

FOREWORD

This report was prepared in the Flight Dynamics Laboratory, Research and Technology Division, Wright-Patterson AFB, Ohio, under Project 6146, Task 614611, entitled "Carbon Dioxide and Water Vapor Control Techniques." This document summarizes the investigations and results of work performed in the Atmospheric Regeneration and CO₂ Control Laboratories. Dr. John P. Allen is the project engineer for this work, which, begun in July 1963, is of a continuing nature, and will be reviewed in this and in future reports.

In this report problems of carbon dioxide management in a closed system are discussed, followed by a description of a technique for evaluating some materials and/or methods for its control. Included in this report are performance figures resulting from the investigation which will provide a basis of comparison of other materials performances in similar investigations.

Contrails



ABSTRACT

A laboratory device was assembled for a closed air loop analysis of carbon dioxide removal agents. Lithium hydroxide, potassium hydroxide, soda lime, and molecular sieve 5A were used for establishing the adaptability and capability of this device for evaluation of other carbon dioxide removal agents. Carbon dioxide absorption curves from 1 percent and/or 5 percent carbon dioxide in laboratory air were obtained. Borax solution and amine solutions or solids showed some carbon dioxide removal capacity which will be further investigated for quantitative data on the removal process.

This technical documentary report has been reviewed and is approved.

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TABLE OF CONTENTS

SECTION	PAGE
INTRODUCTION	1
BACKGROUND INFORMATION	1
Scope of the Investigation	1
Techniques for Removal of CO ₂	2
Theoretical Considerations	3
DEFINITION OF THE PROBLEM AREA	4
EXPERIMENTAL PROCEDURE	5
EXPERIMENTAL TESTS AND RESULTS	7
Apparatus Volume	7
Leakage	7
Time Period of CO ₂ Absorption	7
Solid CO ₂ Absorbers	7
Liquid CO ₂ Absorbers	9
Discussion of Tests and Results	11
SUMMARY AND CONCLUSION	15
REFERENCES	16
BIBLIOGRAPHY	17



ILLUSTRATIONS

FIGURE		PAGE
1.	Closed Air Loop For CO ₂ Absorber Evaluation	18
2.	Volume Calibration with 1033 cc Flask on 0 to 5% Range	19
3.	Volume Calibration with 2033 cc Flask on 0 to 1% Range	19
4.	Volume Calibration with 1033 cc Flask on the 0 to 1% Range	20
5.	CO ₂ Absorption by Soda Lime Canister	20
6.	CO ₂ Absorption by Soda Lime Bulb	21
7.	CO_{e} Absorption by Soda Lime Bulb, Sequent to Figure 6	21
8.	CO ₂ Absorption by LiOH·H ₂ O	22
9.	CO ₂ Absorption by LiOH Anhydrous	22
10.	CO ₂ Absorption by Molecular Sieve, 5A	23
11.	CO ₂ Absorption by Na ₂ CO ₃ , Granular	23
12.	CO ₂ Absorption by Amberlite IRA-401S Resin	24
13.	CO _a Absorption by Borax, Anhydrous	24
14.	CO ₂ Absorption by o-Phenylene Diamine	25
15.	CO ₂ Absorption by Rexyn RG-6 (OH) Resin	25
16.	CO ₂ Absorption by Cellulose Acetate, Granular	26
17.	CO ₂ Absorption by KOH Solution	26
18.	CO _a Absorption by Distilled Water	27
19.	CO ₂ Absorption by Dilute Pyruvic Acid	27
20.	CO ₂ Dilution Effect by 1 x 8 Inch Test Tube	28
21.	CO ₂ Absorption by Aqueous Suspension of Amberlite IRA-401S Resin, 2.28 Grams	28
22.	CO ₂ Absorption by Aqueous Suspension of Amberlite IRA-401S Resin, 2 cc	29
23.	CO ₂ Absorption by KC1, 0.1 Molar v	29



ILLUSTRATIONS (CONT'D)

FIGURE		PAGE
24.	CO _B Absorption by TRIS Solution	30
25.	CO ₂ Absorption by KH ₂ PO ₄ , .06 Molar	30
26.	CO ₂ Absorption by K ₂ HPO ₄ , 0.013 Molar	31
27.	CO ₂ Absorption by Na ₂ HPO ₄ , .06 Molar	31
28.	CO _a Absorption by EDTA, 5% Solution	32
29.	CO ₂ Absorption by Urea, 4% Solution	32
30.	CO ₂ Absorption by Ethylene Diamine, 1% Solution	33
31.	CO₂ Absorption by Ethylene Diamine, 1% Solution, Sequent to Figure 30	33
32.	CO ₂ Absorption by Ethylene Diamine, 1% Solution, Regenerated by Boiling	34
33.	CO ₂ Absorption by Saturated Borax Solution	34
34.	CO ₂ Absorption by Borax Solution, 0.1 Molar	35
	TABLES	
		PAGE
1.	Man's CO ₂ Production Every 24 Hours	2
2.	Molecular Sieve CO ₂ Absorption at 1% CO ₂ in a 1655-cc System	8
3.	CO ₂ Removal by Solid Absorbers	12
4.	CO ₂ Removal by Liquid Absorbers	13
5	CO Absorption by Solid Absorbers	14

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FDL TDR 64-67 Part I

INTRODUCTION

The control of carbon dioxide (CO_2) in aerospace cabin atmospheres has been the theme of many investigations having as their aim the presentation of data for incorporation into the design of environmental control systems. The investigations have ranged from simple absorption systems to complex processes of absorption in which catalytic reduction of CO_2 with hydrogen was used to ultimately recover the oxygen from the CO_2 . Many factors in the CO_2 absorption process are limiting and controlling in the overall process of CO_2 removal. To attempt to evaluate the many factors significant to CO_2 control systems would be a herculean undertaking, but the investigation of specific factors having significant application to and a limiting effect on an engineering design would provide a great return for the effort expended. It is intended in this work to devise a laboratory technique to investigate some of the many facets of the CO_2 control processes by the absorption, adsorption, or persorption process and to evaluate some of the effects which are presenting difficulty in the engineering application of the data obtained.

BACKGROUND INFORMATION

SCOPE OF THE INVESTIGATION

The carbon dioxide control problem in manned aerospace closed systems is determined by the metabolic limits of the spaceman and the extent of his activity. Some specific figures that can be used in discussing CO_2 control quantitatively are the amount of CO_2 produced per day and the concentrations of CO_2 to be tolerated. Values given for man's CO_2 production range from 0.8 to 1.2 cubic feet per hour, with an average daily CO_2 production of 2.0 to 2.4 pounds (Reference 1). The amount of CO_2 produced varies according to the diet, activity, psychological situation, temperature, and physical well-being; but a figure of 0.1 pound per hour is an acceptable value for use in this investigation. This selection is justified when one considers the tolerances and efficiences assumed in various CO_2 treatment processes. The basic tenet is that the quantity of CO_2 used as the basis for calculation be on the plus side since CO_2 buildup is definitely to be avoided. A CO_2 concentration of 1 percent in the aerospace vehicle cabin atmosphere has been designated the maximum concentration allowable (Reference 1).

