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HAMILTON STANDARD ENGINEERING REPORT
SVRDER 5712

FINAL REPORT
LITHIUM PEROXIDE TEST PROGRAM

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NASA MANNED SPACECRAFT CENTER

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ABSTRACT

This report contains test results defining the operating characteristics of lithium peroxide to the extent required to quantify system level penalties. The effects of chemical catalysts, bed cooling, chemical manufacturing techniques, operating conditions, and chemical handling procedures are evaluated.

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1.0

SUMMARY

During Phase II of the lithium peroxide development program, the effect of chemical catalysts, bed cooling, chemical manufacturing techniques, operating conditions, and chemical handling procedures were evaluated.

The main objective of the catalyst evaluation tests was to identify a catalyst material which would promote oxygen evolution at chemical bed temperatures which are conducive to good carbon dioxide removal performance.

Initial catalyst testing consisted of twenty-two (22) full scale tests which evaluated MnO, FeSO₄, MnO₂, TiO₂, MnO + silver/copper, AgNO₃, and CaO as catalyst materials. Additionally, small scale tests were performed on twenty-four catalyst materials. The results of the catalyst tests indicated that MnO catalyst tended to stabilize test performance and allow for more even oxygen generation, but none of the catalysts evaluated truly promoted low temperature oxygen evolution to the degree necessary to achieve efficient use of the available oxygen.

Bed cooling tests were conducted using passive, passive/dynamic, and dynamic cooling in test canisters both with and without internal heat conduction rods. Passive tests consisted of cooling one face of the test bed to simulate direct sublimator cooling. Passive/dynamic cooling consisted of cooling all four sides of the chemical bed and dynamic cooling utilized liquid cooling coils running through the chemical bed.

Results of thermal control testing showed that low bed temperature (310°F) could be maintained using any of the three cooling configurations if the canister had internal heat conduction rods, but temperatures ran as high as 755°F for the same cooling configuration without internal conducting rods during passive and passive/dynamic testing. This was due to the low thermal conductivity of the lithium peroxide chemical. The low conductivity caused very high temperature gradients between heat removal points and indicated that heat removal paths must be distributed through the chemical bed.

In addition to the thermal control tests, a new lithium peroxide test canister (similar in size to the Portable Life Support System LiOH canister) which employed passive/dynamic cooling and could be recharged without the making or breaking of liquid lines was fabricated and tested. Results of testing showed that this cooling configuration could maintain the chemical bed temperature in the desired operating range.

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1.0 SUMMARY (Continued)

Off-design tests were conducted to evaluate the effect of inlet dew point variation, inlet flow rate variation, and inlet carbon dioxide concentration variation. These tests demonstrated, in general, that carbon dioxide removal performance and oxygen production increase with increased inlet dew point while the actual effect of mass flow rate and carbon dioxide concentration variation appears to be small.

Procedural testing consisted of eight manufacturing process tests and six canister loading process tests. Test results provided the information required to write manufacturing process, chemical storage, and canister loading specifications for lithium peroxide. These specifications are presented in Section 7.0 as an appendix to this report.

The results of this test program indicate that optimum overall performance is obtained with bed temperatures from 390°F to 474°F. Within this range, the higher bed temperatures deter the carbon dioxide removal performance but enhance the oxygen evolution performance due to thermal decomposition of the lithium peroxide. In addition, the MnO catalyst proved to be the most effective catalyst because it tended to stabilize performance and provide maximum oxygen generation (up to 60 per cent of required O₂ for metabolic consumption and leakage). In order to obtain optimum performance from the MnO catalyst, the crewman's metabolic rate must be above 1300 BTU/hr., so as to provide a CO₂ flow rate sufficient to attain a bed temperature above 390°F. A maximum heat removal rate of 600 BTU/hr. is required to maintain optimum bed temperatures.

A comparison between a lithium peroxide system and a lithium hydroxide system using the same canister and test conditions reveals that the Li₂O₂/O₂ system is approximately 16 per cent lighter and occupies about the same volume as the LiOH/O₂ system for the 0.5 mm Hg CO₂ limit; it is about 25 per cent lighter and about 10 per cent smaller for the 4.0 mm Hg condition.

2.0

INTRODUCTION

In an attempt to reduce weight and volume of expendables required for environmental control systems, a number of chemicals have been evaluated to determine their suitability as an oxygen generation/carbon dioxide removal material. Chemicals such as Na_2CO_3 , K_2O_3 , Li_2O_2 , $\text{Ca}(\text{O}_2)_2$, Na_2O_2 , KO_2 , Li_2O_2 , Na_2O_2 , and K_2O_2 have been investigated for this application with varying degrees of success.

The most promising of these materials has been lithium peroxide (Li_2O_2). Testing has shown that the chemical is stable in operation and has high carbon dioxide removal capability (0.96 lb- CO_2 /lb- Li_2O_2) and acceptable oxygen generation potential (0.35 lb- O_2 /lb- Li_2O_2). With these capabilities, a lithium peroxide oxygen generation/carbon dioxide removal system offers substantial reductions in system weight and volume for expendables.

During the previous phase (Phase I) of the lithium peroxide development program, the effect of varying certain parameters such as test bed geometry, operating temperature, chemical bulk density, inlet dew point, and addition of catalysts was evaluated. It was found during Phase I that chemical performance was a function of bed geometry, bed temperature, bulk density, and inlet dew point. Also, the addition of catalyst materials such as NiSO_4 seemed to improve chemical performance.

Phase II of the lithium peroxide test program extended the results of the Phase I study. Catalyst addition was studied in greater detail with the final objective of isolating an optimum catalyst material. Means of controlling bed temperature were also evaluated. The effect of chemical manufacturing and handling techniques, as well as the effect of off-design operating conditions also were investigated during the Phase II study.

