weight: 379.2 gms.
Weight gain: 27.5 gms.
Insulation: Asbestos paper.
Bath temperature: -18°C.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	Hydrated mixed oxide.	One, 5/16",
2	" " "	One, 3/16"
3	" " "	---
4	" " "	---

No discomfort was experienced at any time during the test by reason of excessive carbon dioxide, but the oxygen percentage became dangerously low after about 40 minutes. This was the only canister studied in this report in which the low percentage of oxygen was not accompanied by a relatively high percentage of carbon dioxide; it is significant that this canister was filled with hydrated mixed oxide which will absorb more carbon dioxide per volume of oxygen liberated than will K_2O_4 .

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Graph 18

Canister type: Flat.
Packed and used: November 18, 1940.
Initial weight of material: 279.0 gms.
Insulation: Asbestos paper.
Bath temperature: -18°C.

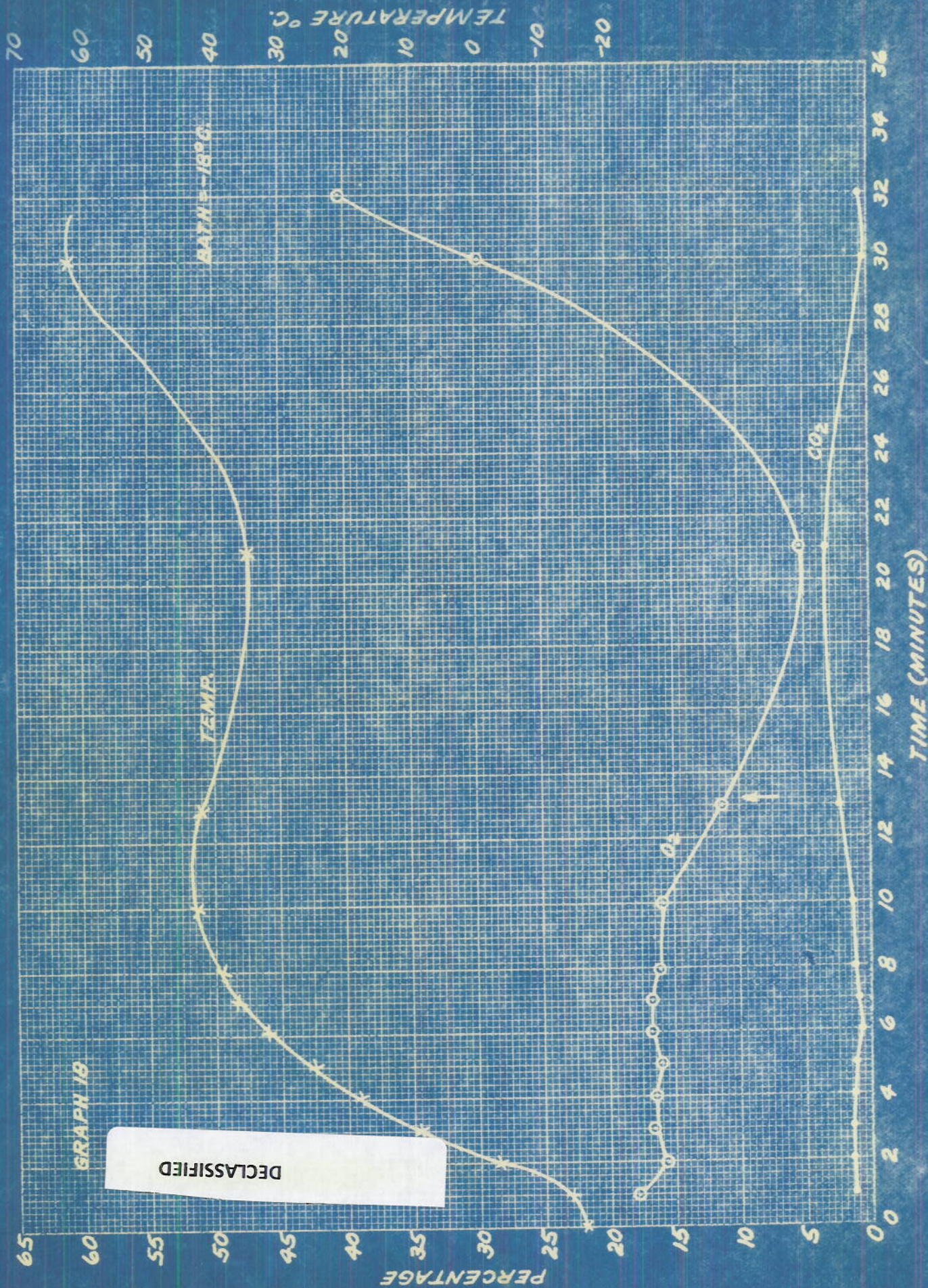
<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	One, 3/8"
2	K ₂ O ₄ + 4 gms LiOH	--
3	K ₂ O ₄	--
4	K ₂ O ₄	--

Considerable difficulty was experienced in the first 24 minutes and an extra breath was required at 13 minutes. The canister reaction was evidently proceeding satisfactorily at 32 minutes when the canister became plugged.

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GRAPH 19

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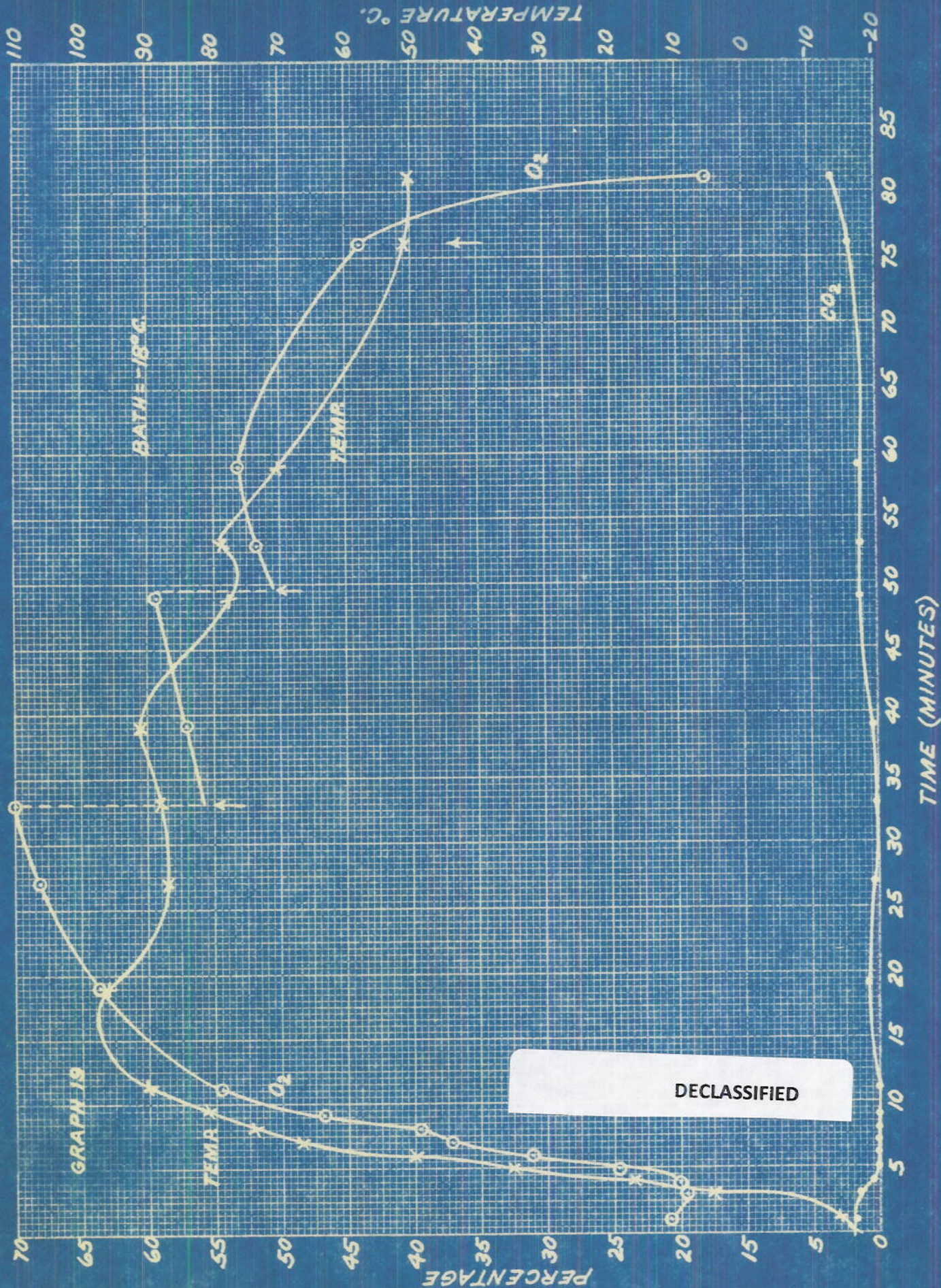
Graph 19

Canister type: Round.
Packed and used: November 27, 1940.
Insulation: Woolen bag.
Bath temperature: -18°C.
Material weight: 280 gms.
Weight gained: 41.5 gms.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	Two, 3/8"
2	"	Two, 5/16"
3	"	--
4	"	--

This canister started more quickly than any previous one at this temperature and oxygen was vented about 7 times between 6 and 27 minutes. Observers were changed at 33 and 49 minutes; all three observers experienced "sleepy" extremities due probably to excessively rapid breathing rates. The air supply bag became empty at 76 minutes and an extra breath was taken in. The material in the canister was almost completely reacted.