From this basic figure of 0.1 pound of CO₂ per hour, calculations and conversions reveal the following information. When the molecular weight of CO₂ is taken as 44 and its molecular volume 22.267 liters as given by Quinn and Jones (Reference 2), the production of CO₂ per hour is:

```
0.1 lbs.
45.359 grams
22.9127 liters
.819 cu. ft.
.887 cu. ft. (at 14.7 psi 80°F)
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Even these figures are not directly applicable to CO_2 control processes because the removal processes are based on 10-, 15-, 20-, and 30-minute cycles. Upon conversion of the above production figures to shorter time intervals, the quantity of CO_2 production, based on 2.4 pounds every 24 hours, changes to the values given in Table 1.

TABLE 1

Man's CO₂ Production Every 24 Hours

1 min.	10 min.	15 min.	20 min.	30 min.
.0016 lbs. .756 grams .382 liters .0134 cu. ft.	.016 lbs. 7.56 grams 3.82 liters .13 cu. ft.	.024 lbs. 11.34 grams 5.73 liters .20 cu. ft.	.032 lbs. 15.12 grams 7.64 liters .268 cu. ft.	.05 lbs. 22.68 grams 11.46 liters .40 cu. ft.

The tabulated data indicates directly the quantity of CO_2 in weight and volume to be removed and/or transferred. These values must necessarily be corrected for pressure differential because the aerospace cabin might be at either 5, 7.5, 10.0, or 14.7 psia. The lower pressures will alter the CO_2 weight and volume relationship since, at reduced pressures, the respiratory quotient is raised even though the oxygen consumption is about the same, and the quantity of CO_2 in the blood is decreased because of the release of more CO_2 , and the increase in rate and volume of breathing (Reference 3).

TECHNIQUES FOR REMOVAL OF CO2

The proposed techniques for removing CO_2 from aerospace vehicle cabin atmospheres are many, and may be classed generally as chemical, physical, or electro-chemical in principle. The chemical techniques range from a simple base-plus- CO_2 reaction to those involving oxygen evolution from superoxides. The latter are reactions of CO_2 with potassium superoxide and silver superoxide (References 4 and 5). The principle of physically removing CO_2 involves adsorption (Reference 6), solution concepts (Reference 7), and is further extended into membrane- and resin-separation of CO_2 (Reference 8). The electrochemical concept of CO_2 separation involves the formation of electrically transported ions through an anionic membrane after which the CO_2 is released as a gas (Reference 8).

The summary and conclusions of various reports on techniques of CO_2 removal and control include both favorable and unfavorable comments on the capabilities of the respective techniques. The lithium hydroxide (LiOH) technique was successfully used in the Mercury capsules and is being used in biomedical space capsules. But, because this technique is not a regenerative one, its use is necessarily limited to missions of short duration. An evaluation of this technique (Reference 9) revealed some problems with irritation from LiOH dust. However, when LiOH was used with CO_2 and water its reaction was consistent with theoretical discussions of this concept.

In several reports (References 10, 11, 12, 14, and 15), the adsorption of CO_2 on molecular sieves, silica gel, activated carbon, and alumina is discussed and the capacities of each are graphically presented along with supporting data which provides a basis for design and operation of a CO_2 removal technique for regenerating the CO_2 absorber. Graphs are also



included to show the minimum and maximum quantities of adsorbent for various partial pressures of CO_2 .

Other removal techniques are the freeze-out method by which the CO_2 air mixture is cooled to below its frost point (Reference 12), and the absorption-by-solution (into spray or packed towers) method. Both techniques appear at first to be beset with difficulties when they are applied to the prescribed conditions for manned atmospheres. For example, in the freeze-out technique, special consideration must be given to the power requirements for maintaining the proper cooling temperatures, for providing high enough air flows in the short recirculation time of the air, and for fulfilling the factors involved in handling low quantity (less than 1 percent) of CO_2 in the air. Then, the spray-tower technique, of course, would have no place in a zero-gravity environment.

But, a solution-absorption technique employing the more recent microporous membrane liquid-gas separators reveal great potential for modifications of current CO₂ adsorption on molecular sieves (as indicated by proposed low-temperature molecular sieve CO₂ removal systems, Reference 12). And, relatedly, intermediate temperatures offer a good area for investigation of loading capacities and controlling characteristics.

Endeavors, to date, with liquid-gas separation by microporous membranes support the emphasis on its potential and the subsequent need for development of this approach to solution-absorption of CO_2 from the air stream. A photosynthetic gas exchanger as designed by the General Electric Company (Reference 13) uses a microporous membrane for exchange of both O_2 and CO_2 in the solution of salts. Here, the problem was physical blockage of gas-exchange membrane by algal cells, but still the gas passage and quantities were considered adequate for this use. The photosynthetic gas-exchanger report recommended further work to evaluate a class of membranes of silicon rubber for diffusing and removing CO_2 from the air into a solution; the toxicity of this material to algal cells was a significant fact.

THEORETICAL CONSIDERATIONS