The results of the Phase II study are presented in the following sections. Test data results are presented in Section 4.0 and the analysis of these results is presented in Section 5.0. Conclusions and recommendations resulting from the Phase II study are presented in Section 6.0. Specifications for lithium peroxide manufacture, storage, and cartridge loading are presented in Section 7.0 as an appendix to this report.

3.0 PROGRAM DEFINITION

This section defines the program objectives, test objectives, program description, test conditions, test facility, test hardware and planned test sequence.

3.1 Program Objectives

The overall program objective was to define the operating characteristics of lithium peroxide to the extent required to quantify system level penalties incurred with a lithium peroxide removal/O₂ supply system. The specific objectives were:

- . To advance the state-of-the-art in life support system technology.
- . To develop specific components and subsystems for incorporation into an advanced portable life support system.

3.2 Test Objectives

The following objectives were established for the test program:

- . Determine an optimum catalyzing agent, considering MnO, FeSO₄, MnO₂, and NiSO₄, to promote oxygen evolution.
- . Establish an optimum method of heat transfer to maintain the bed within the desired operating range, utilizing the techniques described herein.
- . Conduct an analysis via gas chromatography, of the effluent gas to determine if either noxious or toxic gas constituents are present.
- . Generate procedural documents which specify chemical handling requirements, canister loading procedure, and the granule manufacturing process requirements.
- . Evaluate off-design performance of the optimum material.
- . Generate data to quantify all system level penalties accrued by the Li₂O₂ subsystem (e.g. - establish the feed water requirement due to the heat of reaction of the chemical).
- . Evaluate the feasibility of a bed cooling technique which will allow chemical recharge without requiring disconnection or connection of fluid cooling lines.

3.3 Program Description

The test program, which had a sum total of 69 tests, was composed of the following set of test series.

1. Catalyst Evaluation
2. Bed Cooling Evaluation
3. Procedural Tests
4. Off-Design Evaluation
5. End Item Canister Evaluation

A test summary is given in Section 3.7.

Catalyst Evaluation

Catalyst evaluation tests were conducted to evaluate the effect of the addition of certain materials to lithium peroxide in order to promote low temperature oxygen evolution. The chemicals evaluated as catalyst materials during full scale testing were manganese oxide (MnO), iron sulfate ($FeSO_4$), manganese dioxide (MnO_2), titanium dioxide (TiO_2), manganese oxide plus silver coated copper, silver nitrate ($AgNO_3$), and calcium oxide (CaO). Approximately fifty catalyst substances were evaluated prior to selecting the above materials for full scale testing.

Catalyst testing was conducted at a metabolic rate of 2000 BTU/hr. with the final three tests run using a variable metabolic profile. Various bed operating temperatures were utilized for each catalyst material in order to determine an optimum temperature range associated with each catalyst.

Bed Cooling Evaluation

A thermal control evaluation was conducted to determine the most effective way to cool the chemical bed. Three different means of bed thermal control were evaluated. These were passive cooling, passive/dynamic cooling, and dynamic cooling. For passive and passive/dynamic cooling, test canisters both with and without internal heat conduction rods were evaluated. Passive cooling consisted of cooling one face of the test canister to simulate the use of a sublimator. Passive/dynamic cooling consisted of cooling all four sides of the test canister, while dynamic cooling utilized internal cooling coils through the chemical bed.

Procedural Tests

Tests were conducted to establish the criteria for and verify the validity of specifications which were formalized for material storage, material manufacture, and cartridge loading. The sum total for this test series was 14 tests, all at baseline conditions, and was made up of the following tests.

3.3 Program Description (Continued)

1. Manufacturing process - The granule manufacturing process was closely monitored to accumulate the information needed to generate a formal procedure. Two distinct granule sizes, each made with an appropriate percentage of two different binders, were tested twice for each combination of granule size and binder for a total of eight tests. These tests were to verify the performance repeatability of granules manufactured per the process specification.
2. Canister loading process - Canisters were loaded, one-third of a bed at a time, and vibrated at three distinct vibration levels. Two tests were run for canisters loaded at each distinct vibration level to determine the optimum loading technique. A total of six tests were performed and a formal loading procedure was generated based on these results.

Off-Design Evaluation

The following off-design tests were conducted to obtain additional operational information. A total of nine tests were run during this series.

1. Inlet water vapor variation - A total of three tests at baseline conditions were conducted with the following levels of water vapor flow rate: no water vapor present, 50°F and 85°F dew points.
2. Inlet CO₂ variation - A total of four tests at baseline conditions were run for the following levels of CO₂ flow rate: No CO₂, CO₂ corresponding to metabolic rates of 500, 1000 and 3000 BTU/hr.
3. Total flow rate variation - A total of two tests were run, using the variable metabolic profile, with the system flow rate at five cfm for one test and nine cfm for the other.

End Item Canister Evaluation

A new Li₂O₂ test canister design which allowed recharging of the liquid cooled cartridge without the making or breaking of liquid lines was fabricated and tested. A total of seven tests were run to demonstrate the feasibility of the new canister design. This test canister was delivered as a contract end item upon completion of testing.

3.4 Test Conditions

This section defines the test conditions employed for both the constant and variable metabolic profiles employed during the test program.