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Graph 20

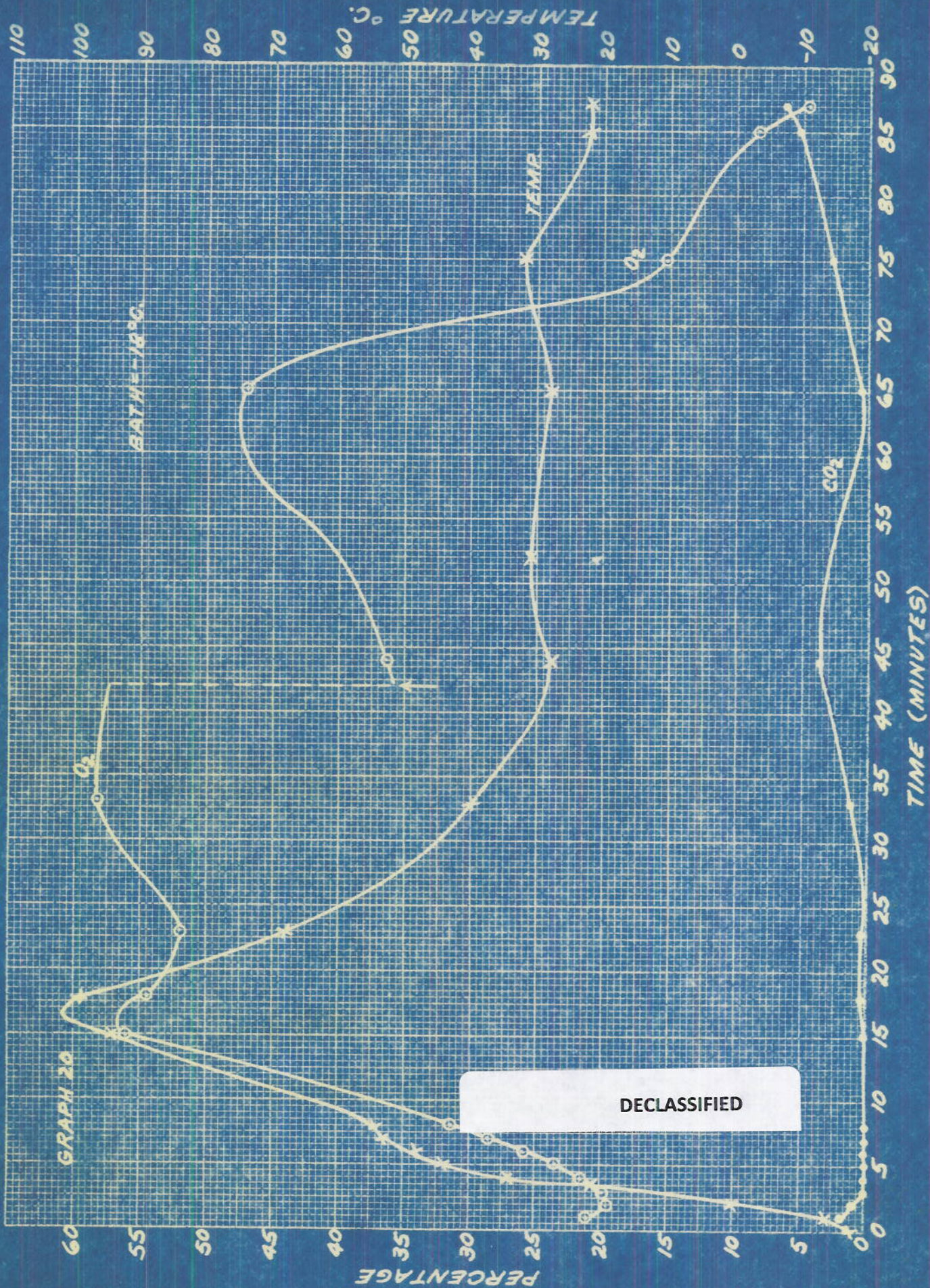
Canister type: Round.
Packed and used: November 28, 1940.
Insulation: None.
Bath temperature: -18°C.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	Two, 3/8"
2	"	Two, 5/16"
3	"	--
4	"	--

The starting rate for this canister was very similar to that of the previous test (Graph 19). Attention should be called to the fact that the temperature fell to a much lower level in this case; the absence of insulation on this canister and/or normal breathing after 10 minutes may satisfactorily account for this difference. Observers were changed at 10 and 42 minutes.

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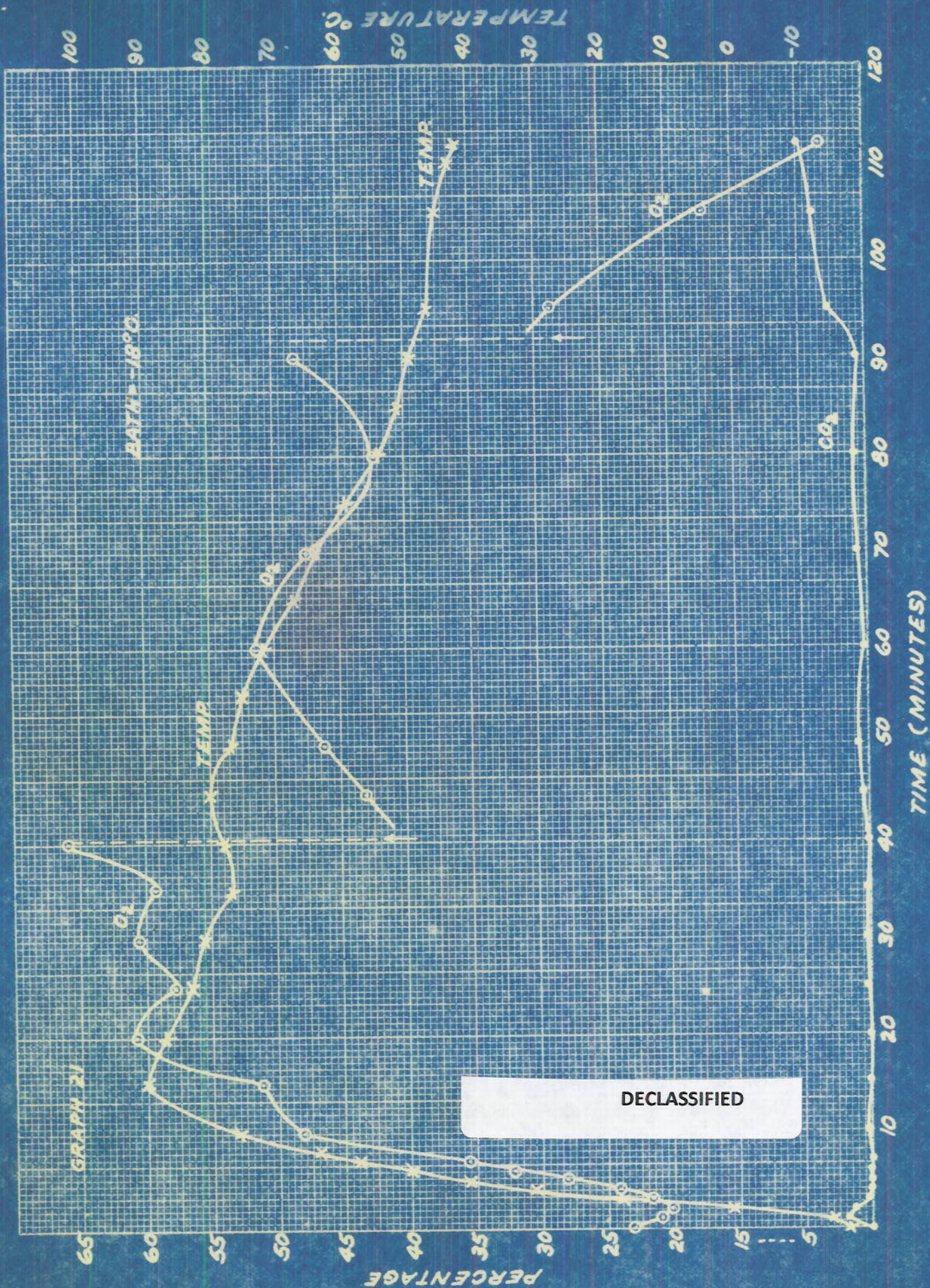
Graph 21

Canister type: Round.
Date packed and used: November 29, 1940.
Insulation: Woolen bag.
Bath temperature: -18°C.
Initial material weight: 281.5 gms.
Final Material weight: 314.5 gms.
Weight increase: 33.0 gms.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K ₂ O ₄	Two, 3/8"
2	K ₂ O ₄	Two, 1/4"
3	K ₂ O ₄	
4	K ₂ O ₄	

Normal breathing was resumed after 3 minutes and the canister reacted satisfactorily until about 95 minutes. Observers were changed at 40 and 92 minutes.

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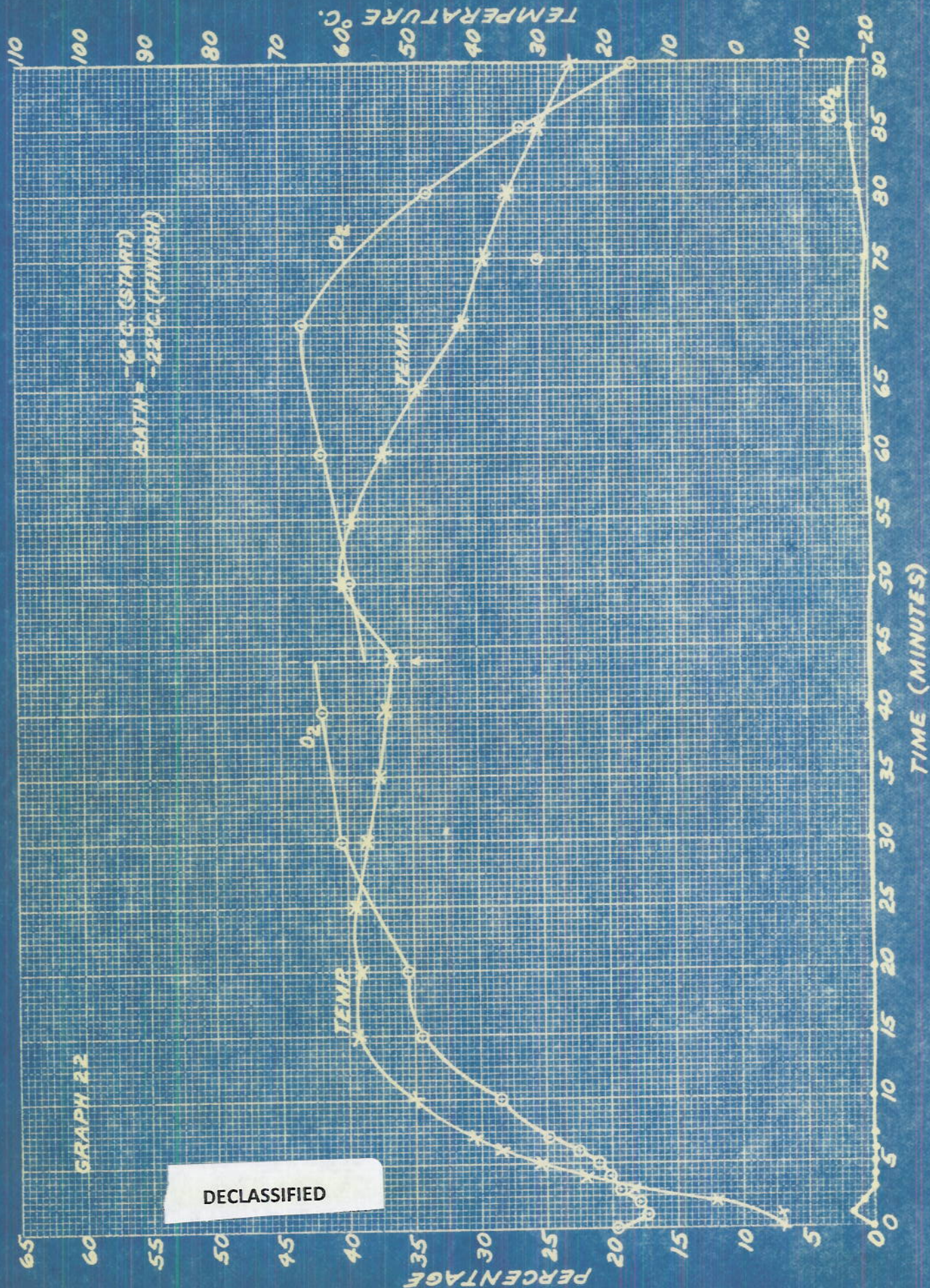
Graph 22

Canister type: Round.
Packed and used: December 3, 1940.
Insulation: None.
Bath temperature: -6°C. to -22°C.
Initial weight of material: 264.0 gms.
Weight increase: 60 gms.

<u>Layer</u>	<u>Material</u>	<u>By-Passes</u>
1	K_2O_4	Two, $3/8''$
2	K_2O_4	Two, $1/4''$
3	K_2O_4	
4	K_2O_4	

The initial temperature of the bath and canister was -6°C. which corresponds to the temperature at 10,000 feet elevation; the bath was gradually cooled until at the end of the test its temperature was -22°C. which corresponds to that found at 20,000 feet elevation. Normal breathing was resumed after the first few minutes and observers were changed at 44 minutes.