Carbon dioxide removal techniques have evolved to a stage such that the capacities and efficiences of the techniques have been defined sufficiently for exploratory application to simulated manned-sized space capsules. The direct application of experimental data to engineering designs results in the discovery of certain hidden "facts" which may involve changes in the capacities, efficiencies, time rates of change, etc. One of these facts is the toxic or poisoning effect of water vapor on the molecular sieve adsorption capacity for CO₂. This is essentially the preferential absorption of water over that of CO₂ such that the desired CO₂ absorption is nullified. This effect in experimental models of sieve systems for CO₂ removal is controlled either by freeze-out of water or by drying agents. One engineering design (Reference 10) thus provided for some preferential water vapor absorption by increasing the amount of sieve available for the process, and in one estimate provided up to 9.3 pounds of molecular sieve per man. This manner of handling the problem appears unjustified in view of the experimental data obtained in laboratory runs on CO₂ absorption capacities of the molecular sieve. Experimentally, for a molecular sieve process at 15 psi and 7.6 mm Hg CO₂, 2.1 pounds of sieve material would absorb up to 8.5 percent of its weight of CO₂ at 77°F. This amounts to almost 4 times the rate of production for longer than a 30-minute period, or 8 times the production rate over 15 minutes.

The CO₂ removal concept is thus in need of a technique for obtaining a water-free gas stream or of a CO₂ absorber or adsorber technique that is unaffected by the presence of



water or possibly enhanced by the presence of water. Hydrophobic membranes permeable to gases and possibly differentially permeable to gases would provide one answer. Work is progressing along this vein. Also the CO₂ absorption by ion exchange resins (References 7 and 16) and membranes of ion exchange resin provide for CO₂ diffusion unaffected by the presence of water. The characterization of materials that could act in this capacity could provide an impetus to their application to CO₂ control in the range required for atmospheric control.

DEFINITION OF THE PROBLEM AREA

From the foregoing discussion it becomes evident that the CO_2 removal from an air stream by means of a regenerable absorber or adsorber, is directly related to the process of water vapor removal. The presence of water vapor limits severely the quantitative removal of CO_2 by molecular sieve materials. A "water-proof" molecular sieve would seem to solve the problem but this idea has not as yet been investigated.

Another concept would be to use an absorption principle in which the presence of water is required for CO_2 absorption This type of CO_2 absorption occurs with the organic amines. This concept is discussed in Reference 15 but the data presented is limited in scope. An extension of this type of CO_2 absorption investigation appears merited.

The absorption of CO_2 from a low-percentage CO_2 content in air constitutes a real problem when no more than 1 percent (or more desirably, considerably less than 1 percent) must be maintained in the air. To maintain a 1-percent CO_2 content means that to remove 1 volume of CO_2 , 99 volumes of air must have passed through the removal device. With this requirement must also be considered the efficiency of the process and the capacity or degree to which the absorber can be loaded. In solid absorber systems, to maintain an air passage great enough to result in an air mixture containing less than the 1 percent maximum CO_2 , the air must be continually processed at a high mass flow, but must always contain the low percentage of CO_2 .

The absorption process must necessarily have an efficiency of less than 100 percent to attain the required CO_2 air mixture control and will range downward to zero percent according to how close to saturation the absorber is. The most effective portion of the absorption process would be that portion above the value where removal of CO_2 would equal CO_2 production by the source; this production source value, as noted earlier, is established by man's physiology to be 0.1 pound of CO_2 per hour. The data given in Table 1 establishes basic figures for the removal process. From these figures, for a cycling process, with a 10-minute cycle, the removal process must remove at least 7.56 grams of CO_2 or 3.818 liters. On a percentage basis, 381.8 liters of a CO_2 air mixture at 1-percent CO_2 must be processed every 10 minutes with a 100-percent CO_2 removal efficiency; otherwise, the CO_2 percentage in the air will rise. For a 20-minute cycle, 763.6 liters of CO_2 air mixture with 1-percent CO_2 must be processed under the same requirements, to just balance the CO_2 production. If a .5-percent CO_2 level is the maximum CO_2 limit, then the amount of air or 763.6 liters would be processed every 10 minutes with 100-percent removal efficiency.

From this discussion, it is evident that direct CO₂ absorption from air requires a high air flow and high mass velocity to achieve such a complete removal with high efficiency. Flow-through absorbers are effective, but will apparently require high power inputs to attain the mass air flows needed to operate a CO₂ removal unit of dimensions commensurate



with the volume requirements for an atmosphere-control device. From the literature (Reference 10), an absorber with 28 pounds of molecular sieve for a 3-man crew appears an excessive amount of absorber even though intended for both water and CO₂ removal. Emphasis in the work on CO₂ absorbers has been on high margins of safety by oversizing absorber beds and flow-through rates. Marginal operational modes in exploratory phases could more realistically define the limiting factors, and more fully evaluate the design data.

EXPERIMENTAL PROCEDURE