Constant Metabolic ProfileFull Scale

Total Gas Flow Rate (lb/hr)	8.4 ± 0.2
Inlet Gas Temperature ($^{\circ}\text{F}$)	85 ± 2
Inlet Pressure (3.7 psia nom.)	191 ± 2
Inlet Dew Point ($^{\circ}\text{F}$)	70 ± 1
CO_2 Flow Rate (lb/hr)	(0.39 ± 0.02)
	(0.31 ± 0.01)

Variable Metabolic Profile

<u>Metabolic Expenditure (BTU/hr)</u>	<u>Mission Duration (min)</u>
2000	45
2500	30
500	15
2000	45
2500	30
500	15
2500	60

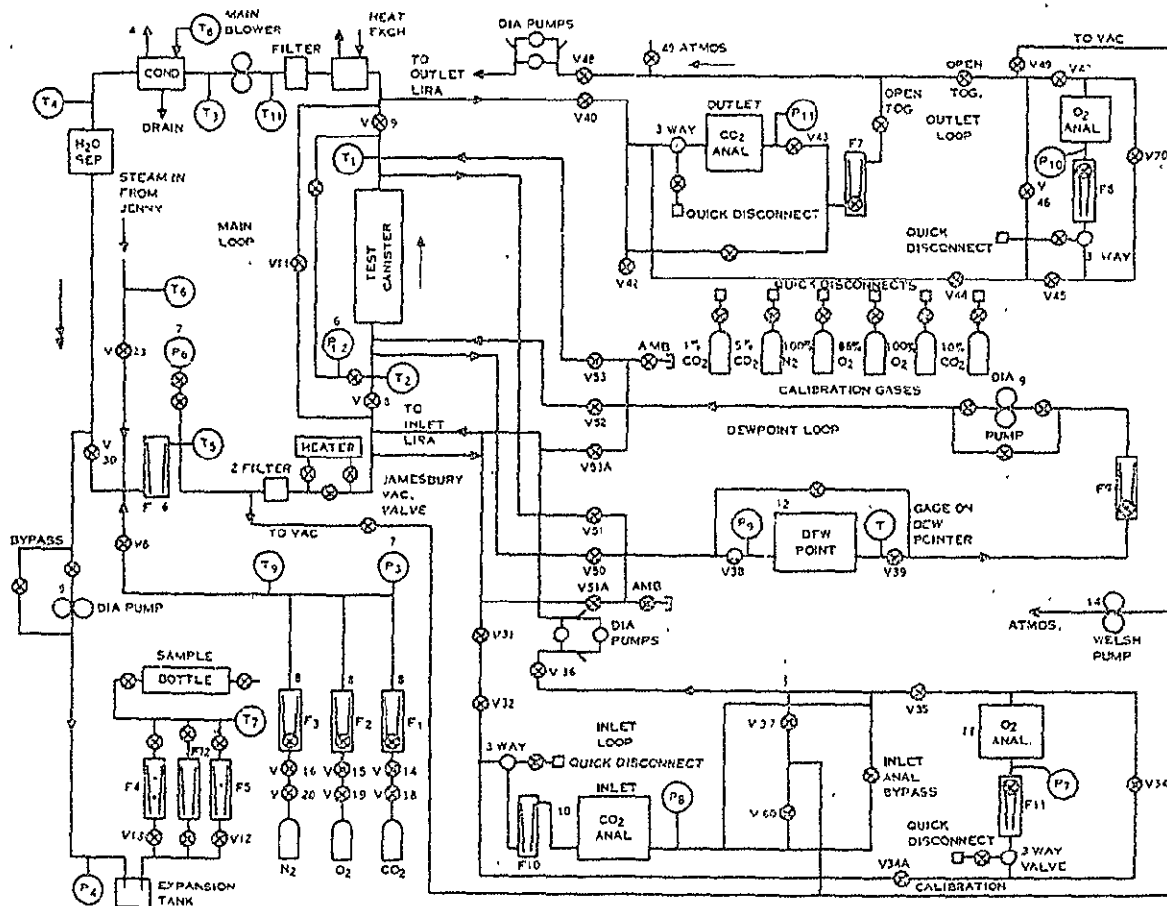
For the variable profile tests, the total rate, inlet temperature, and pressure are the same as for the constant metabolic profile. The carbon dioxide flow rate and the dew point are as defined below:

<u>Metabolic Rate (BTU/hr)</u>	<u>Dew Point ($^{\circ}\text{F}$)</u>	<u>CO_2 Flow Rate (lb/hr)</u>
500	55.	0.98 ± 0.005
2000	70.	0.39 ± 0.02
2500	80.	0.488 ± 0.02

3.5 Test Facility

The testing was performed using the Li_2O_2 test rig (Fig 21) which is shown schematically in Figure 3-1 and is depicted in Figures 3-2 and 3-3. A close-up of a canister mounted in the rig is shown in Figure 3-4. This test facility is a closed loop system having a total volume of about 1.5 ft³ to simulate the internal volume of a space suit and portable life support system. The metabolic processes of the crewman are simulated by feeding carbon dioxide and water vapor into the system at the desired metabolic level and simultaneously bleeding gas from the system to account for the crewmen's metabolic oxygen consumption. All gas conditions