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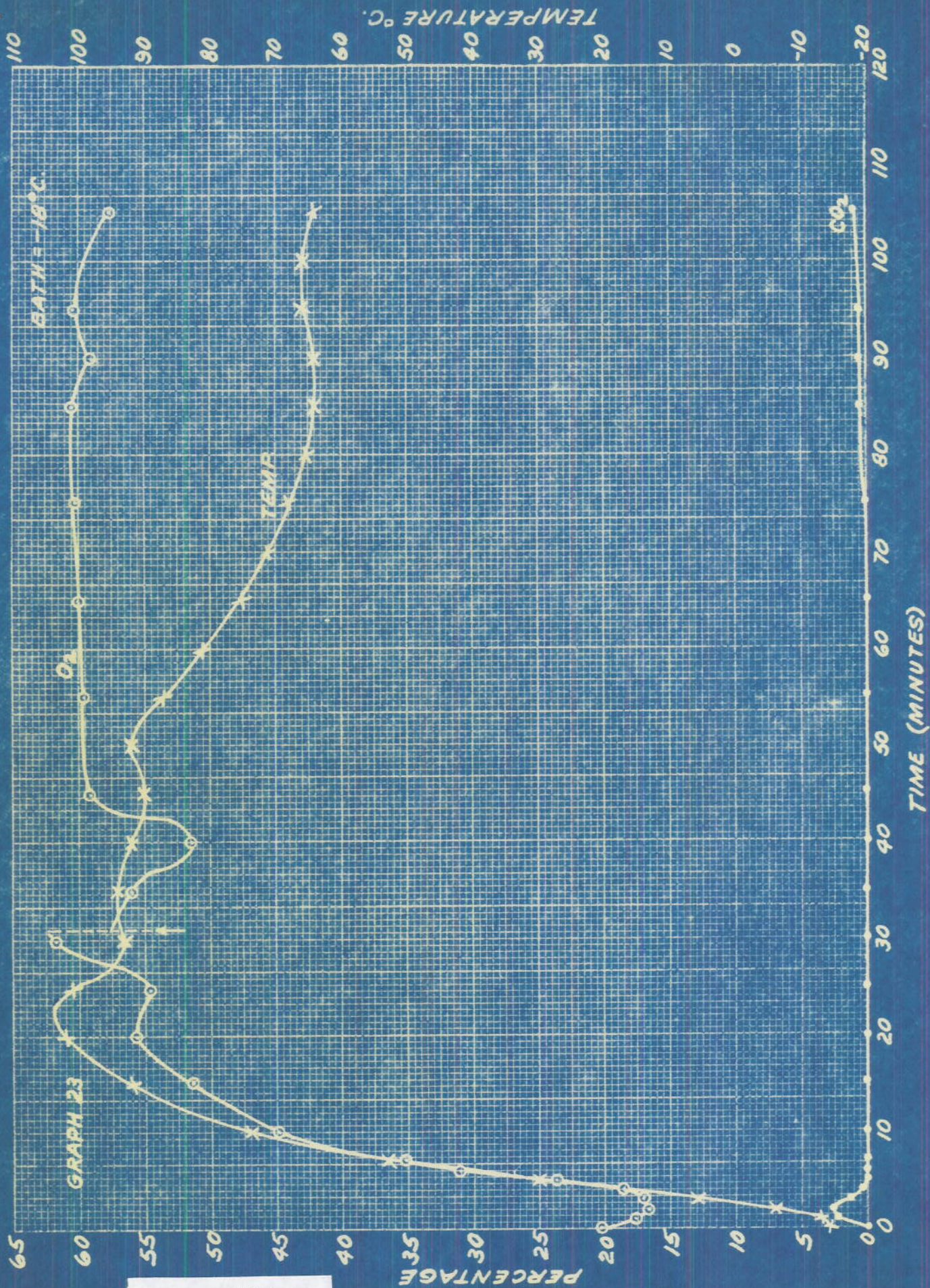
Graph 23

Canister type: Round.
Packed and used: December 20, 1940.
Insulation: Woolen bag.
Bath temperature: -18°C.
Material used: K₂O₄, 225 cc. available oxygen per gram.

<u>Layer</u>	<u>By-Passes</u>	<u>CO₂ absorbed cc/gm</u>	<u>O₂ evolved cc/gm.</u>
1	--	173.6	214.7
2	--	116.4	169.1
3	--	73.6	123.8
4	--	87.0	96.9

Only one breath was used to start this canister and normal breathing was resumed after 6 minutes; the air supply bag was vented 7 times between 6 and 10 minutes. Observers were changed at 31 minutes. The air supply became low at 105 minutes and breathing had to be discontinued. A total of 24.5 liters of CO₂ were absorbed, an average of 230 cc/minute. A total of 40.0 liters of O₂ had been evolved, an average of 381 cc/minute; however, this latter figure is higher than that actually used by the observer because an unknown quantity of O₂ was vented during the test.

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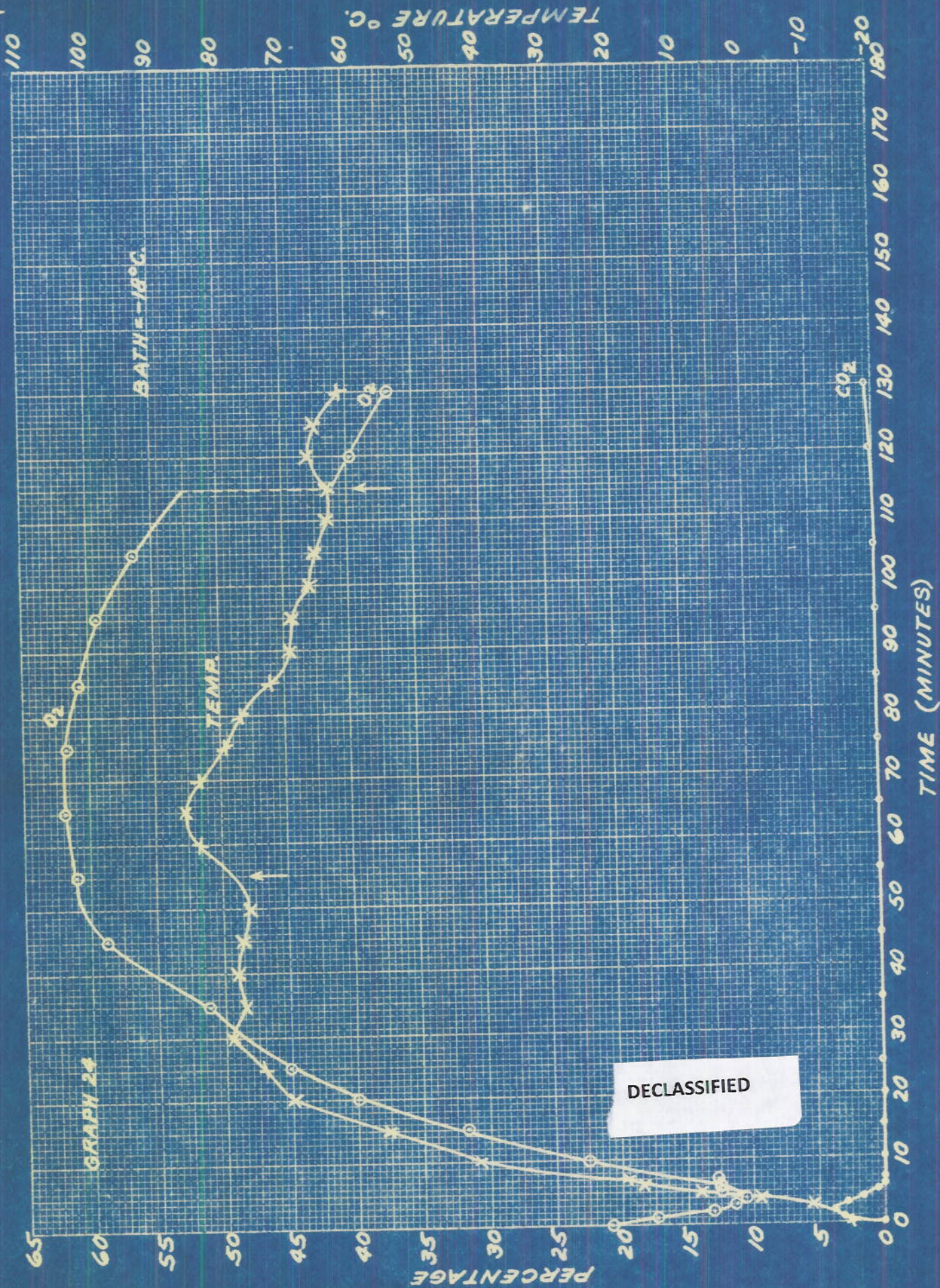
Graph 24

Canister type: Round.
Packed and used: December 31, 1940.
Initial weight material: 296 gms.
Weight increase: 15 gms.
Insulation: Woolen bag.
Bath temperature: -18°C.
Material used: K₂O₄, 225 cc available O₂ per gram.

<u>Layer</u>	<u>By-Passes</u>	<u>CO₂ absorbed cc/gm.</u>	<u>O₂ evolved cc/gm.</u>
1	Two, 3/8"	155.5	177.4
2	Two, 5/16"	166.2	191.3
3	---	132.5	163.0
4	---	69.4	113.9

Normal breathing was employed throughout this test and observers were changed at 55 and 115 minutes. Approximately 73% of the total available O₂ was evolved during this test.