To evaluate some of the experimental and engineering data on CO₂ removal techniques, an apparatus providing for a closed air loop was assembled. The closed air loop (Figure 1) consisted of a recirculating vacuum-blower pump with a bypass valve to provide for an air-flow control. With valving and flowmeters, various CO₂ concentrations could be obtained in a completely closed air loop. An infrared CO₂ analyzer monitored the CO₃ concentration. A U-shaped tube containing soda lime provided the means for changing the CO₂ concentration to the desired percentages of 1 to 5 percent. Drierite was used to dry the air and CO₂ so that water would not interfere with the analyzer. Through the use of a gas dispersion tube, a plastic cell for gas dispersion, or gas absorption bulbs, liquids and solids were evaluated as to CO₂ absorption. During the evaluation of the solutions, an ice bath served to condense the water from the air loop before the drierite drying.

The components of the closed air loop apparatus are as follows:

- 1. Air-circulating pump, DynaVac Pump, Model 3, Cole-Parmer Instrument and Equipment Company.
- 2. Flow meter, Model 622BBV, Tube No. 603, Matheson Company.
- 3. Filter unit, glass wool, 60 cc, brass container.
- 4. CO₂ L/B infrared analyzer and amplifier, Model 15A, range 0 to 5 percent, Beckman Company.
- 5. Angus recorder, Model AW, 0 to 50 ua, Esterline Company.
- 6. Flow meter, Tube No. 2-85A, 0 to 2 cfh, Brooks Rotameter Company.
- 7. Flow meter, FB Model 10A3135A, 0 to 100 percent.
- 8. Stainless steel spherical tank, volume, 10 liters.
- 9. U-tubes, with soda lime or Drierite, volume, 90 cc.
- 10. Drierite tank, 350 cc (approx.) with screen cone.
- 11. Test tube (1 inch x 8 inch), with sintered-glass gas diffuser, extra coarse, volume 76 cc.
- 12. Absorption bulb, internal volume, 60 cc, Fleming-Martin.



- 13. Phosphorous anhydride tube, internal volume, 60 cc.
- 14. Valves, 2-way, three ports.
- 15. Needle valve.
- 16. Flow meter, 0 to 2.0 cfh, FP Tube No. 04-38A.
- 17. Ice bath, thermos bottle No. 8640, test tube (1 x 8 inch).
- 18. Air-loop tubing No. 44-P, 1/4 inch, Imperial "Poly-Flo".

The air-circulating pump in the air loop produced a flow of .4 to 2 cfh or 58.6 liters per hour, and could be controlled with the bypass valve to flows of .2 cfh. This value in flows per minute is .003 cfm or 97 cc per minute. Ordinarily, evaluations were made at 0.8, 1.0, or 1.6 cfh, or respectively, 377.5, 471.8, and 755.0 cc of air per minute.

Measurements of CO_2 content were accomplished with the Beckman infrared analyzer, which had a 0 to 5 percent range. Water-pumped nitrogen, after it passed through a silica gel cartridge, was the zero gas, and 5-percent CO_2 in nitrogen was the calibration gas. A range selector was used in conjunction with the Esterline Angus recorder so that two recording ranges, 0 to 5 percent and 0 to 1 percent full-scale deflection, were available.

The flow meters and a manometer served to monitor the air flow throughout the air loop. Glass wool in a 60-cc-volume filter unit provided for ample air filtration. The air loop tubing with its 2-way brass valves allowed adequate control of the various system components. Temperature and pressure control were not attempted and were at ambient, $25\pm5^{\circ}$ C and 740 ± 10 mm Hg. The pressure drops throughout the loop were not given consideration at this time.

The air in the loop was composed of $\mathrm{CO_2}$ in laboratory air. Oxygen was not given any consideration in this phase of the work. The $\mathrm{CO_2}$ was admitted to the air loop from the $\mathrm{CO_2}$ supply and the air plus $\mathrm{CO_2}$ were allowed to recirculate until the $\mathrm{CO_2}$ analyzer indicated a constant trace on the recorder at the percentage required. Leakage was definitely a problem with so many connections. The rate of leakage was significant over hourly periods of time but over the 10 to 15 minute intervals during which the measurement of the $\mathrm{CO_2}$ absorption was recorded, the leakage was slight.

A $\mathrm{CO_2}$ absorption determination consisted of obtaining a percent of $\mathrm{CO_2}$ in the air loop, and then valving it into the $\mathrm{CO_2}$ absorbing device. The $\mathrm{CO_2}$ removal from the air stream was recorded against a time interval so that rate of removal could be observed and the initial and final $\mathrm{CO_2}$ concentrations recorded. The weight change of the absorber was recorded in some instances. These weights were not significant for lithium hydroxide and soda lime since the water liberated in the reaction was removed from the absorber and absorbed in the drierite tubes. A comparison of the curves provided a basis for qualifying the absorbing materials as to their $\mathrm{CO_2}$ removal capacities.



EXPERIMENTAL TESTS AND RESULTS

APPARATUS VOLUME

The internal volume of the air loop was measured by a dilution effect of a certain concentration of CO₂ in the loop. A volumetric flask was introduced into the air loop so that a volume of laboratory air diluted the original CO₂ concentration. A 1-liter flask with an internal volume of 1033 cc when valved into the air loop reduced the CO₂ percentage from 3.60 to 1.80 percent (Figure 2). A 2-liter flask with an internal volume of 2030 cc reduced the CO₂ percentage from 0.88 to 0.30 percent (Figure 3). A duplicate test with the 1-liter flask changed the CO₂ percentage from 0.60 to 0.31 percent (Figure 4). This would calculate to a figure of approximately 1040 cc for the internal volume of the air loop. A surge tank with an internal volume of 615 cc was used in initial runs so that the volume was 1655 cc in initial evaluations. Thus, the volume changed as modifications were made to the air loop as to tubing lengths and the drierite containers. However, in view of the leakage discussed below these volume values of 1040 and 1655 cc were considered reasonably accurate.

LEAKAGE