Figure 3-1



Lithium Peroxide Test Rig Schematic

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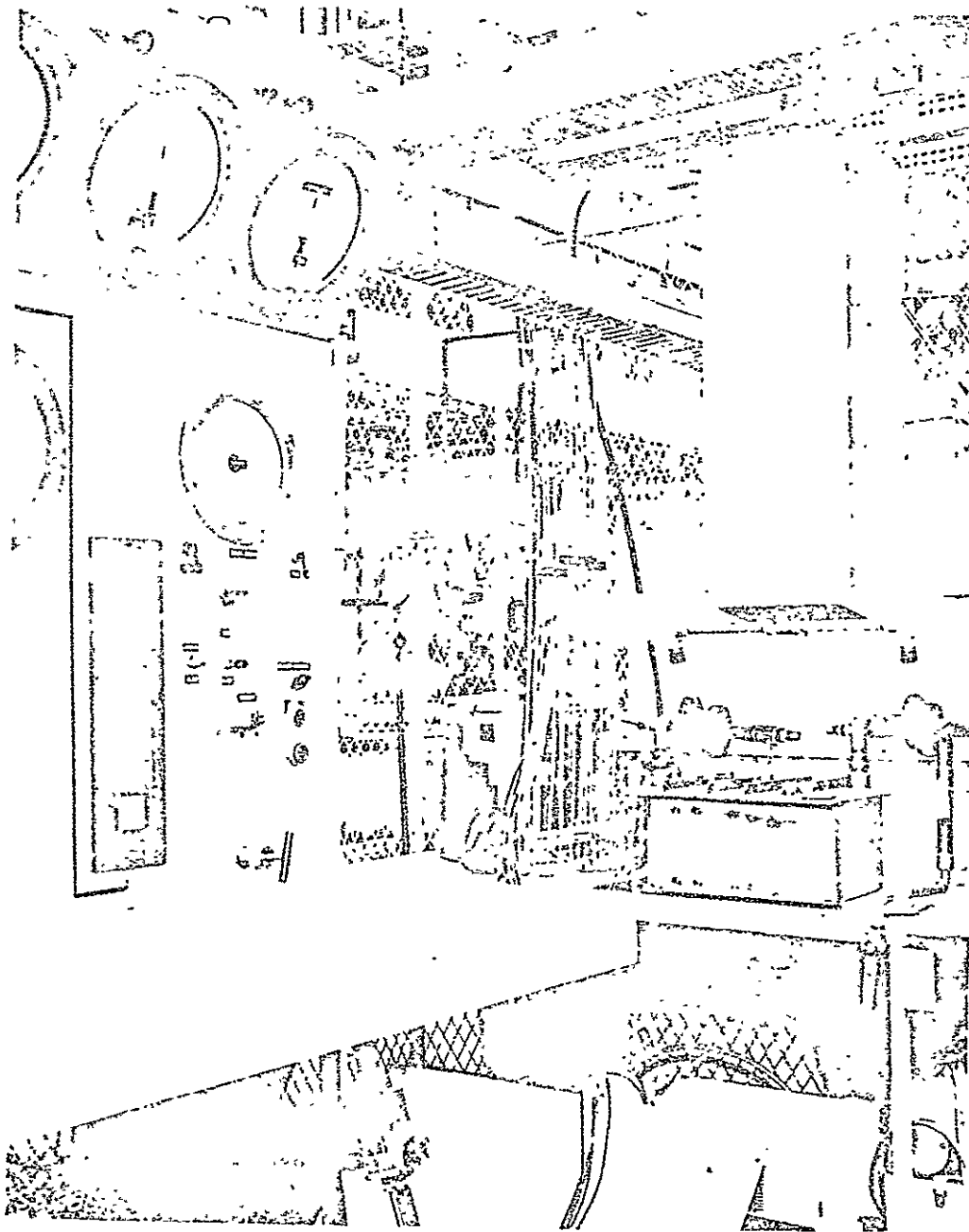
Division of United Aircraft Corporation
P.O. Box 1000
Bridgeport, Conn. 06602

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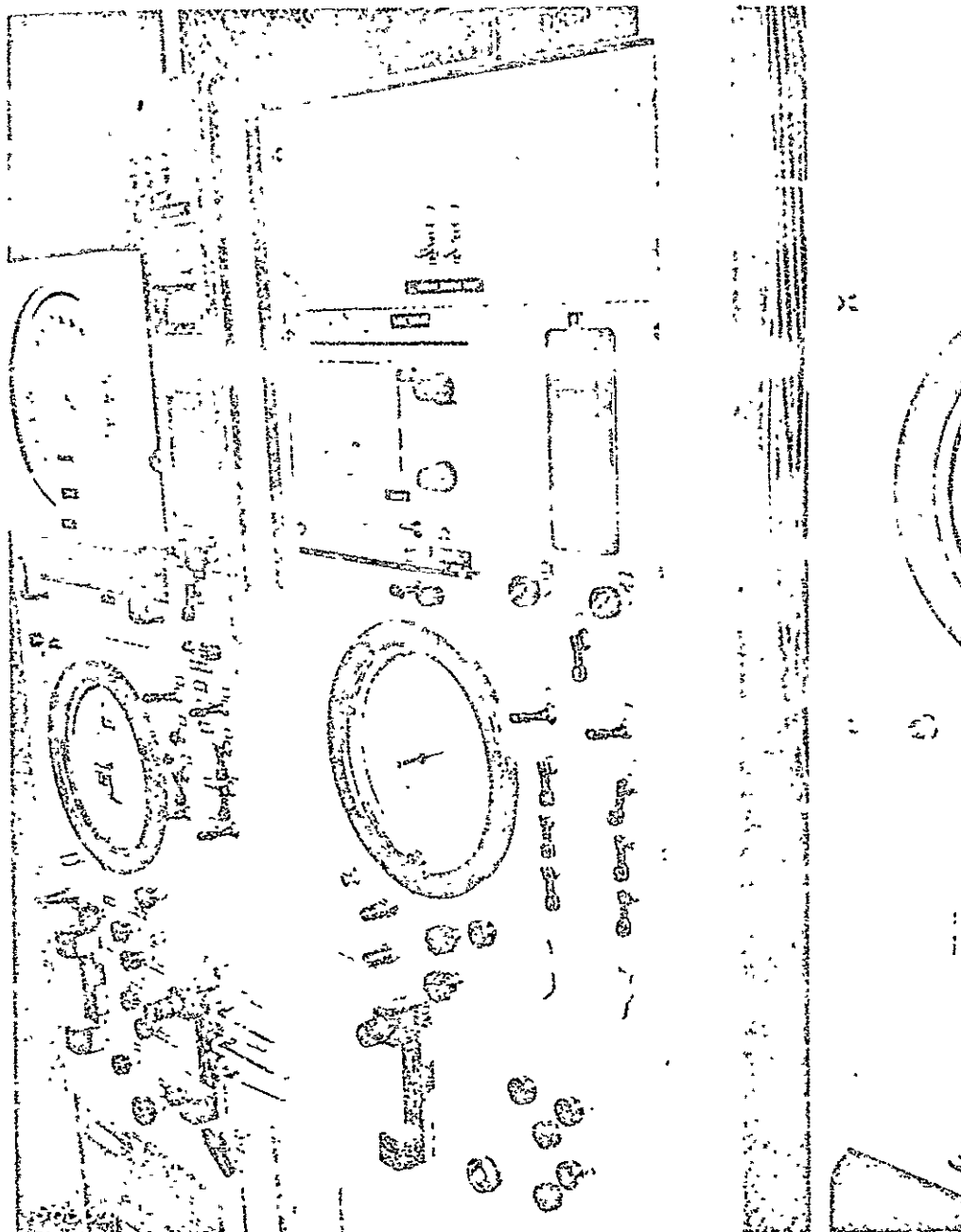