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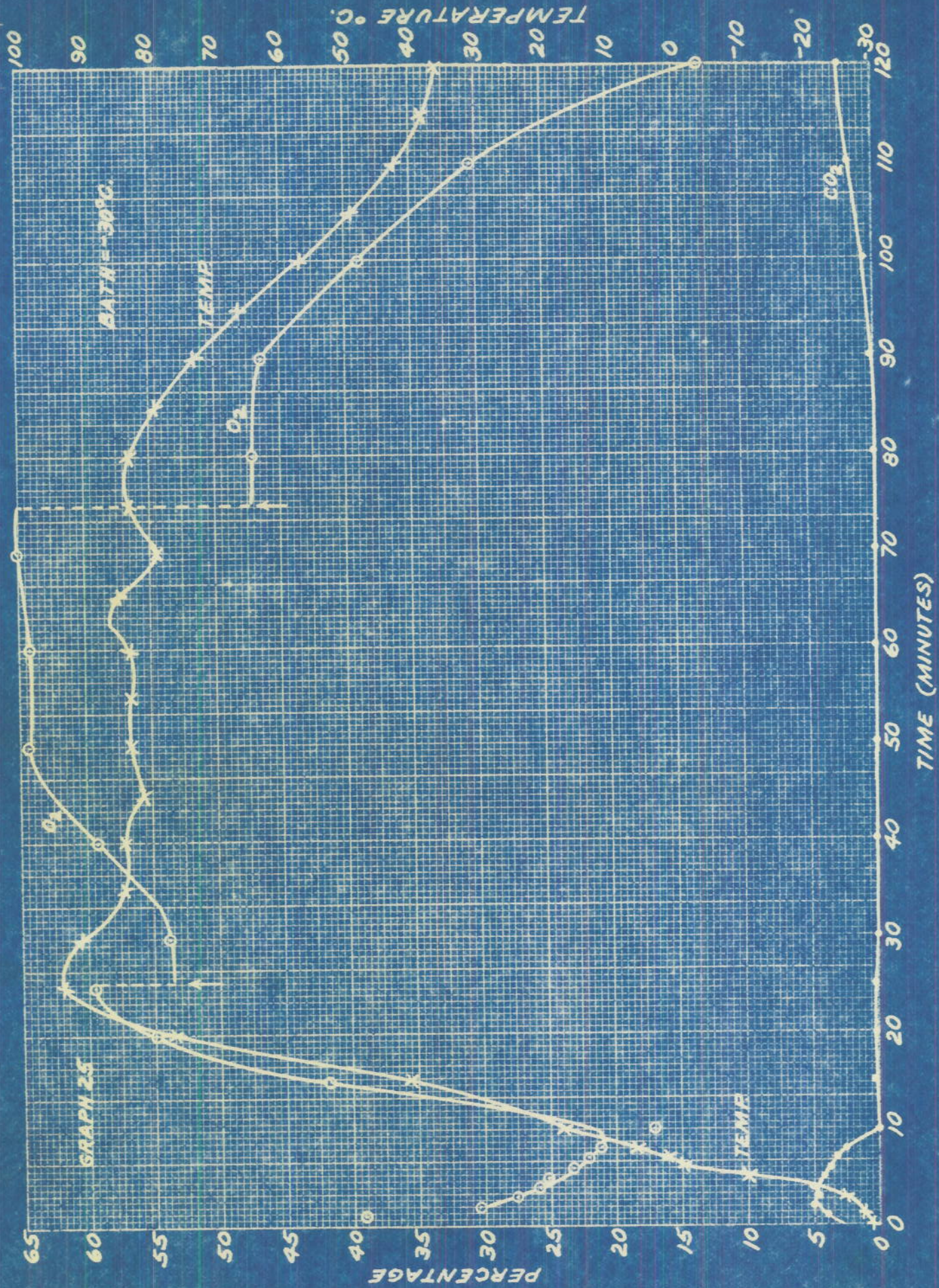
Graph 25

Canister Type: Round.
Packed and used: January 3, 1941.
Initial weight material: 283.5 gms.
Weight increase: 15.9 gms.
Insulation: Woolen bag.
Bath temperature: -30°C.
Material used: K_2O_4 , 225 cc. available O_2 per gram.

<u>Layer</u>	<u>By-Passes</u>	<u>cc. CO_2 Absorbed per gm.</u>	<u>cc O_2 evolved per gm.</u>
1	Two, 1/4"	158.5	216.3
2	Two, 3/16"	150.0	184.0
3	Two, 1/8"	113.5	145.5
4	--	107.3	123.0

The air supply bag was partially filled with oxygen before starting the test at this low temperature. Normal breathing was employed throughout the test. Observers were changed at 25 and 75 minutes. Excess air was vented at 20 minutes.

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PLATE #22

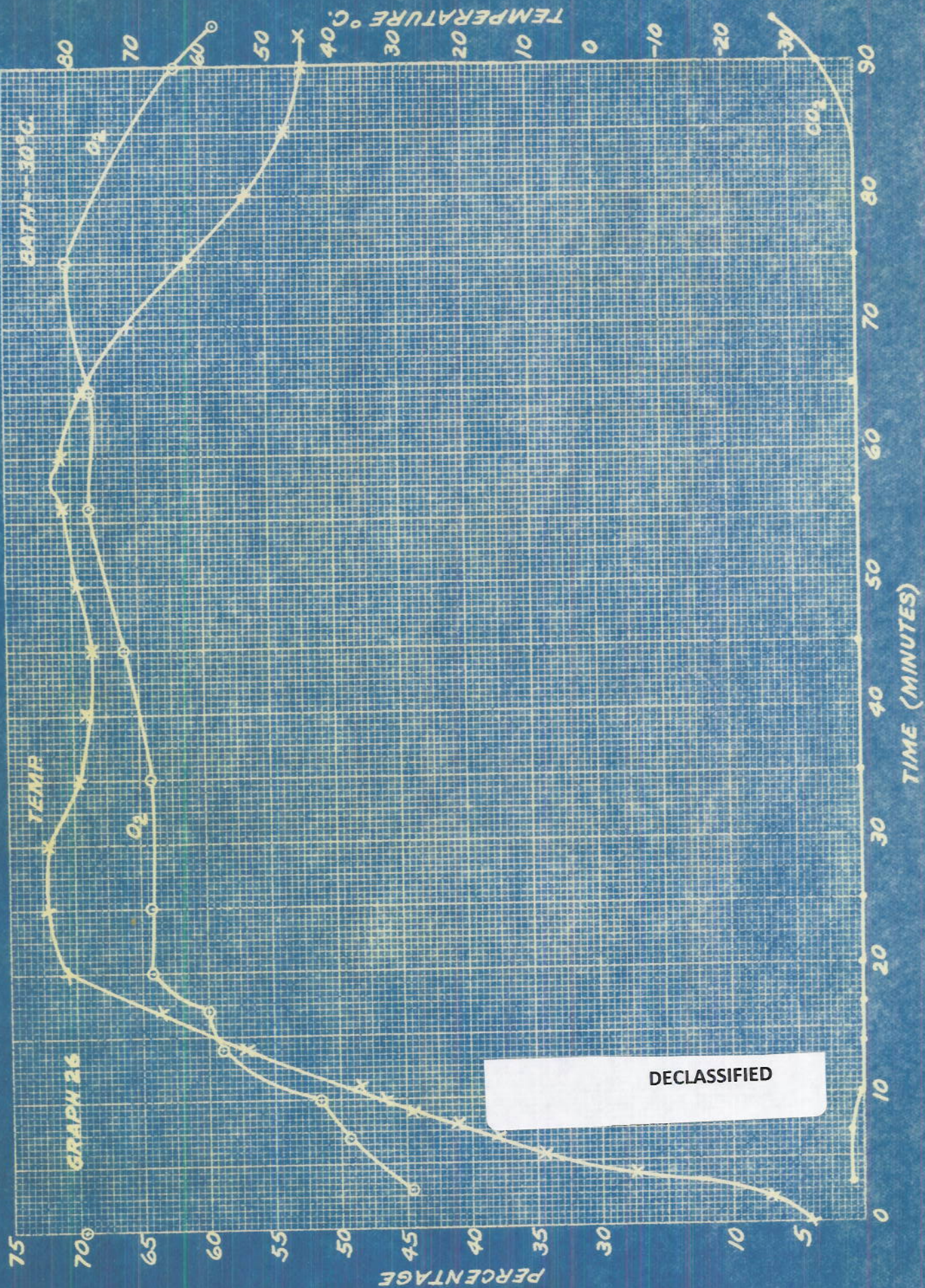
Graph 26

Canister type: Round.
Packed and used: January 7, 1941.
Initial weight material: 280.8 gms.
Weight increase: 18.1 gms.
Insulation: Woolen bag.
Bath temperature: -30°C.
Material used: K₂O₄, available O₂, 225 cc. per gram.

<u>Layer</u>	<u>By-Passes</u>	<u>CO₂ absorbed cc/gm.</u>	<u>O₂ evolved cc/gm.</u>
1	Two, 3/8"	157.8	216.7
2	Two, 1/4"	158.4	183.8
3	Two, 1/8"	119.5	143.7
4	---	106.6	122.2

This canister was started in the same manner as the previous one (Graph 25). The bag was vented three times at 17 minutes. A total of 36.5 liters of CO₂ were absorbed and 46.8 liters of O₂ evolved. A total of 21,000 foot-pounds of work were done during this test. This amount of work probably accounts for the high value of 500 cc. of O₂ consumed per minute.

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Graph 26.5

Canister type: Round.

Packed and used: January 8, 1941.

Initial weight material: 286.1 gms.

Weight increase: None.

Insulation: Woolen bag.

Bath temperature: 2°C.

Material used: K_2O_4 ; available O_2 = 225 cc/gm.

<u>Layer</u>	<u>By-Passes</u>	<u>CO₂ absorbed (cc/gram)</u>	<u>O₂ evolved (cc/gram)</u>
1	Two, 1/4"	147.4	195.0
2	Two, 3/16"	136.9	187.8
3	Two, 1/8"	129.2	206.3
4	--	126.4	217.9

For this and the following work tests, the breathing apparatus was the same as that used previously except that several feet of rubber tubing were eliminated. The bag and canister were harnessed on the observer's chest and most of the distance from the face-piece to the canister was occupied by the brass adapter and valves.

In this particular test the observer walked at the rate of 3 miles per hour, except when samples were being taken; the total distance walked was 1.7 miles. As the reaction neared completion, some clogging was noted, due probably to the unusually high temperature attained by the canister (no thermometer was used). The bag was vented six times from 12 to 20 minutes.

A total of 38.5 liters of CO_2 were absorbed, an average of 875 cc/minute. A total of 57.7 liters of O_2 were evolved, an average of 1310 cc/minute; a reasonable value for the quantity of O_2 lost by venting is 12 liters, leaving an average value of O_2 consumed by the observer of about 1000 cc/minute.

The data in the following table are not shown graphically since only four samples were taken for analysis:

<u>Time</u>	<u>Minutes</u>	<u>Sample</u>	<u>%CO₂</u>	<u>%O₂</u>	<u>Remarks</u>
10:00	0	--	--	--	Start
10:10	10	1	0.0	50.5	--
10:12	12	--	--	--	Started venting bag.
10:20	20	2	0.0	60.8	Have vented 6 times.
10:30	30	3	0.0	80.6	--
10:40	40	4	1.6	53.7	Canister clogging.
10:44	44	--	--	--	Stopped.

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Graph 27

Canister type: Round.
Packed and used: January 9, 1941.
Initial material weight: 292.2 gms.
Weight increase: 0.6 gms.
Outside temperature: 0°C.
Initial canister temperature: -18°C.
Insulation: None.

<u>Layers</u>	<u>By-Passes</u>	<u>CO₂ absorbed cc/gm.</u>	<u>O₂ evolved cc/gm.</u>
1	Two, 1/4"	98.7	171.1
2	Two, 3/16"	78.3	92.9
3	Two, 1/8"	60.9	70.5
4	--	62.4	54.2

The observer walked continuously at 3 miles per hour, and the breathing rate was normal for the amount of work being done. Throughout this test the valves did not function properly and much air was exhaled directly into the bag instead of through the canister; this probably accounts for the poor efficiency of the canister. The average oxygen consumption was 940 cc/minute and the average CO₂ was 800 cc/minute.