The air loop revealed some leakage of the contained gases. With CO_2 in the air loop, the CO_3 leakage rate was higher at the higher percentages of CO_2 . It was found that, with 3.20 percent of CO_3 in the system, after 1 hour, the CO_2 concentration was 2.95 percent; after 2 hours, 2.70 percent; 3 hours, 2.45 percent. These values indicate a leakage of 0.25 percent per hour. Other values indicated leakage rates either more or less than 0.25 percent per hour; however, the leakage was a small factor in the overall process. At a concentration of 1 percent of CO_2 in the air loop, the leakage was less than the 0.25 percent per hour rate indicated by the various test runs in which the CO_2 absorption was low, and a graph of this CO_3 concentration remained within 0.02 percent of the initial CO_2 concentration over the 10-minute interval for the absorption process.

TIME PERIOD OF CO. ABSORPTION

As the CO_2 air mixture was passed through the absorber, the time interval over which the CO_2 percentage was recorded was arbitrarily limited to 10 minutes. However, lesser time intervals were considered valid when the curve indicated a removal rate comparable to that of an arbitrary standard absorber.

SOLID CO2 ABSORBERS

Soda lime in either a canister or a U-tube revealed a good CO₂ absorption. Figure 5 shows almost complete absorption of CO₂ by soda lime in a canister from a 5-percent CO₂ air mixture in 10 minutes. The end point at zero was at the first unit on the graph and corresponded to the zero gas trace. The soda lime was used to adjust the CO₂ percentages in the air loop and to remove all CO₂ from the loop when this removal was required. Since some water is released in the reaction, weight measurements of the amount of CO₂ absorbed by the soda lime were not obtained. The soda lime used was 8 to 14 mesh, indicator grade (Fisher reagent), and had a 30 percent by weight CO₂ absorption capacity. Figures 6 and 7 show CO₂ removal curves in shorter time intervals with 1-percent CO₂ in the air loop.

Lithium hydroxide monohydrate was also used in CO₂ absorption tests and the graphs



obtained indicated over short time periods complete and rapid CO_2 removal from a 1-percent CO_2 concentration in air (Figure 8). Weight measurements of the phosphorous anhydride tube containing the LiOH revealed that the water formed in the absorption process was being evolved from the reaction mixture. In one absorption run a weight loss of 7 milligrams was measured even though the infrared CO_2 analyzer indicated a complete removal of CO_2 from the air stream.

Baking the LiOH monohydrate at 225°C to drive off the water of crystallization produced anhydrous LiOH which revealed a greater weight increase when used for $\mathrm{CO_2}$ absorption (Figure 9). This weight increase, however, did not correspond to the weight of $\mathrm{CO_2}$ calculated in the air loop; instead, a weight increase of 17 mg was measured. This corresponds to 8.59 cc of $\mathrm{CO_2}$, but at a starting percentage of 0.95-percent $\mathrm{CO_2}$ in the air loop, this would correspond to 9.88 cc of $\mathrm{CO_2}$ in the air (or 19.52 mg of $\mathrm{CO_2}$). This fact was significant in the use of LiOH only as an "absorber qualifier" and not as a standard of measurement when making $\mathrm{CO_2}$ absorption comparisons.

Molecular sieve, No. 5A, as 1/8-inch pellets in a weighed absorption bulb, was used in this series of tests as a standard for the total absorption of CO_2 in the air loop. The molecular sieve (without the bulb) weighed 36.834 grams, and was sufficient to absorb 1.84 grams of CO_2 at a 5-percent loading capacity. This molecular sieve capacity was also sufficient to make eventual saturation with CO_2 a remote possibility in these absorption runs.

The absorption of $\mathrm{CO_2}$ by the molecular sieve was complete and rapid in the time period of 10 minutes (Figure 10); the weight increase was .138 grams when a 4.9-percent $\mathrm{CO_2}$ air mixture was used. And, at a 4.9-percent $\mathrm{CO_2}$ concentration and air mixture volume of 1655 cc, this would give 81.09 cc of $\mathrm{CO_2}$, corresponding to 0.160 grams of $\mathrm{CO_2}$ under standard conditions. The difference in weight is explained by leakage in the air loop, which was greater at the higher percentages.

Other runs, with 1-percent $\mathrm{CO_2}$ as the maximum $\mathrm{CO_2}$ concentration, provided weight additions of 30, 28, 29, and 33 milligrams. Table 2 indicates these weight to volume relationships; the various weights given show the different molecular sieve adsorptions of $\mathrm{CO_2}$ in the several tests, and also represent some water absorption from the air. And, since 1 percent of the 1655 cc internal volume equals 16.55 cc of $\mathrm{CO_2}$, from the table, a weight of 30 milligrams would indicate 15.17 cc (17.103 cc at 27°C and 740 mm) if the entire weight increase were all $\mathrm{CO_2}$. (These evaluations were made during a series of runs in which solutions were also being evaluated so that some water vapor was present in the air loop and on the desiccant materials in various percentages of saturation.)

TABLE 2

Molecular Sieve CO₂ Absorption at 1% CO₂ in a 1655-cc System

Weight Diff. (mg)	Equiv. Volume (cc)	Volume at 27°C, 740 mm (cc)
28	14.364	16.188
29	14.870	16.758
30	15.176	17.103
33	16.694	18.814



Other solid materials evaluated for CO_2 adsorption were sodium carbonate (32.97 grams), sodium bicarbonate plus sodium carbonate, Amberlite IRA-401S ion exchange resin (21.170 grams), borax (5 to 10 mesh), o-phenylene diamine, Rexyn RG6 OH form ion exchange resin, and cellulose acetate. None of these solids compared closely with the molecular sieve or the soda lime in CO_2 absorption capacity. Related curves are given in Figures 11 to 16.

Some solid materials that were tried but with which much difficulty of air passage was experienced were the Wilson No. 43 gas mask mix, sodium bicarbonate, and asparagine. No absorption curves were obtained.

LIQUID CO2 ABSORBERS