View of Flow Meter Panel and Canister in Rig

Figure 3-2

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View of Inlet and Outlet Instrument Panels

Figure 3-3

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Li_2O_2 Test Canister Mounted In The Test Rig

3.5 Test Facility (Continued)

into the lithium peroxide canister are identical to those which would be the effluent from a space suit for the metabolic conditions being tested. Included in the rig instrumentation are the following:

<u>Instrument</u>	<u>Accuracy</u>
1. O ₂ Bleed Flowmeter	± 2% F.S.
2. CO ₂ Make-up Flowmeter	± 2% F.S.
3. Diluent Make-up Flowmeter (N ₂ , He)	± 2% F.S.
4. Loop Sample Flowmeter	± 2% F.S.
5. Loop Sample Flowmeter	± 2% F.S.
6. Loop Flow Flowmeter	± 2% F.S.
7. Pressure Gage (Delta P ₁₋₂)	± 5 mm Hg
8. Pressure Gage (P ₃)	± 0.15 psi
9. Pressure Gage (P ₄)	± 0.15 psi
10. Pressure Gage (P ₆)	± 0.15 psi
11. Pressure Gage (P ₇)	± 0.15 psi
12. Pressure Gage (P ₈)	± 0.15 psi
13. Pressure Gage (P ₉)	± 0.15 psi
14. Pressure Gage (P ₁₀)	± 0.15 psi
15. Pressure Gage (P ₁₁)	± 0.15 psi
16. Temperature Indicator	± 1°F
17. Thermocouples (All)	± 1°F
18. Dewpointer (Cambridge)	± 5%
19. CO ₂ Analyzer (MSA Lira)	± 5%
20. CO ₂ Analyzer (MSA Lira)	± 5%
21. Beckman O ₂ Analyzer	± 0.5%
22. Beckman O ₂ Analyzer	± 0.5%
23. Scale	± 1/16 lb.
24. Speedmax Temperature Indicator	± 2°F
25. Cooling Water Flowmeter	± 2% F.S.

3.6 Test Hardware

A total of 4 test canisters were used during the program. They are shown in Figures 3-5 through 3-9. Test canisters number 1 and 2 were fabricated from aluminum while canister number 3 and the prototype cooling canister were fabricated from stainless steel. Canister number 2 contained internal heat conduction rods. The dimensions of these canisters are defined as follows:

Canister No. 1

Flow area = 50 in² (5 in x 10 in)
Maximum Length = 10 1/4 in.
No internal heat conduction rods

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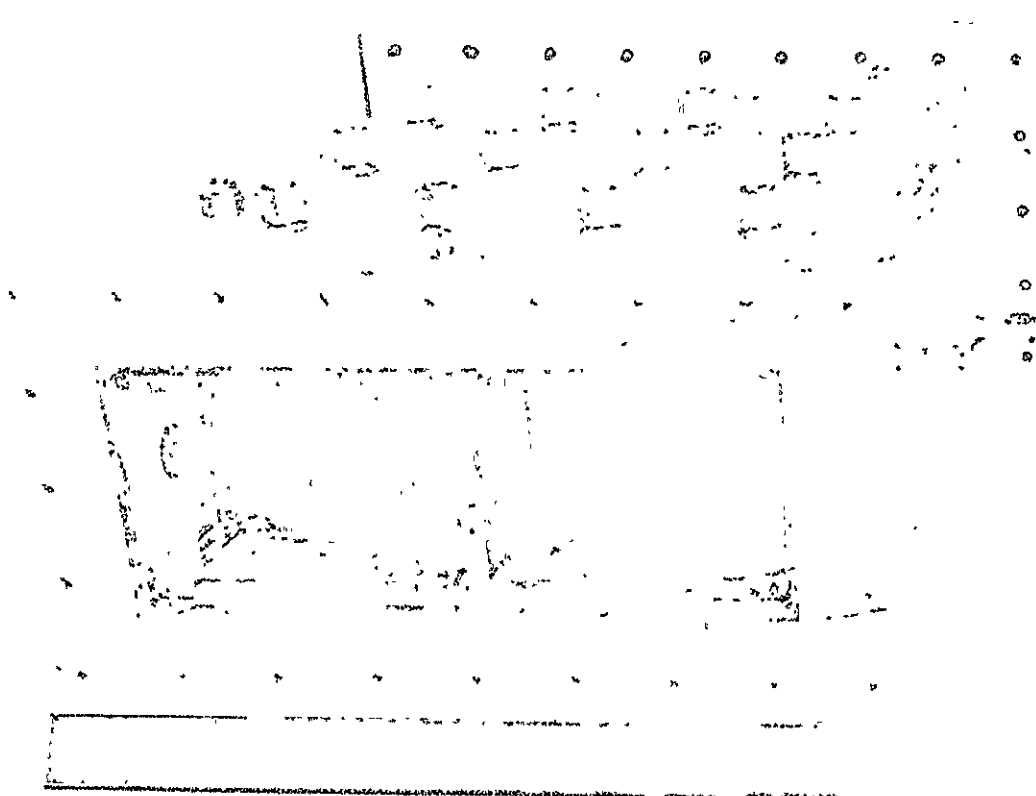
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Canister #1
Inc internal heat conduction prior