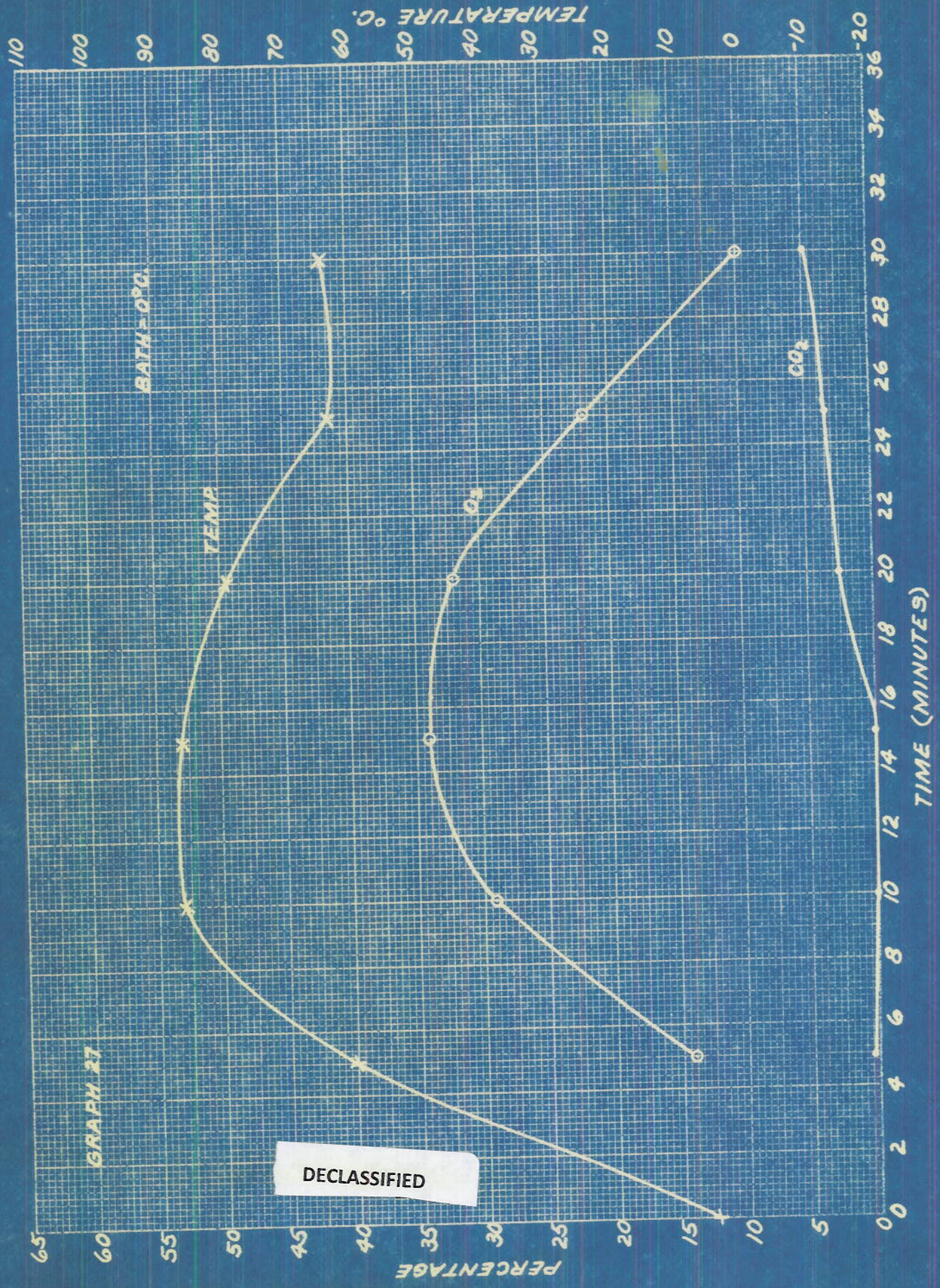
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GRAPH 27

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PLATE #24



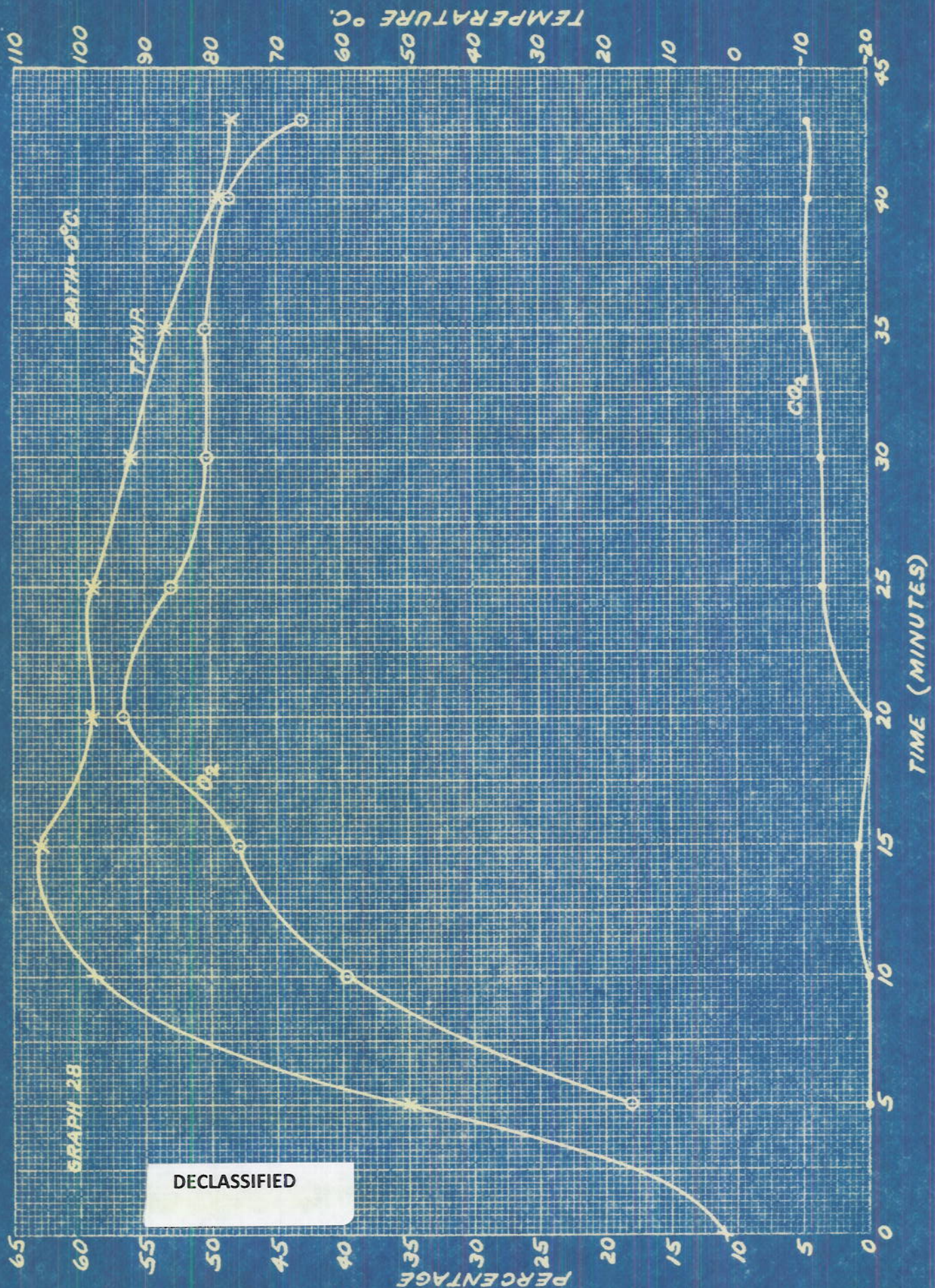
Graph 28

Canister type: Round.
Packed and used: January 13, 1941.
Initial material weight: 281.6 gms.
Weight increase: 1.1 gms.
Outside temperature: 0°C.
Initial canister temperature: -18°C.
Insulation: Woolen bag.

<u>Layer</u>	<u>By-Passes</u>	<u>CO₂ absorbed cc/gm.</u>	<u>O₂ evolved cc/gm.</u>
1	Two, 1/4"	121.5	179.0
2	Two, 3/16"	94.5	127.0
3	Two, 1/8"	99.3	143.6
4	--	114.5	167.6

The operating conditions for this canister were the same as those for the previous test (Graph 27). The bag was vented after 10 and 15 minutes. The material was found at the end of the test to have evolved 70% of its available oxygen.

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Graph 29

Canister type: Round.
Packed and used: January 14, 1941.
Initial material weight: 273.5
Weight increase: None
Outside temperature: -3°C.
Initial canister temperature: -18°C.
Insulation: Woolen jacket.
Material: K₂O₄, available oxygen 221 cc/gm.

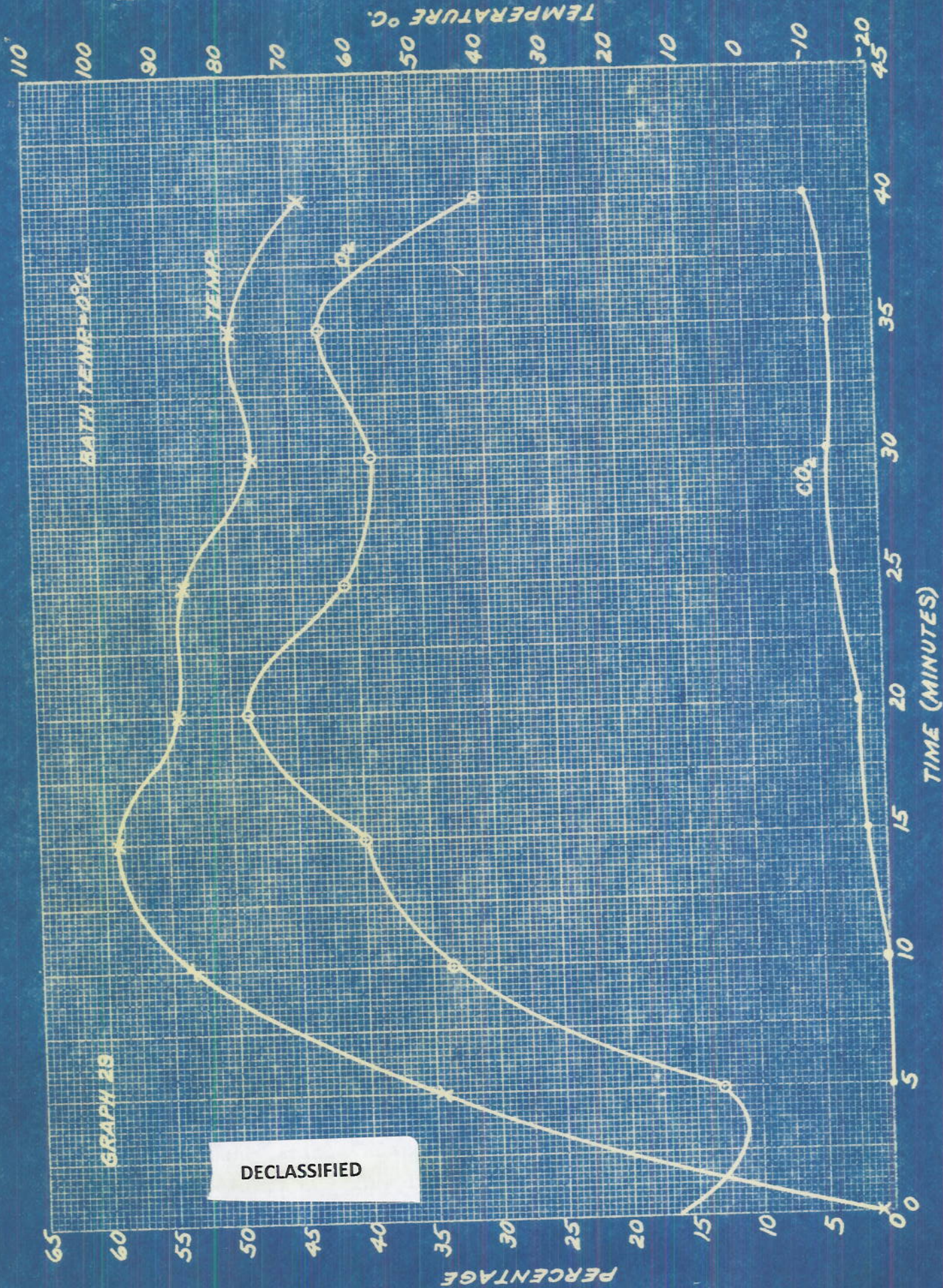
<u>Layer</u>	<u>By-Passes</u>	<u>CO₂ absorbed cc/gm.</u>	<u>O₂ evolved cc/gm.</u>
1	Two, 1/4"	129.6	196.0
2	Two, 3/16"	118.0	159.8
3	Two, 1/8"	83.0	111.7
4	--	80.0	120.8

Operating conditions for this canister were the same as the two previous ones. Considerable difficulty was experienced in the early minutes of the test. Bag was vented three times at 8 to 10 minutes. Resistance to breathing became high after 30 minutes; after the test, it was found that this resistance was caused by the plugging of the top layer of material. The total evolution of oxygen was 64% of the theoretical.