Liquid absorbers were evaluated in either a test tube (1 x 8 inch) with a gas-dispersion sintered-glass EC tube or in a lucite cell using a plastic tube as the gas dispersion device. The test tube utilized 25 or 50 ml of the aqueous solutions of the absorbers, whereas, in the plastic cell, 10 or 15 ml of the solutions were used. The test tube has an internal volume of 76 cc; the lucite cell, 32 cc. The lucite cell was a 2-inch-diameter cylinder with two 1/4-inch NPT openings in one face of the cylinder. The other face of the cylinder was a polyethylene film, 5 mils thick. The surface of the film away from the cylinder was the flat face of another cylindrical cell having a volume of 27.6 cc and two 1/4-inch NPT openings which were connected with tygon tubing.

Dilute potassium hydroxide solution, 55 cc of a .99-percent solution, was used as an absorption medium in the test tube. The air loop was modified so that an ice bath condensed most of the water vapor prior to the passage of the air mixture through the drierite tubes. The curves of absorption indicate rapid and almost complete absorption of the CO_2 from a 4.1-percent CO_3 air mixture; Figure 17 is a typical absorption curve.

Water will dissolve CO₂ from the air at quantities as referenced in Quinn and Jones (Reference 2). This solution effect was evaluated with the test-tube technique and the curve in Figure 18 reveals the absorption which took place over a 10-minute interval. A longer absorption period (up to 110 minutes) revealed a continuing decrease in the percentage of CO₂ absorbed, but this decrease is related to leakage. This absorption of CO₂ into water is a factor to be considered when buffers and other similar solutions are evaluated over long absorption times but was found to have little or no significance in the comparative evaluation of absorption or solution effects by the various solutions considered in this work.

Other solutions were evaluated either in the test tube with the fritted gas-dispersion tube or in the plastic cell. Potassium hydroxide, dilute pyruvic acid, TRIS buffer, phosphate buffers, and ethylene diamine were evaluated as CO₂ absorbers. Leakage for the plastic cell air loop was less than 0.1-percent CO₂ in the 10-minute evaluation period when 5-percent CO₂ was used in the air loop (Figure 19).

Distilled water, 15 ml in the plastic cell, revealed only slight CO₂ absorption when compared to that shown by Figure 18. Acidulated water using 2 ml of 3-percent pyruvic acid to 15 ml of distilled water in the plastic cell produced a curve no different from the leakage curve (Figure 19).

Leakage from the "bubbling test tube" absorption method was 0.25 percent for a 10-minute period (Figure 20). Therefore, significant absorption should indicate a CO₂-percentage change greater than this value. Distilled water, 25 ml in the test tube, showed a



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Part I
0.50 to 0.70 percent CO₂ change in 10 minutes (Figure 18).

Ion-exchange resin IRA-401S, 2.28 grams, was added to the distilled water, producing a suspension of the resin, and the $\rm CO_2$ absorption from a 5-percent $\rm CO_2$ air mixture was recorded (Figures 21 and 22). From the straight part of the curve in Figure 21, we see that an 0.80-percent $\rm CO_2$ change was obtained in 10 minutes. Also, there was a drop from 5-percent $\rm CO_2$ to 3 percent, and the curve was still dropping after 15 minutes.

Carbon dioxide absorption evaluations using the test tube method revealed that with TRIS (tris hydroxymethyl aminomethane) buffer at pH 7.4, the CO₂ absorption was less than for distilled water. Potassium chloride at 0.1 molar was also evaluated for CO₂ absorption by this technique and showed little difference from that of distilled water (Figure 23). However, TRIS buffer at pH 10.3 showed a good absorption curve (Figure 24).

Phosphate buffers containing the potassium and sodium phosphates showed $\mathrm{CO_2}$ absorption roughly corresponding to their pH's. Potassium mono-basic phosphate solution at .06 molar revealed at pH 4.80 the same curve as for distilled water at pH 6.1 (Figure 25). The di-basic potassium phosphate at pH 8.75 showed more absorption of $\mathrm{CO_2}$ than did sodium di-basic phosphate at .06 molar with a pH of 9.0 (Figures 26 and 27). Absorption of $\mathrm{CO_2}$ by ethylene diamino tetraacetic acid made with KOH was comparable to that absorbed by dilute KOH (Figure 28). Urea solution at 1 gram per 25 ml of distilled water revealed a $\mathrm{CO_2}$ reduction of an 0.80-percent $\mathrm{CO_2}$ in the air mixture in 10 minutes (Figure 29). Ethylene diamine (technical grade) was used at 5-percent solution to dilute 5 to 20 ml of distilled water. This dilution had a pH of 11.8. The $\mathrm{CO_2}$ absorption by this solution (Figures 30 and 31) was very good and showed, upon saturation with $\mathrm{CO_2}$, a removal rate similar to that of distilled water. Absorption of $\mathrm{CO_2}$ by 15 ml of 5-percent ethylene diamine with a pH of 11.9 showed good absorption in the plastic cell. The pH of the ethylene diamine solution after saturation with $\mathrm{CO_2}$ was 8.1. Further absorption of $\mathrm{CO_2}$ by this solution at this pH was slight and similar to that of distilled water.

An attempt to regenerate the absorption capacity was made by boiling the ethylene diamine solution for 10 minutes with vigorous stirring. Upon cooling, this solution had a pH of 9.8 and showed further CO_2 absorption capacity; Figure 32 gives the CO_2 removal rate. And, upon continuation of the CO_2 absorption, the pH of this solution was 8.05. A second boiling for 15 minutes with vigorous stirring produced a solution of 9.8 and a regeneration of the CO_2 absorption capacity very similar to the regeneration produced upon the first boiling.

A saturated borax solution was used for CO₂ absorption. Quinn and Jones (Reference 2) obtained tabulated data on CO₂ absorption by salt solutions in which saturated borax solution was indicated to have a CO₂ absorption coefficient of 21.75 as compared to that of 0.98 for potassium chloride at 0.82 molar. Other inorganic salts revealed absorption coefficients comparable to that of KC1. The curve of CO₂ absorption by borax solutions revealed a rate which compares favorably with that of dilute KOH solution (Figures 33 and 34). Removal of CO₂ from the borax solution was not attempted.