Figure 4-5

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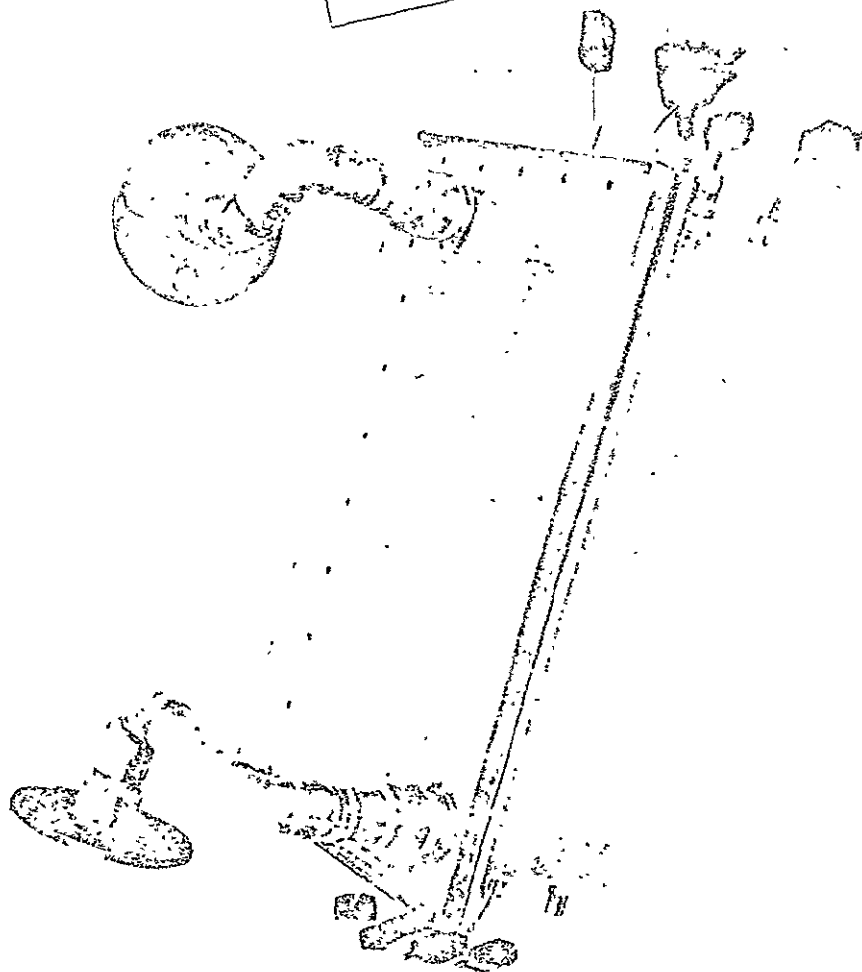
Canister #2
(with internal heat conduction rod)

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STEP 6/11

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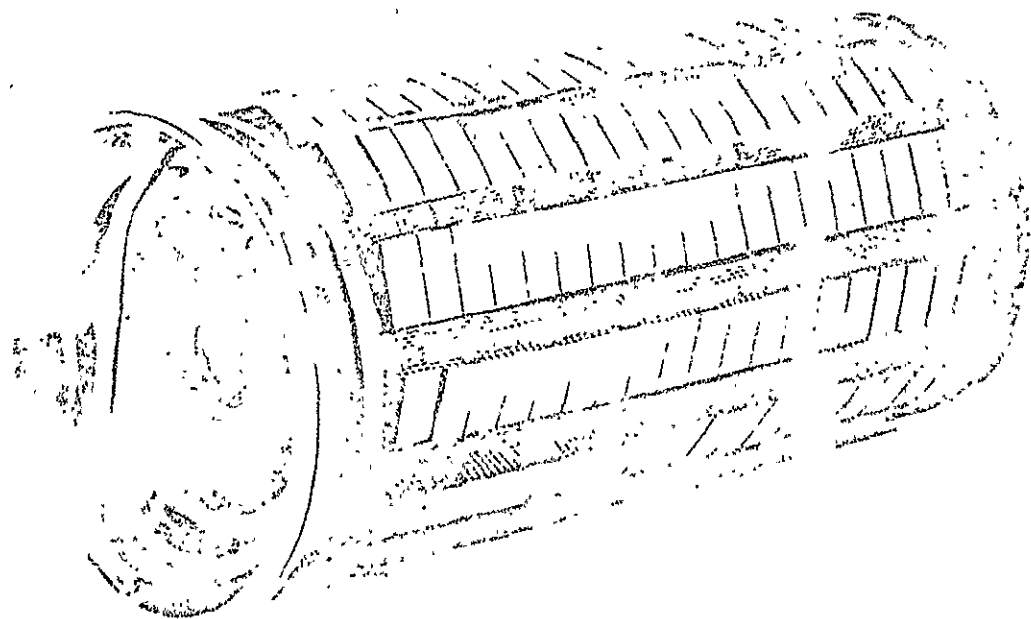


Li₂O₂ Test Canister #3

Figure 3-7

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Prototype Cooling Cartridge
(inlet air)

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Prototype Cooling Canister
(Canister #4)

Figure 3-9

3.6 Test Hardware (Continued)

Canister No. 2

Flow area = 50 in² (5 in x 10 in)
Maximum Length = 10 1/4 in.
With internal heat conduction rods

Canister No. 3

Flow area = 92.5 in² (6.6 in x 14.0 in)
Maximum Length = 4 in.
No internal heat conduction rods

Canister No. 4

Diameter = 4.6 in.
Length = 10.0 in.
Radial flow configuration - Prototype cooling configuration

3.7 Planned Test Sequence

This section defines the test sequence as originally planned.

The following designations are defined to simplify the description of each test.