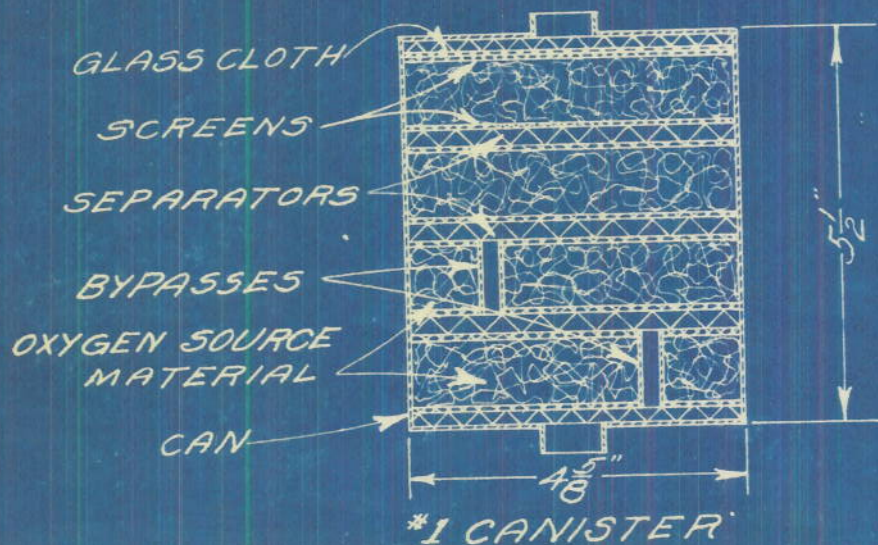
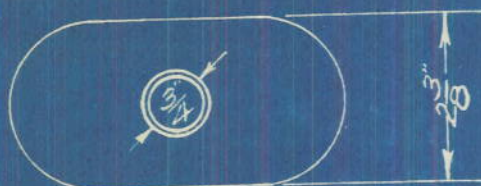
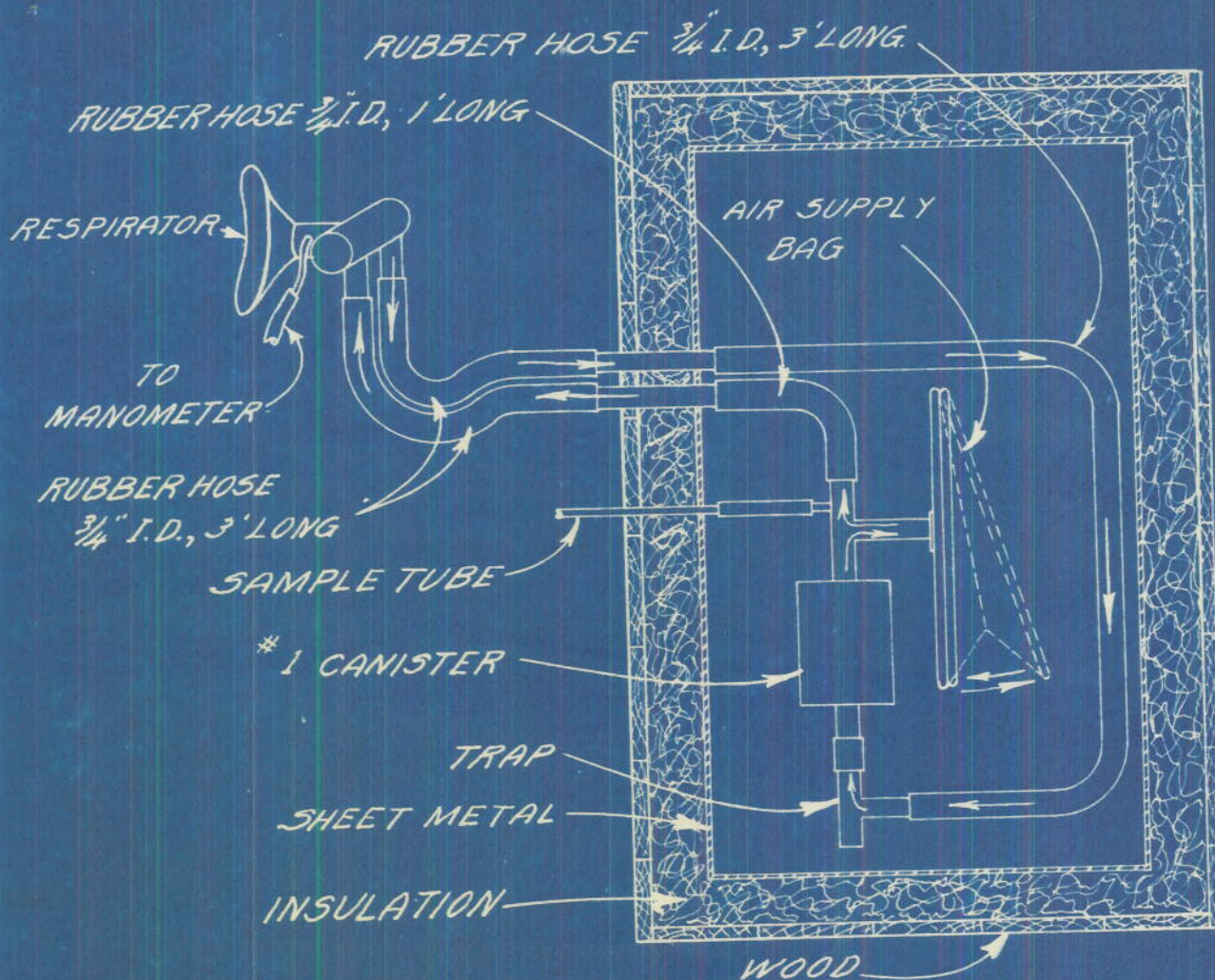
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GRAPH 29

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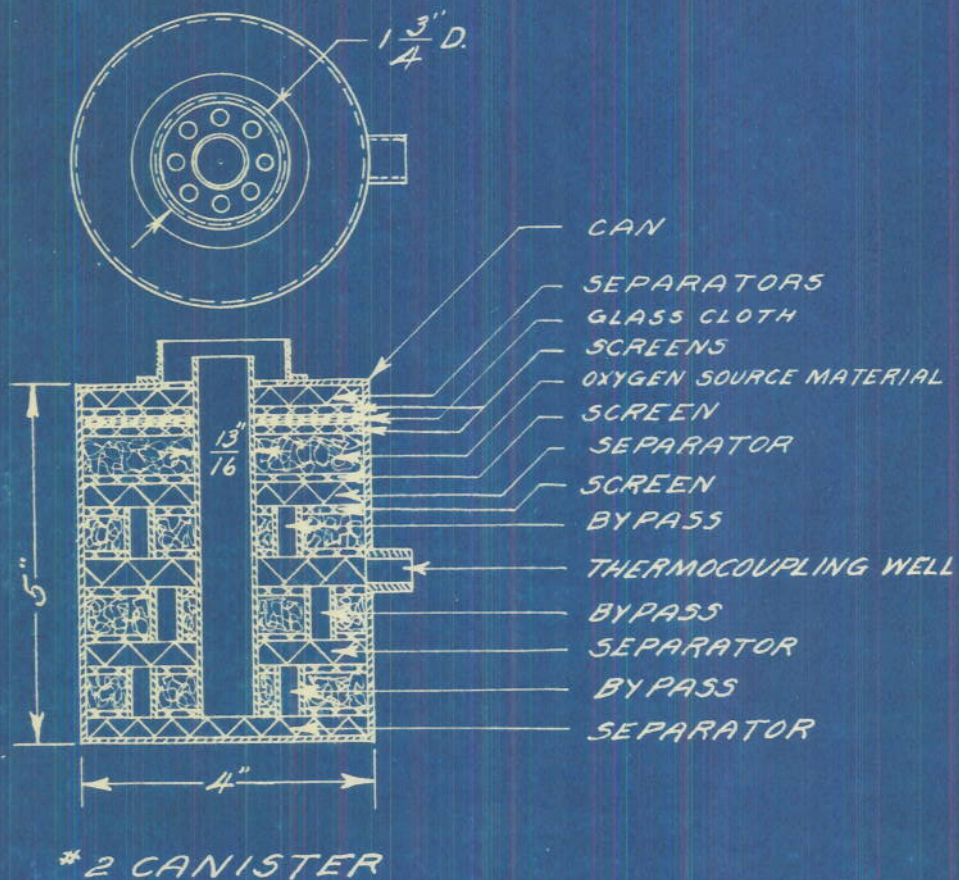
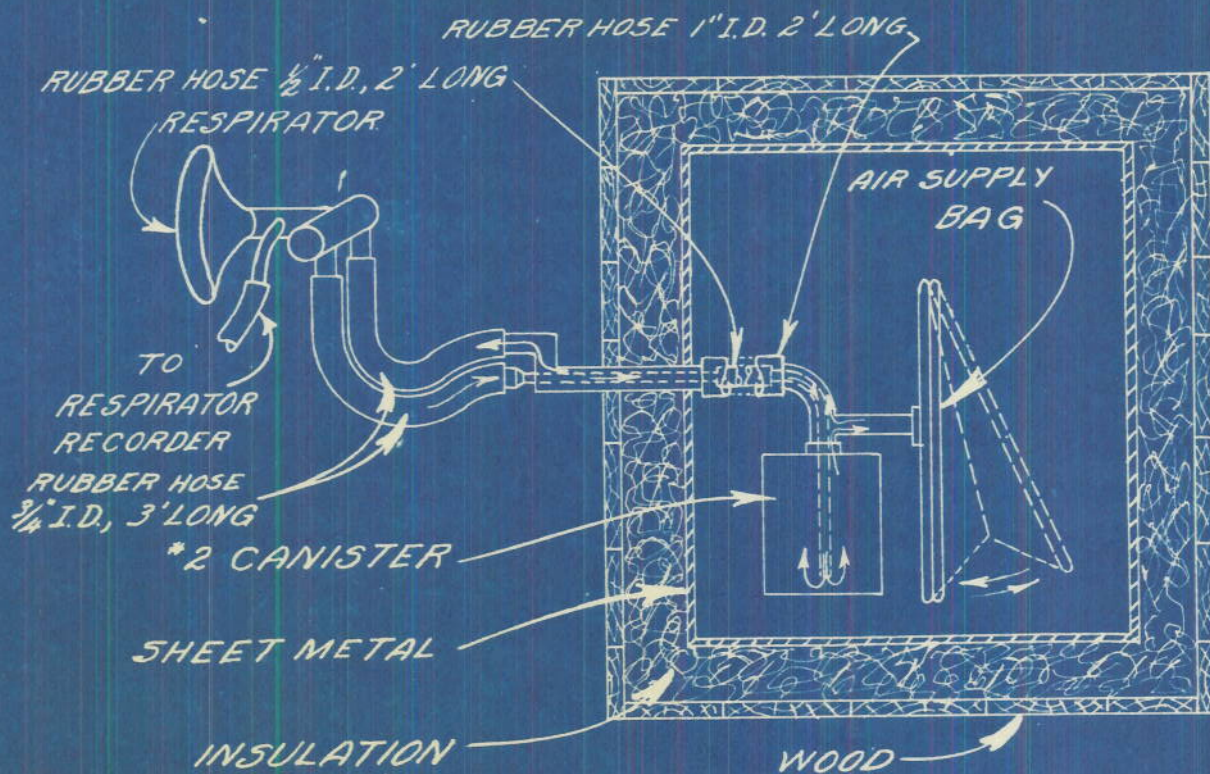