The $\mathrm{CO_2}$ removal rates for saturated borax solution were with 1-percent $\mathrm{CO_2}$ air mixtures. With 0.95-percent $\mathrm{CO_2}$, a decrease in $\mathrm{CO_2}$ percentage to 0.37-percent was recorded in 10 minutes. In an additional 10-minute period beyond the first 10-minutes, the percentage of reduction went to 0.17 percent. A repeat run with 0.1 molar borax solution revealed comparable data, in that the $\mathrm{CO_2}$ percentage reduction in a 9-minute period, beginning at 0.67-percent $\mathrm{CO_2}$ air mixture, was reduced to 0.37 percent.



A water "blank" using the bubbling test tube technique showed a 0.05-percent reduction in the CO₂ percentage value in a 10-minute period on the 0 to 1 percent range.

DISCUSSION OF TESTS AND RESULTS

The test apparatus used in this work is directly applicable to a study of solid and liquid $\mathrm{CO_2}$ absorbers. Since one of the requirements of a regenerable $\mathrm{CO_2}$ removal system is that the absorber "absorb and desorb" at least 7.56 grams of $\mathrm{CO_2}$ per 10-minute period, a quantitative relationship can be established to correlate the capacity and effectiveness of this test apparatus to the requirements of a "one-man sized" system. The 1.04-liter internal volume of the absorption system must be correlated with a simulated aerospace vehicle cabin volume of approximately 500 cubic feet (1 cubic feet equals 28.316 liters) which calculates to a volume ratio of 1 to 15000. This figure may be misleading when interpolations are made.

The absorption curves of Tables 3 and 4 present an initial dip in the CO_2 concentration during the first minutes of the time interval (Figure 20). This was due to the dilution process when the air flow was introduced to the absorber container. This container introduced a volume of air with little or no CO_2 and it was analyzed as it was being mixed while the total air volume was recirculating through the loop. To mix completely, 2 to 3 minutes were required, after which the measurements of the CO_2 concentrations were considered valid, and the curve tracings returned to "normal." Extending the curve back to the initial start of the drop would give a continuous CO_2 removal rate coupled with a dilution effect. The test tube method and the plastic cell method revealed this dip in the curve more so than did the solid material absorption bulb method.

From the test data on the curves and tabulations in Tables 2 and 3, the values for CO₂ removal by the various materials show that the molecular sieve (Figure 10) and lithium hydroxide (Figures 8 and 9) are rapid and complete CO₂ removal agents for the short-time interval. Comparable liquid agents are the KOH solutions (Figure 17) and the ethylene diamine solutions (Figures 30, 31, and 32). These results with ethylene diamine are to be expected based upon past experience with CO₂ absorption by amine solutions. Mono-ethanolamine was used by the Navy for CO₂ control in submarine atmosphere control. The reaction kinetics of the absorption and the regeneration processes require further investigation. The stability of the ethylene diamine during regeneration processes and the toxicity of such a system's components would need to be evaluated.

The ion exchange resins used in this investigation were the anionic type with amine groupings. CO_2 was absorbed to some extent but here the CO_2 removal process was complicated in its interpretation since the higher pH's of the strong base resins were undoubtedly an uncounted influence in CO_2 absorption. The slopes of the absorption curves were shallow and over the 10-minute period revealed little CO_2 removal. The results of the resin absorption using the resins suspended in water showed significant CO_2 absorption but at a low rate. Amberlite IRA-401S gave a value of 8.275 cc of CO_2 removed in 6 minutes by a 2-cc quantity of resin suspended in 50 ml of distilled water (Figure 21).

The liquid CO_2 absorption curves indicated that some effects needed further investigation. The borax solutions, the urea solution, and the ethylene diamine solutions indicated CO_2 absorptions significantly more than that of water or the buffer solutions. Ethylene diamine solutions were comparable to that of KOH and showed a regeneration capability. The borax solution was not investigated further.

The solid CO_2 absorber tests indicated that the resins and the o-phenylene diamine had a capacity for absorbing CO_2 . Further evaluations should be conducted to reveal the significance of the CO_2 removal capacities. Cellulose acetate revealed slight CO_2 removal capacity.



The solid absorbers showing rapid CO_2 removal are compared in Table 4 on a minute-by-minute basis to show the rapid initial CO_2 removal from the air. This initial drop in CO_2 percentage in the air loop was not without a dilution effect brought about by the added volume of the absorber container.

TABLE 3

CO₂ REMOVAL BY SOLID ABSORBERS

Agent	Percenta Start	ge of CO ₂	Time (Min.) Interval
Soda Lime, 36.227 g.	1.0 1.0	.01 .01	5 10
Soda Lime canister	5.0	.15	10
LiOH·H ₂ O, 28.674 g.	.98	0	10
LiOH (-H ₂ O) 16.437 g.	.94	0	9
Molecular Sieve, 5A, 36.834 g.	4.9 4.7	.1 .25	10 10
Amberlite IRA 401S 21.17 g.	5,0	4.6	9
Cellulose Acetate 9.05 g.	.95	.88	10
o-Phenylene Diamine 7.0 g.	.93	.88	10
Na ₂ CO ₃ , 32.971 g.	.98	.94	10
Rexyn RG-6 Resin 21.103 g.	1.0	.98	10
Borax, Anhydrous 27.512 g.	1.0	.98	6



TABLE 4

CO₂ REMOVAL BY LIQUID ABSORBERS

AGENT	F	Percentage of CO ₂		Time	
NOBIVI	Start	% Removed in 5 to 10 Min. Int.	End	Interval (Min.)	
Distilled Water,	2.7	.25	1.95	10	
KC1, 0.1 Molar	4.6	.25	2.55	10	
KH ₂ PO ₄ .06 Molar, pH 4.8	4.95	.25	3.9	10	