- a. Baseline Condition - The metabolic profile for the test is constant at 2000 BTU/hr. (conditions per Section 3.4).
(Metabolic rate is 1600 BTU/hr. for testing performed after January 15, 1970)
- b. Variable Profile - The variable metabolic profile is defined per Section 3.4.
- c. Canisters 1, 2, 3 and 4 (defined in Section 3.6) are designated as C1, C2, C3 and C4 respectively.
- d. BT - Lithium peroxide bed temperature.
- e. Subscripts a, b, c, etc. are used to define repetitive testing to the same test description.
- f. TED - To be determined after more information is available.

3.7 Planned Test Sequence (Continued)

Test Sequence

Catalyst Evaluation

<u>Test No.</u>	<u>No. of Tests</u>	<u>Description</u>
MSC2-1	2	Baseline conditions - MnO catalyst-low cooling
MSC2-2	2	Repeat MSC2-1, except high cooling
MSC2-3	2	Repeat MSC2-1, except FeSO ₄ catalyst
MSC2-4	2	Repeat MSC2-3, except high cooling
MSC2-5	2	Repeat MSC2-1, except MnO ₂ catalyst
MSC2-6	2	Repeat MSC2-5, except high cooling
MSC2-7	2	*Baseline conditions - composite 1.3-low cooling
MSC2-8	2	*Repeat MSC2-7, except high cooling
MSC2-9	1	*Repeat best test of MSC2-7 or MSC2-8
MSC2-10	<u>3</u> 21	Variable metabolic profile - best material - sample effluent for contaminants

* Or evaluate other cooling levels with the most promising catalyst

Bed Cooling Evaluation

<u>Test No.</u>	<u>No. of Tests</u>	<u>Description</u>
<u>Passive Cooling</u>		
MSC2-11	2	Baseline conditions - passive cooling - without conductive members
MSC2-12	2	Repeat MSC2-11, except with conductive members
<u>Passive/Dynamic Cooling</u>		
MSC2-13	2	Baseline conditions - passive/dynamic cooling - without conductive members
MSC2-14	2	Repeat MSC2-13, except with conductive members
<u>Dynamic Cooling</u>		
MSC2-15	2	Baseline conditions - cools perpendicular to gas flow - low temperature coolant
MSC2-16	2	Repeat MSC2-15, except high temperature coolant
MSC2-17	2	Repeat MSC2-15, except cools parallel to gas flow
MSC2-18	2	Repeat MSC2-17, except high temperature coolant
MSC2-19	1	Repeat best cooling test
MSC2-20	<u>3</u> 20	Variable metabolic profile - best cooling technique

3.7 Planned Test Sequence (Continued)

<u>Test No.</u>	<u>No. of Tests</u>	<u>Description</u>
<u>Manufacturing Process</u>		
MSC2-21	2	Baseline conditions - granule size A - binder X
MSC2-22	2	Repeat MSC2-21, except with binder Y
MSC2-23	2	Repeat MSC2-21, except granule size B
MSC2-24	2	Repeat MSC2-23, except with binder Y
<u>Canister Loading Process</u>		
MSC2-25	2	Baseline conditions - vibration level 1
MSC2-26	2	Repeat MSC 2-25, except with vibration level 2
MSC2-27	2	Repeat MSC2-25, except with vibration level 3
<u>Off-Design Evaluation</u>		
<u>Test No.</u>	<u>No. of Tests</u>	<u>Description</u>
<u>Inlet Water Vapor Variation</u>		
MSC2-28	1	Baseline conditions - no water vapor present
MSC2-29	1	Baseline conditions - 50°F dew point
MSC2-30	1	Baseline conditions - 85°F dew point
<u>Inlet CO₂ Variation</u>		
MSC2-31	1	Baseline conditions - no CO ₂ present
MSC2-32	1	Baseline conditions - CO ₂ flow rate for 500 BTU/hr.
MSC2-33	1	Baseline conditions - CO ₂ flow rate for 1000 BTU/hr.
MSC2-34	1	Baseline conditions - CO ₂ flow rate for 3000 BTU/hr.
<u>Total Flow Rate Variation</u>		
MSC2-35	1	Baseline conditions - 5 cfm flow rate
MSC2-36	1	Baseline conditions - 9 cfm flow rate
<u>End Item Canister Evaluation</u>		
MSC2-37	2	Baseline Conditions
MSC2-38	2	Variable Profile
MSC2-39	1	To be determined
69 Tests Total		

3.8

Test ResultsData Reduction

Reduction of the test data was accomplished using the Li_2O data reduction computer program, H-137. This program, run on the UNIVAC 1108, performed the analytical reduction of the data and the performance results are provided in both tabular and graphical form as a function of time. The performance curves included the following for each test:

1. Li_2O_2 Canister Outlet CO_2 Partial Pressure vs. Time
2. Li_2O_2 Canister O_2 Generator Rate vs. Time
3. Li_2O_2 H_2O Removal Rate vs. Time
4. Total CO_2 Removed vs. Time
5. Total O_2 Generated vs. Time
6. CO_2 Utilization Efficiency vs. Time
7. O_2 Utilization Efficiency vs. Time
8. CO_2 Removal Efficiency vs. Time
9. CO_2 Removal Rate vs. Time
10. Li_2O_2 Canister Inlet Temperature vs. Time
11. Li_2O_2 Canister Outlet Temperature vs. Time
12. Li_2O_2 Canister Inlet Pressure vs. Time
13. Li_2O_2 Canister Inlet Flow Rate vs. Time
14. Li_2O_2 Canister Inlet H_2O Vapor Flow Rate vs. Time
15. Li_2O_2 Canister Inlet CO_2 Flow Rate vs. Time
16. Li_2O_2 Canister Inlet CO_2 Partial Pressure vs. Time
17. Li_2O_2 Canister Heat Removal Rate vs. Time
18. Li_2O_2 Bed Temperature vs. Time

Chemical AnalysisGas Sample Analysis

Periodic gas samples were taken from the test rig loop and stored for chemical analysis after the test was completed. These were analyzed for N_2 , O_2 , CO_2 , CO , H_2O and trace constituents with a gas chromatograph.