APPARATUS #1



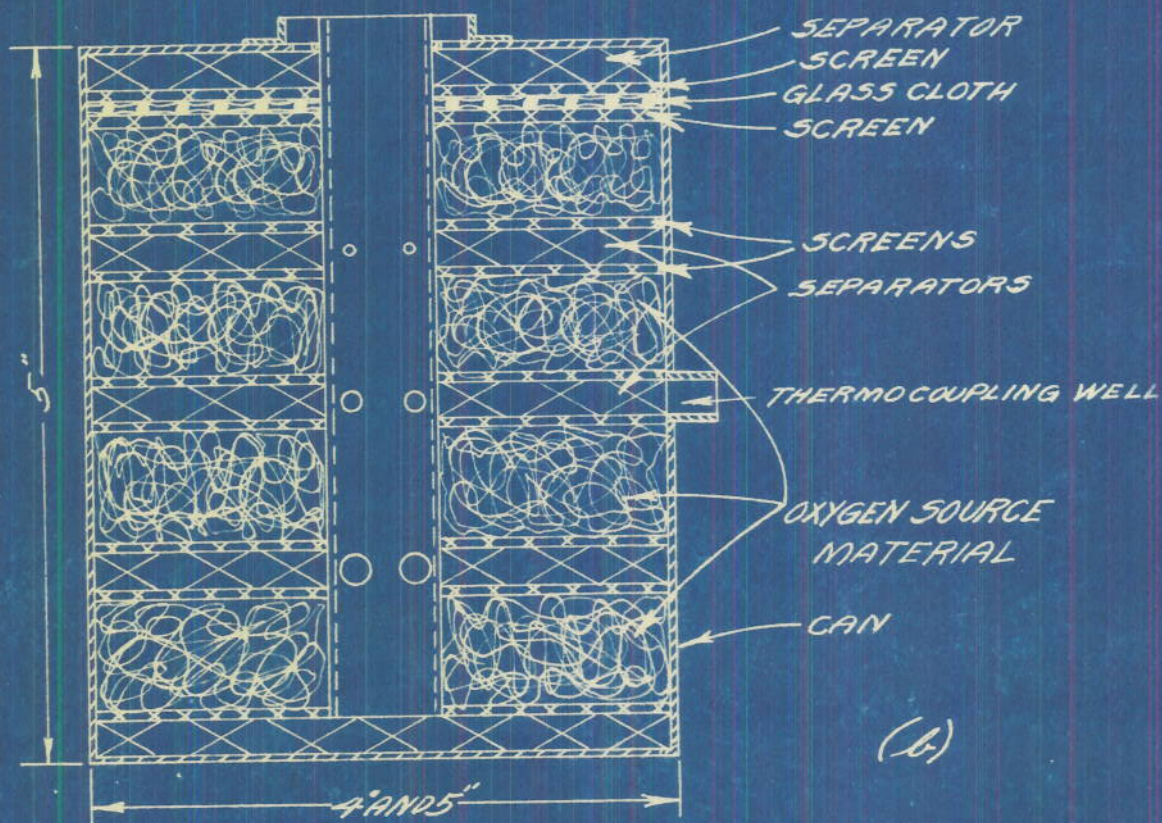
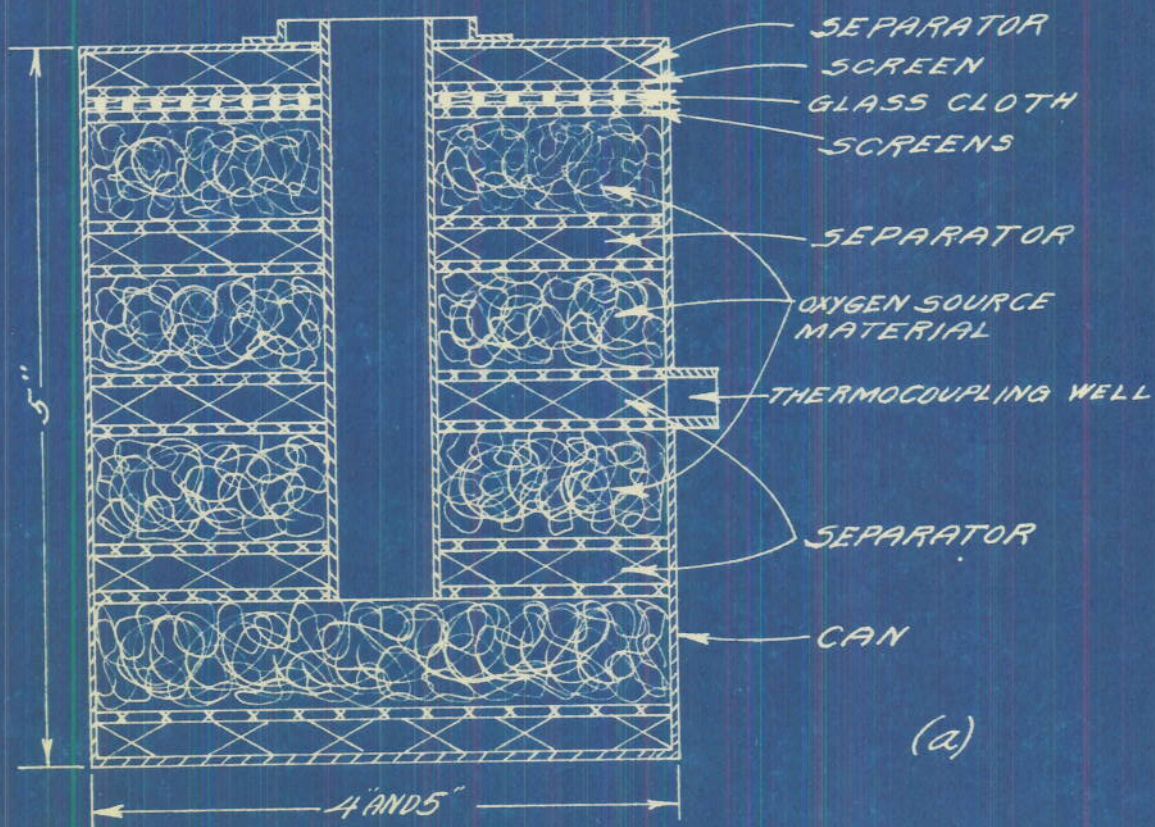
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APPARATUS #2



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CANISTERS



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DISCUSSION OF RESULTS

13. Catalyzed Materials. The tests on catalyzed materials with water vapor alone indicate that copper and manganese salts greatly increase the rate of liberation of oxygen; most other catalytic materials tested showed smaller increases (see data on Table III, Appendix.) While the few breathing tests on catalyzed material showed no promising results, more thorough testing is required to furnish definite information on this subject. Further testing may show that the effect due to catalysts, when the reaction is with water vapor alone, will be decreased in the presence of CO_2 .

14. Hydrates. Samples of mixed oxide absorbed moisture and on heating appeared to be stable at temperatures as high as 50°C . At higher temperatures (62°C .) reaction took place and oxygen was liberated. The oxygen liberated from a given heated sample, when completely reacted, was greater than that due to a reaction between sodium peroxide and the water present which indicates that part of the water reacted with the K_2O_4 . There is no definite proof that the water formed a hydrate of sodium peroxide and was not merely adsorbed on the surface of the material. Yet, water vapor and pure K_2O_4 will react and liberate oxygen under the conditions of hydration of the mixed oxide. The samples, with varying amounts of water, all started to react about the same temperature when heated (62°C .) which is more indicative of hydrated than absorbed water. From these data, it is believed that sodium peroxide in a mixed oxide can be hydrated and that the hydrate is reasonably stable at temperatures up to 50 or 55°C .

15. Breathing Tests. The canister tests plainly show one difficulty which is inherent in chemical oxygen source materials. It is that the reactions of CO_2 and H_2O with the oxides are not instantaneous. The first breath blown into a canister may show little reaction; depending upon the amount of "reactive" moisture that reaches the canister material. Reactive moisture is moisture that reacts with the oxide to free O_2 and facilitate the absorption of CO_2 , whereas unreactive water is that which an oxygen source material may absorb without liberating O_2 (e.g., formation of a hydrate). Reaction with CO_2 then depends on the reaction of the CO_2 with these compounds. The plates show that in some cases the canister reactions are slow at the start of the test and the oxygen in the bag falls to values which are too low for breathing purposes. The cause of the slow initial reaction is the lack of sufficient moisture in the gas stream. There is sufficient moisture in the air as it leaves the lungs but some is lost before it takes part in the reaction with material to set free O_2 and aid in the absorption of CO_2 . There are three places it can be lost: the first is in the tubing between the mouth and canister; in cold temperatures this is the largest loss. The second is in the canister where water may be taken up on reacted material and not reach the active material. The third loss is the water of hydration which can be taken up by Na_2O_2 but

[REDACTED]

not by K_2O_4 . The discussion on the hydrates shows that the Na_2O_2 in (K_2O_4 - Na_2O_2) mixture can also remove water. Loss of moisture in all three places has been experienced in the canister tests.

16. Where long tubing is used (3 or 4 feet) and loops upward to the bottom of a canister as in apparatus #1 a large amount of water collects in this tube. This loss percentage of the total water exhaled depends on the temperature of the tube. Where the tube is long, the air may enter the canister saturated with moisture at the tube temperature. At the temperature of $-18^\circ C$. the vapor pressure of water is 0.94 mm., compared with 48 mm. at body temperature, $37^\circ C$. With apparatus #1 water always collected in the loop, at times in quantities that filled a section of the $3/4$ inch tubing. Exhaled air was necessarily forced through the water and in some cases carried the water into the canister (See graphs 12, 13, 14 and 15). When the water was not kept liquid by the breathing, it froze and at times closed the tube. The trap (Plate 28) was added to collect the water in later tests; in test #13, 40 cc of water were collected. This loss of moisture can be reduced in the construction of the apparatus.