K₂HPO₄, 0.013M pH 8.75	4.80		3.40	7	
Empty Test Tube	5,00	.1	4.15	10	
Na ₂ HPO ₄ , .06 Molar pH 9.0	4.5	.25	2.90	10	
КОН, .99%	4.6		.05	10	
	4.2		.35	10	
EDTA, 5% in .5% KOH	5.0		3.95	7	
Pyruvic Acid, Dilute	4.65	.05	4.55	10	
Amberlite Resin, 2 g/50 cc Water 2.28 g. per 50 cc Water	er 5.00	.45	3 . 45	10	
2 cc/50 cc Water	3.90	.50	1.20	10	
Borax, Saturated Sol.	.94	.19	.37	10	
Borax, 0.1 Molar Sol.	1.0	.14	.38	10	
TRIS, .06 Molar pH 10.3	5.0	.70	2.25	10	
Urea Sol. 1 g. / 25 ml H ₂ O	4.90	.50	2.50	10	
Ethylene Diamine, 1:5 Dil. of 5% Sol. pH 11.9	4.85		.05	3	
Ethylene Diamine (as Above)	5,00		.20	7	
Ethylene Diamine (Regen.)	4.90	.8	.80	10	



TABLE 5 ${\rm CO_2~ABSORPTION~BY~SOLID~ABSORBERS}$

	Percentage CO ₂						
Time	LiOH		Molecul	ar Sieve	Soda Lime		
Min.	H⁵O	Anhyd.	lst run	2nd run	F-M Bulb	F-M Bulb	Canister
0	.98	.94	4.9	4.7	1.0	.96	5.0
1	.80	.70	3.0	2.8	0.42	.63	3.0
2	.49	.36	2.0	1.9	0.08	.33	1.8
3	.27	.15	1.3	1.3	0,02	.07	.70
4	.13	.06	0.8	0.8	0.01	.03	.45
5.	.08	.02	0.5	0.6	0.01	.02	. 30
6	.04	.01	0.3	0.45		. 01	.20
7	.02	.01	0.2	0.35		.01	.15
8	.01		0.15	0.30			.13
9	.01						0.13



SUMMARY AND CONCLUSIONS

The apparatus which has been described in this report was adequate in the evaluation of CO₂ removal agents for use in a closed air loop. Lithium hydroxide, soda lime, and molecular sieve, 5A, when used as CO₂ removal agents showed rapid and complete CO₂ removal, as illustrated graphically. Other solids tried were cellulose acetate, borax, sodium carbonate, o-phenylene diamine, and two resins of the amine type, Amberlite IRA-401S and Rexyn RG-6 (OH) form. The amine compounds showed a significant CO₂ removing capacity and should be investigated further.

Liquids used for CO_2 removal were solutions of buffer salts, KOH, borax, ethylene diamine, urea, suspensions of the resin (IRA-401S), pyruvic acid, and EDTA in KOH. The buffer salts were KC1, Na_2HPO_4 , KH_2PO_4 , K_2HPO_4 , and TRIS. The curves of the CO_2 absorption revealed rapid and complete removal with KOH and ethylene diamine solutions. The solutions with high pH's showed more CO_2 removal than solutions with pH's near 8 and lower. Acid pH's showed little or no absorption. Borax, TRIS, and urea solution showed CO_2 removal rates which would justify further investigation.

Regeneration of the CO₂ removal capacity was tried with ethylene diamine solution by boiling for 10 to 15 minutes. The CO₂ absorption capacity was restored but the regeneration process requires further investigation.



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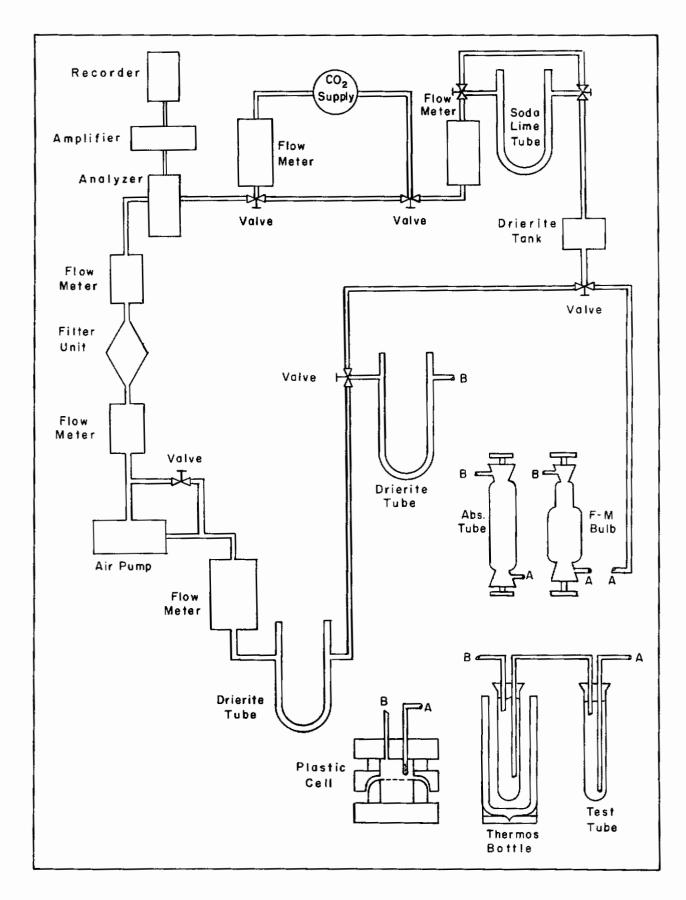


Figure 1. Closed Air Loop For CO₂ Absorber Evaluation 18



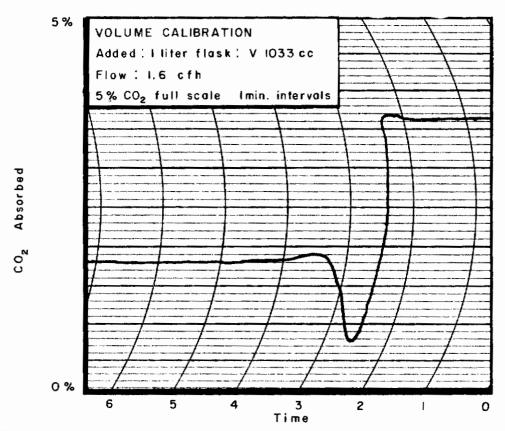


Figure 2. Volume Calibration with 1033 cc Flask on 0 to 5% Range

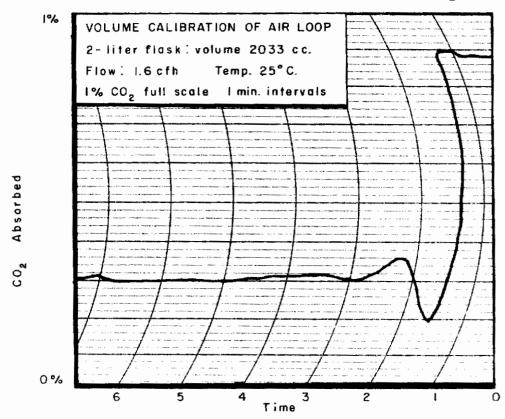


Figure 3. Volume Calibration with 2033 cc Flask on 0 to 1% Range



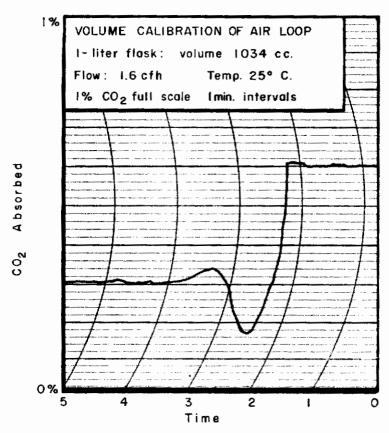


Figure 4. Volume Calibration with 1033 cc Flask on the 0 to 1% Range

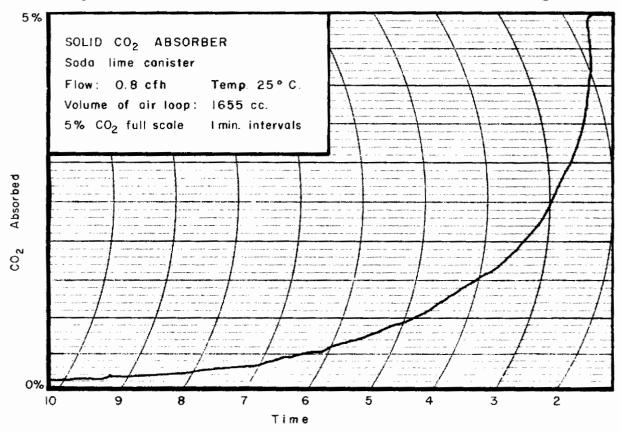


Figure 5. CO₃ Absorption by Soda Lime Canister



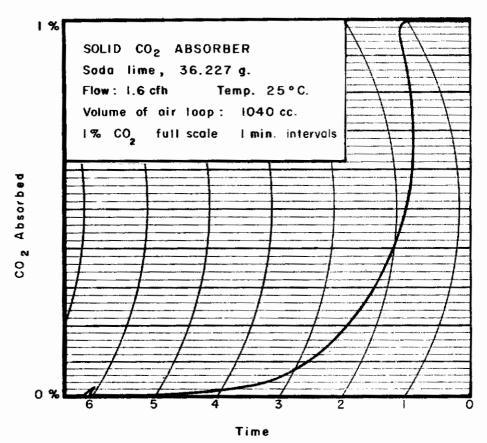


Figure 6. CO₂ Absorption by Soda Lime Bulb

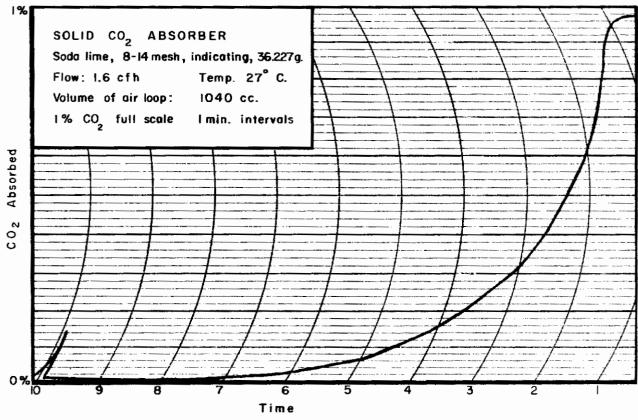


Figure 7. CO₂ Absorption by Soda Lime Bulb, Sequent to Figure 6



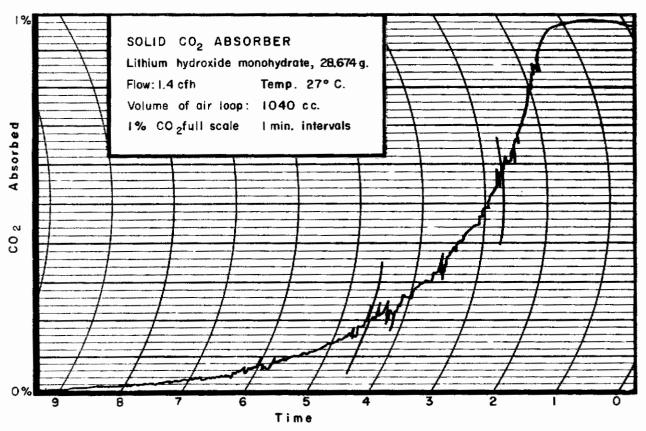


Figure 8. CO₂ Absorption by LiOH·H₂O

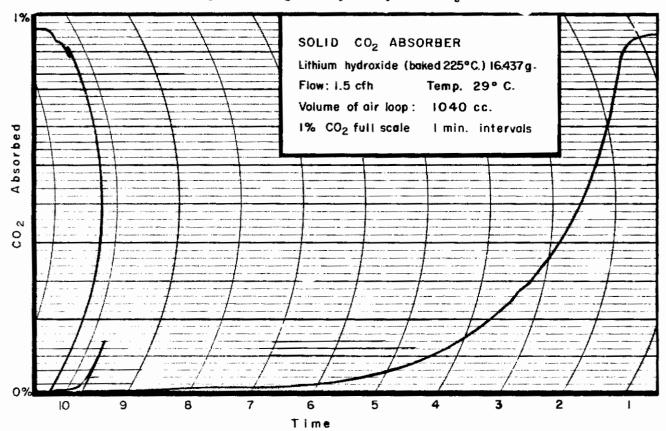


Figure 9. CO₂ Absorption by LiOH Anhydrous 22



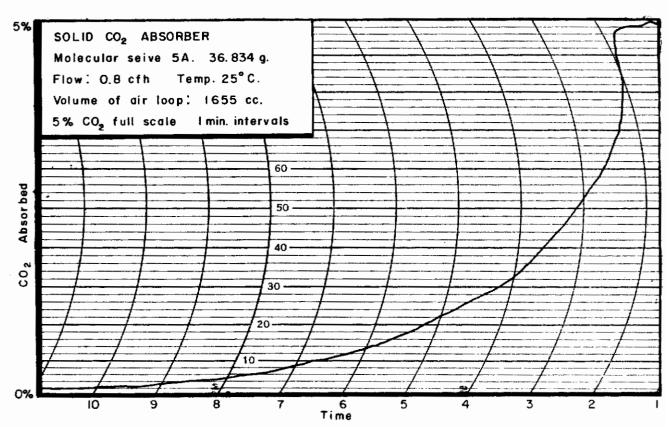


Figure 10. CO₂ Absorption by Molecular Sieve, 5A

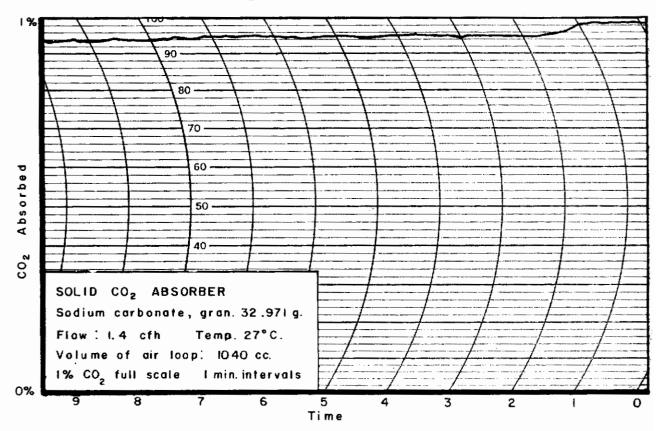


Figure 11. CO₂ Absorption by Na, CO₃, Granular



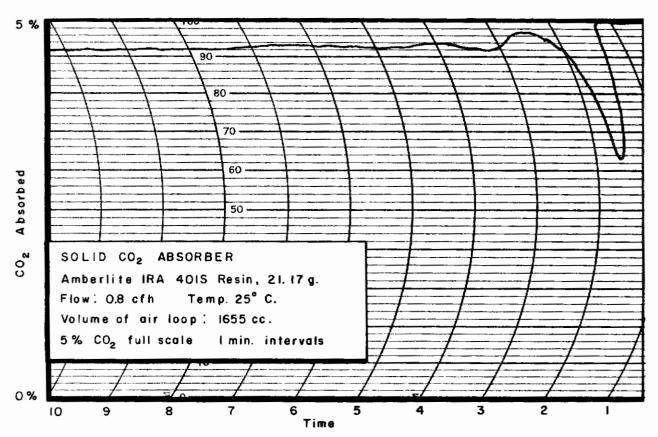


Figure 12. CO₂ Absorption by Amberlite IRA-401S Resin

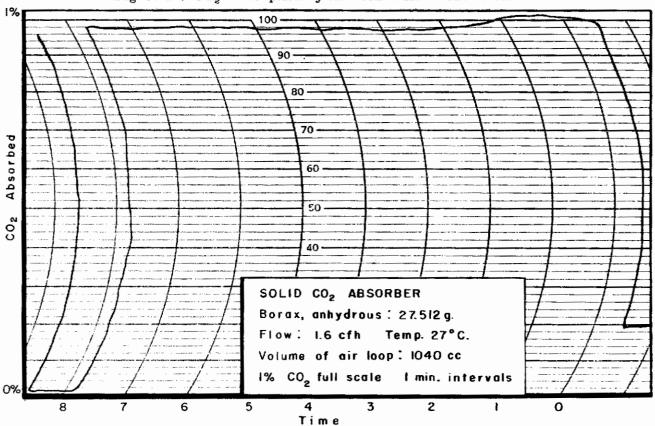


Figure 13. CO_2 Absorption by Borax, Anhydrous



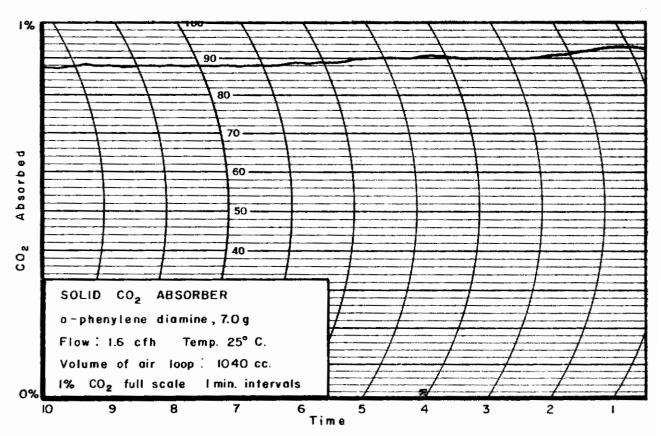


Figure 14. CO₂ Absorption by o-Phenylene Diamine

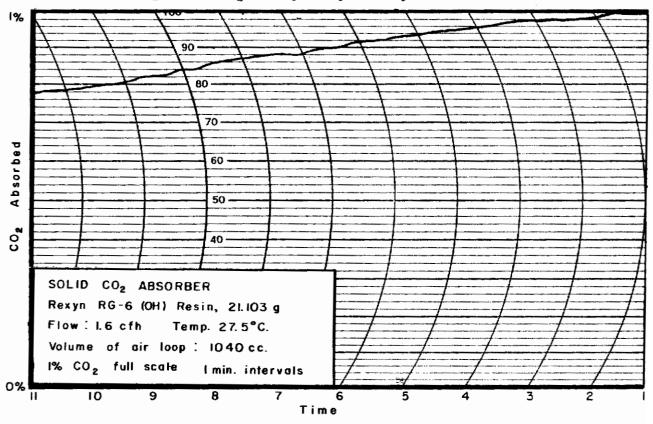


Figure 15. CO₂ Absorption by Rexyn RG-6 (OH) Resin



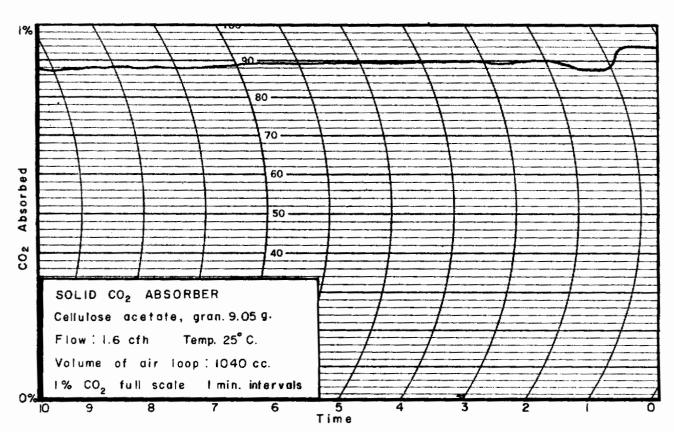


Figure 16. CO₂ Absorption by Cellulose Acetate, Granular

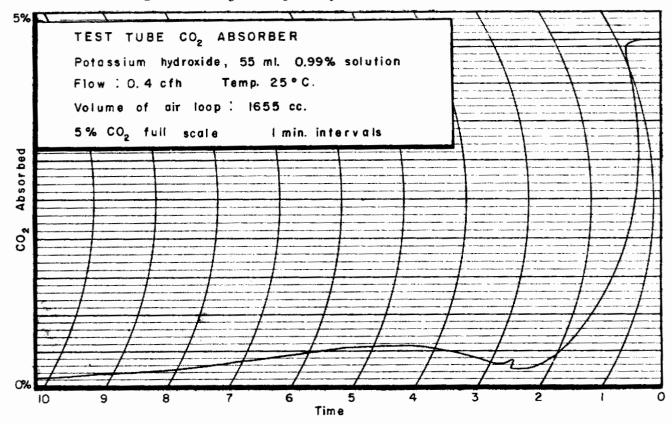


Figure 17. CO, Absorption by KOH Solution



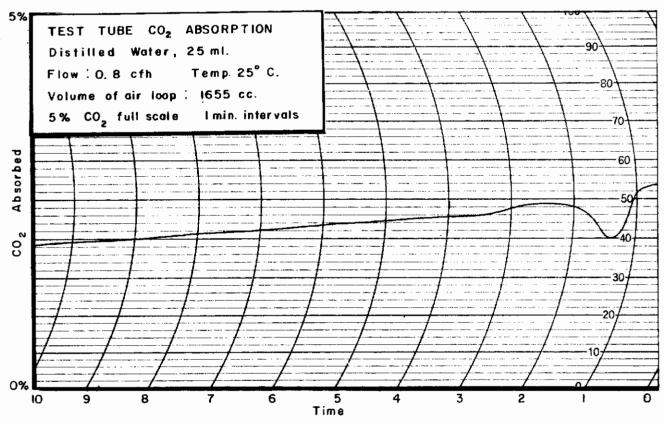


Figure 18. CO₂ Absorption by Distilled Water

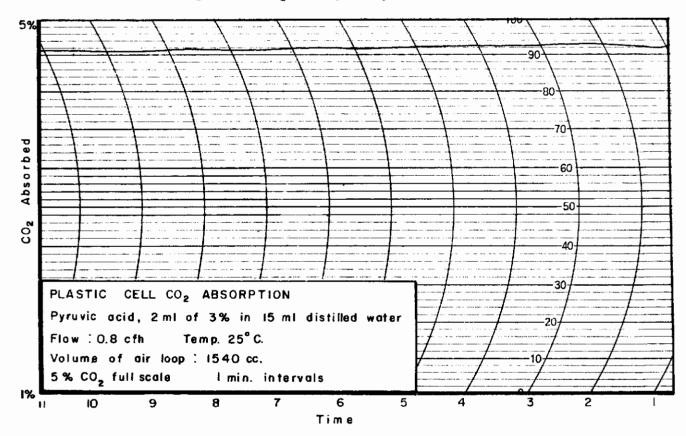


Figure 19. CO₂ Absorption by Dilute Pyruvic Acid 27



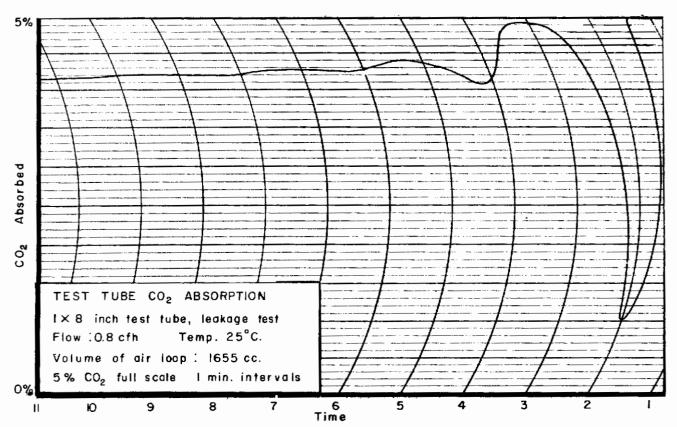


Figure 20. CO₂ Dilution Effect by 1 x 8 Inch Test Tube

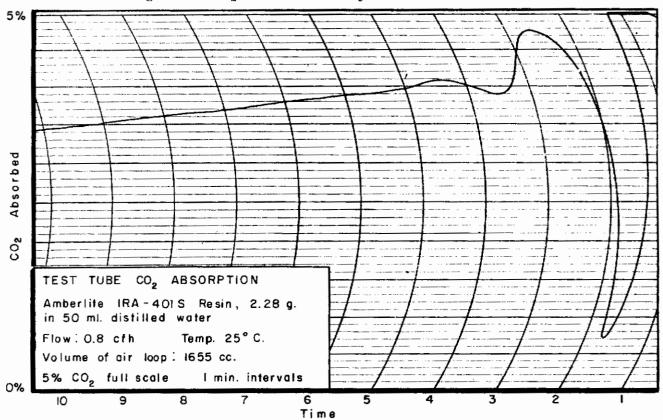


Figure 21. CO₂ Absorption by Aqueous Suspension of Amberlite IRA-401S Resin, 2.28 Grams



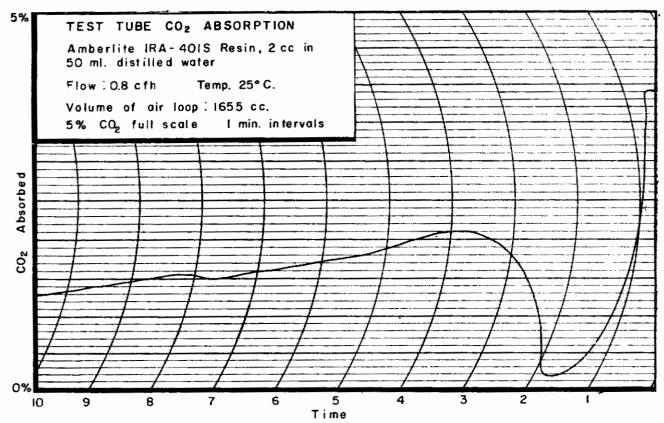


Figure 22. CO₂ Absorption by Aqueous Suspension of Amberlite IRA-401S Resin,

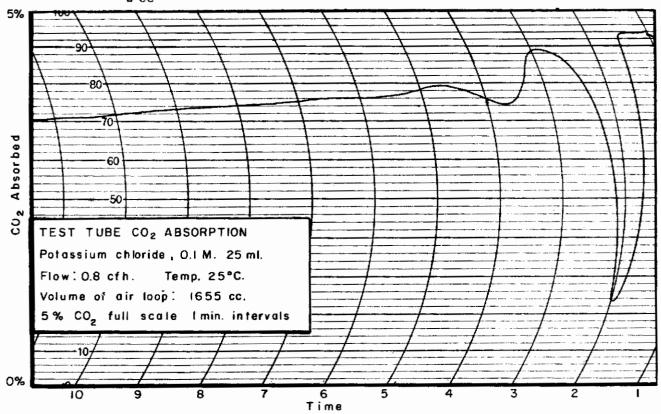


Figure 23. CO₂ Absorption by KC1, 0.1 Molar 29



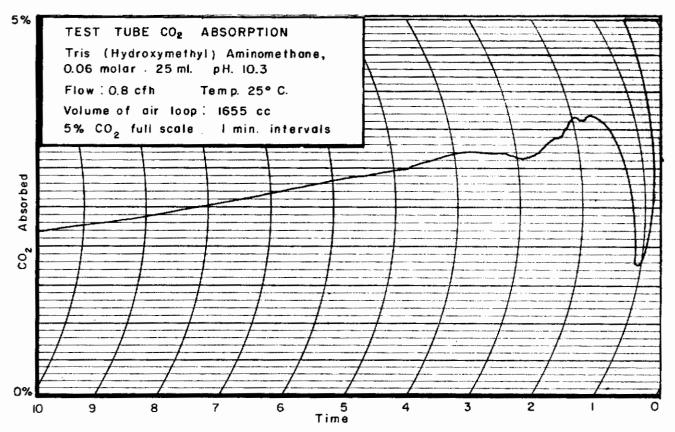


Figure 24. CO. Absorption by TRIS Solution

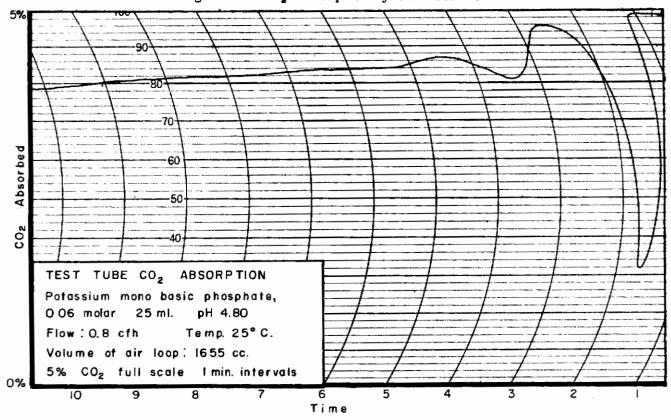
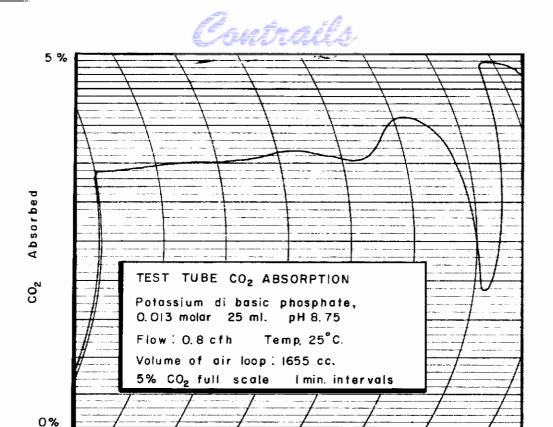


Figure 25. CO₂ Absorption by KH₂PO₄, .06 Molar



Time Figure 26. CO_2 Absorption by K_2HPO_4 , 0.013 Molar

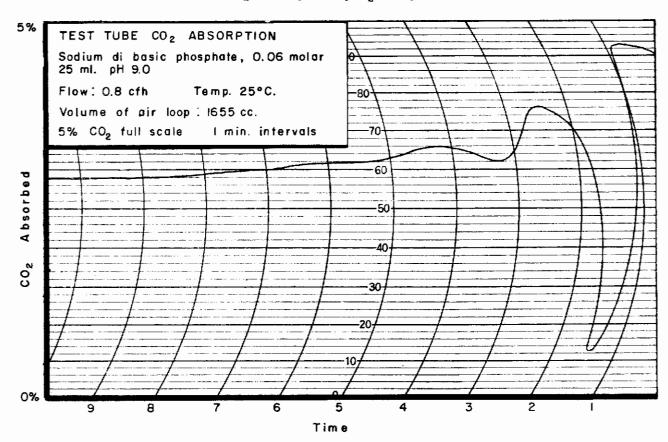


Figure 27. CO₂ Absorption by Na₂HPO₄, .06 Molar



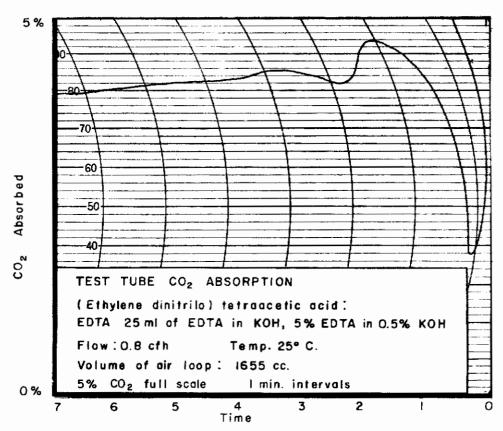


Figure 28. CO₂ Absorption by EDTA, 5% Solution

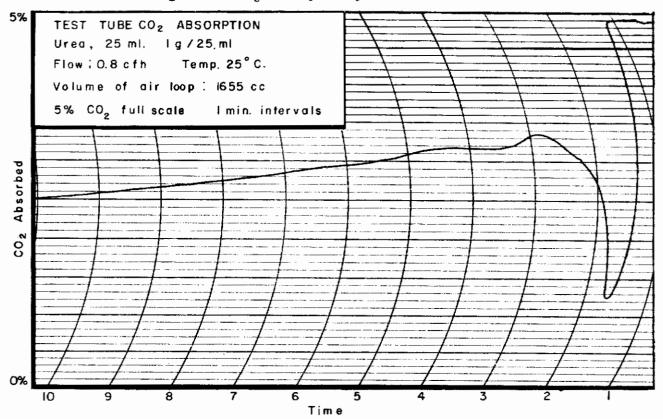


Figure 29. CO₂ Absorption by Urea, 4% Solution 32



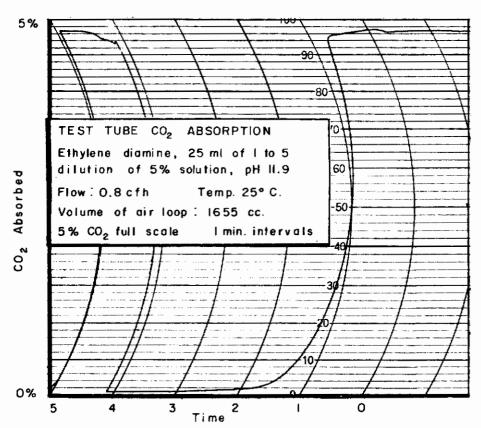


Figure 30. CO₂ Absorption by Ethylene Diamine, 1% Solution

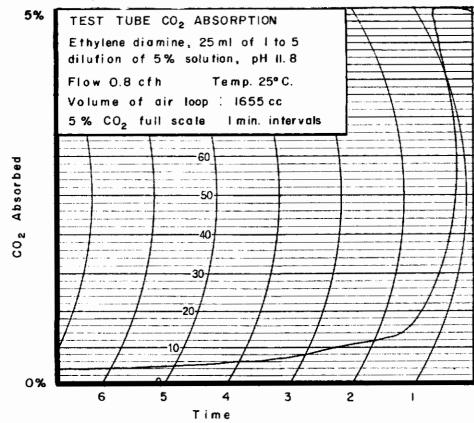


Figure 31. CO₂ Absorption by Ethylene Diamine, 1% Solution, Sequent to Figure 30 33



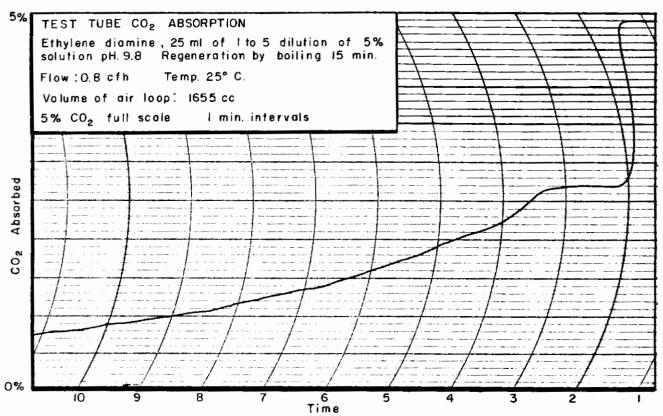


Figure 32. CO₂ Absorption by Ethylene Diamine, 1% Solution, Regenerated by Boiling

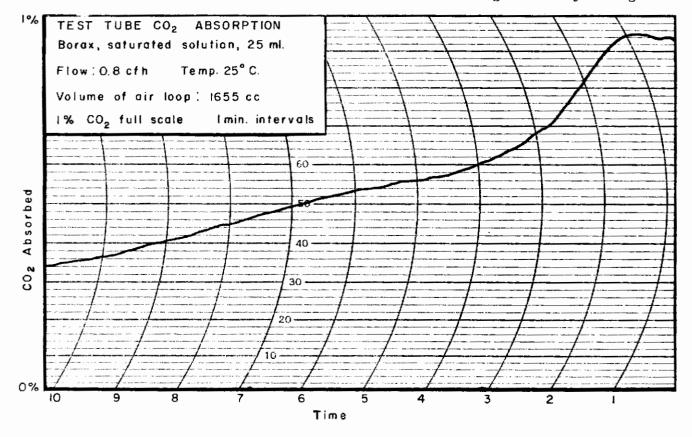


Figure 33. CO₂ Absorption by Saturated Borax Solution 34



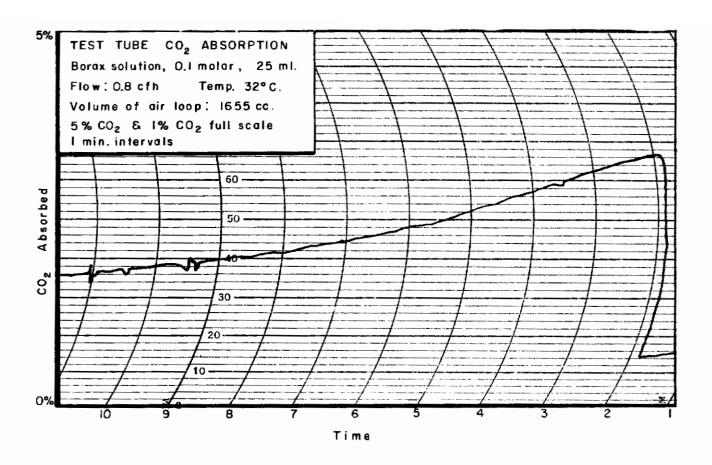


Figure 34. CO₂ Absorption by Borax Solution, 0.1 Molar