17. The loss of water on the reacted material may cause a canister to stop after reaction has once started, and CO_2 will then rise in the bag and breathing become increasingly more unsatisfactory. The excess moisture on the first or reacted layer may be enough at room temperature to cause the material to become soft and then pack tight and clog under the pressure of exhaling. This trouble can only be remedied by a more even distribution of the moisture or by forcing the moisture from one layer to another. Both methods were tried. The moisture was carried through by by-passing a part of the exhaled moisture through the layers with short tubes reaching through the lower layers. (See Plates 28 and 29). To force the moisture along, the canisters were insulated so that the heat of reaction would warm the canister and raise the vapor pressure of water. In this way, the water in a layer being heated by the absorption of CO_2 moved on to the next layer. The results of these tests can be seen in graphs from #23 on. While earlier canisters showed little reaction on the upper layers, with the addition of by-passes or insulation or both, reaction showed in each layer. Graph 23 shows tests on layers with insulation alone; the reaction went better in the first two layers but some reaction showed in all layers. Graph 24 shows that by-passing part of the breath through the first two layers increased the reaction in the third. In 25 by-passing through three layers gave much more even reaction in all four layers. In test 26.5 and later ones, entirely satisfactory results were obtained in the separate layers. The warming of the canisters by the reaction also accomplished two other purposes. The higher temperature decreases the formation of bicarbonate (Report P-1612, par. 11) and also increases the rate at which the reactions take place. At temperature near $100^\circ C$. some moisture has come completely through the canisters while it was reacting.

18. The construction of the round canister was not entirely satisfactory for reaction at cold temperatures. As can be seen from plate 29, the breath came through the lower end of the central tube and then entered the material from below; the cold bottom of an uninsulated canister condensed a large fraction of the exhaled moisture. The above difficulty was remedied in part by the use of the insulating woolen bag; in this way the heat of reaction was largely retained, thus warming the canister throughout. The increasing CO₂ in the walking tests indicate that the canisters are probably too small in cross-section; thus causing flow rates which are too great for complete absorption of CO₂ on the partially used material.

WORK IN PROGRESS

19. Drawings of canisters now under construction are given in plate 30; these canisters are designed specifically to remedy the above-mentioned difficulties. The purpose of the shortened central tube in canister (A) is to allow a layer of material to be placed on the bottom where it must react with any water condensing there or dripping from the inlet tube. The holes in the central tube of canister (B) are to serve as by-passes; it is thought that this type of by-pass will more evenly distribute the moisture than did the older type. The size of the holes will be such that a small amount of the exhaled breath will enter the canister between the third and fourth layers, a larger amount between the second and third, etc.; thus, the sizes and/or numbers of holes will increase toward the bottom of the tube. These holes will have a total opening such that if the first layer becomes clogged, normal breathing can continue through the holes without great difficulty.

20. Canisters of larger cross-section than that of the old type are also under construction; the two features mentioned in the preceding paragraph will be tested in these as well as in the small canisters. The increased volume of these larger canisters will reduce the flow rate of air through the material, thus affording a longer period of exposure (the same effect is to be gained by increasing the width of separators in the present-sized canisters). It is hoped that the increased quantity of material in the larger canisters will result in a proportionately longer life for the canister and perhaps in a better reaction efficiency.

21. The breathing apparatus as shown on plate 29 will be modified by the addition of a second air supply bag; this one connected with the inlet tube. It is believed that this extra bag will be of advantage in tests where the observer is doing some work; a portion of the exhaled breath will be stored in this bag from which it can diffuse slowly through the material.

22. The ideal canister is the largest one which will start quickly and react evenly and completely. It is believed that some combination of the features mentioned above, together with more efficient insulation, will furnish an apparatus which will approach ideal behavior.

CONCLUSIONS

23. The following conclusions may be drawn from the studies presented in this report:

(a) A number of compounds (catalysts) have been shown to increase markedly the rate of reaction between K_2O_4 and water vapor. An adequate study of the effect of these compounds under actual breathing conditions has not, as yet, been made.

(b) Certain hydrates of the mixed oxide (K_2O_4 and Na_2O_2) have proved to be more reactive toward CO_2 than either the anhydrous mixed oxide or the pure K_2O_4 . The use of these hydrates in canisters requires further investigation.

(c) The reactivity of pure K_2O_4 toward exhaled air has proved to be satisfactory at temperatures as low as $0^\circ F.$ and at ordinary atmospheric pressure. Approximately $5/8$ of a pound of K_2O_4 in apparatus #2 (plate 29) provided better than 500 cc. of oxygen per minute for $1-1/2$ hours; at the same time the CO_2 of the exhaled air was almost completely removed. (Graph 26)

(d) Canisters of the size studied so far have been found to be too small to absorb the CO_2 exhaled under working conditions, e.g., more than 80° cc. CO_2 per minute.

(e) The use of metal tubing or excessive lengths of rubber tubing for passage of the exhaled air to the canister must be avoided, thus minimizing the loss of water vapor by condensation on the cold walls of the tubing. Any natural trap in this tubing where condensed water might collect in quantity is also to be avoided.

(f) By-passes may be used to effect a more even distribution of water vapor throughout the canister; in this way, more water vapor reaches the active material in the upper layers and the clogging of the lower layers is eliminated.

(g) An efficient insulating jacket around the canister has been found to be of great value to the procedure of the canister reaction.

RECOMMENDATIONS

24. Final recommendations on this problem must await the completion of tests now in progress at this Laboratory. At present, the round canisters on apparatus #2 (plate 29) have been found to be satisfactory for an observer sitting or standing at ground level, for 1-1/2 to 2 hours without cylinder oxygen, at temperatures down to 0°F.

APPENDIX

Preparations of the Hydrates of the Mixed Oxide

Small weighed samples of the mixed oxide (10-20 grams) were suspended in weighing bottles over aqueous sulfuric acid solutions. The systems containing samples and solutions were then closed to the atmosphere and connected to manometers. In the case of reaction of the oxide with the water vapor present (forming oxygen) an increase would be observed in the manometric pressure. In the case of hydration of the material, no change of pressure would be observed until such time as the sulfuric acid became measurably more concentrated. Meanwhile, however, a steady weight increase would be found in the oxide. Such indeed was the condition when samples were allowed to hydrate slowly in the presence of water vapor at 5 mm (the vapor pressure of water over 58% sulfuric acid at 25°C.)

Larger samples of the mixed oxide (up to 1 pound) were hydrated in a "dry box;" the vapor pressure of water was herein maintained at 5 mm. by the exposure of 60% KOH solutions.

Effect of Catalysts upon the Rate of Reaction Between Water Vapor and Oxygen Source Materials

Air saturated with water vapor at 23.60°C. was drawn through a 5 gram sample of material at the rate of one liter per minute (see plate 27). The increase in volume of the closed system was due to liberated oxygen. Unless otherwise stated, about 10 milligrams of the dry, powdered catalyst was dusted on each sample. The total volume of oxygen liberated at various times and the oxygen source material and catalyst used are given in the following table:

TABLE III

Results of Laboratory Tests on Catalyzed Samples

Sample	Catalyst	Total Oxygen Evolved (cc.)					
		1 min.	2 min.	3 min.	5 min.	8 min.	10 min.
K ₂ O ₄ (96%)	Mn O ₂	3.3	8.0	13.9	27.9	52.1	75.4
"	Mn O ₂	3.4	8.5	14.9	30.9	58.2	76.6
"	Mn SO ₄	3.5	8.9	14.8	29.0	54.1	69.9
"	K Mn O ₄	3.5	9.4	16.2	31.7	58.1	72.7
K ₂ O ₄ (96%)	FeSO ₄ ·7H ₂ O	3.2	10.3	19.5	37.5	62.5	79.5
"	FeSO ₄ ·7H ₂ O	3.5	10.4	19.9	39.0	65.5	82.3
"	Fe O	2.8	7.4	12.7	24.4	43.5	54.3
"	"	2.8	7.5	12.9	24.9	--	--
"	Fe ₂ O ₃	3.0	9.2	17.0	31.5	54.4	70.1
K ₂ O ₄ (96%)	Zn	2.4	6.4	11.2	21.4	38.9	51.4
"	Zn	3.0	7.3	12.0	22.4	39.5	51.0
"	Zn O	2.2	6.5	11.5	21.9	37.3	48.0
"	(VO) ₂ (SO ₄) ₃	2.6	7.3	12.3	22.9	39.5	50.6
"	V ₂ O ₅	3.0	6.2	14.0	26.4	46.9	61.2
"	V ₂ O ₃	3.4	8.9	14.9	27.7	49.8	66.4
"	Ni on silica gel	2.3	7.1	12.5	24.1	42.3	54.8
"	NiCO ₃ ·Ni(OH) ₂	3.1	8.2	13.9	25.9	45.0	57.3
"	Pt on KCl(dil.)	2.3	6.3	10.8	20.9	37.7	48.8
"	Pt on KCl(conc.)	2.8	7.4	12.7	23.6	42.0	53.6
"	K ₂ Cr ₂ O ₇	3.5	8.8	14.1	25.1	42.3	54.1
"	Cr ₂ (SO ₄) ₃	3.4	8.8	14.6	26.6	47.6	62.8
"	Pb O	3.3	8.5	14.1	25.5	44.7	58.8

TABLE III (Continued)

<u>Sample</u>	<u>Catalyst</u>	<u>Total Oxygen Evolved (cc.)</u>					
		<u>1 min.</u>	<u>2 min.</u>	<u>3 min.</u>	<u>5 min.</u>	<u>8 min.</u>	<u>10 min.</u>
K ₂ O ₄ (96%)	None	3.0	8.0	13.6	24.3	40.5	51.8
"	None	3.3	8.7	14.4	25.1	41.5	53.1
K ₂ O ₄ (96%)	Cu	5.4	13.2	22.4	41.2	67.6	81.9
"	Cu	3.9	11.5	19.9	37.3	63.6	80.7
"	Cu ₂ O	6.0	15.6	24.7	41.5	65.8	81.2
"	Cu ₂ O	6.2	15.0	23.8	40.2	63.6	79.0
"	CuSO ₄	6.2	16.3	25.9	44.8	72.8	90.9
"	CuSO ₄	5.5	14.8	24.3	43.6	72.2	90.7
"	CuSO ₄ ·5H ₂ O	6.5	16.2	25.7	45.3	73.8	93.1
"	CuSO ₄ ·5H ₂ O	6.9	16.3	25.6	44.0	72.0	88.4
"	CuCO ₃ ·Cu(OH) ₂	7.3	17.2	26.9	46.3	73.5	89.9
"	CuCO ₃ ·Cu(OH) ₂	7.3	16.8	26.1	43.8	65.5	80.3
"	Cu(C ₂ H ₃ O ₂) ₂ ·H ₂ O	7.1	17.0	27.0	47.3	75.8	92.2
K ₂ O ₄ (90%)	None	1.4	4.5	8.2	17.0	32.2	42.9
"	None	1.6	4.6	8.3	16.6	31.4	40.7
"	Heated with 0.02% Cu SO ₄	1.9	5.8	10.8	22.8	44.1	59.8
"	Heated with 0.1% Cu SO ₄	3.4	9.5	16.3	32.0	56.7	74.3
"	Heated with 0.5% Cu SO ₄	2.8	8.3	15.2	30.8	57.1	75.2
"	Heated with 0.7% Cu SO ₄	3.4	9.0	14.4	29.4	51.6	67.5