 Li_2O_2 Chemical Analysis

Samples of the test beds were collected before and after testing and analyzed for chemical composition. The results of these chemical analyses were correlated with the measured test results to assure the validity of the performance results.

4.0 TEST DATA PRESENTATION

This section presents the data for the 81 actual tests which were completed during this program. The data for carbon dioxide removal, oxygen generation, maximum bed temperature, and heat removal rate is shown in Figures 4-1 through 4-81 for all tests.

4.1 Performance Data

Table 4-1 is a tabulation of the major parameters for each test and the columns of this tabulation are defined as follows:

- (1) Test number
- (2) Canister number
- (3) Time in the test that the canister outlet partial pressure of carbon dioxide reached 0.5, 1.0 and 4.0 mmHg. (minutes)
- (4) Chemical weight (lb)
- (5) Average useable oxygen generation rate (lb/hr). This excludes all oxygen that was vented overboard during peak generation rates.
- (6) Test duration that the oxygen supply requirement (0.36 lb/hr) was exceeded (minutes).
- (7) Total quantity of useable oxygen produced during 4 hours, or at test termination if earlier (lb).
- (8) Total quantity of oxygen generated during 4 hours, or at test termination if earlier (lb).
- (9) Oxygen utilization efficiency at 4 hours, or at test termination if earlier (Percent of theoretical capacity - 0.35 lb. of O₂ per lb. of Li₂O₂).
- (10) Total quantity of carbon dioxide removed during 4 hours, or at test termination if earlier (lb).
- (11) Carbon dioxide utilization efficiency at 4 hours, or at test termination if earlier (Percent of theoretical capacity - 0.96 lb. of CO₂ per lb. of Li₂O₂).
- (12) Chemical type
- (13) Maximum bed temperature (°F)

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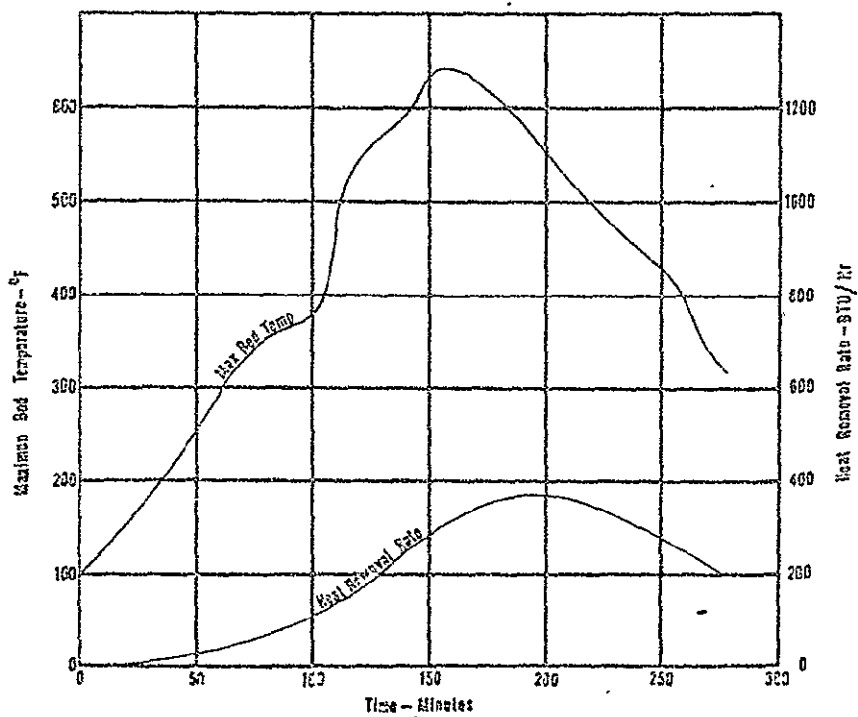


Figure 4-1

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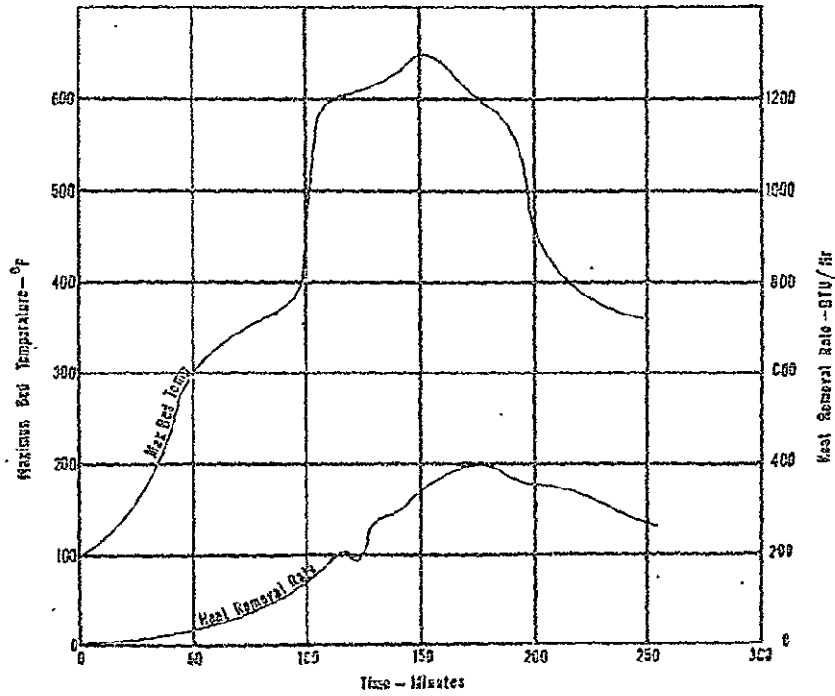
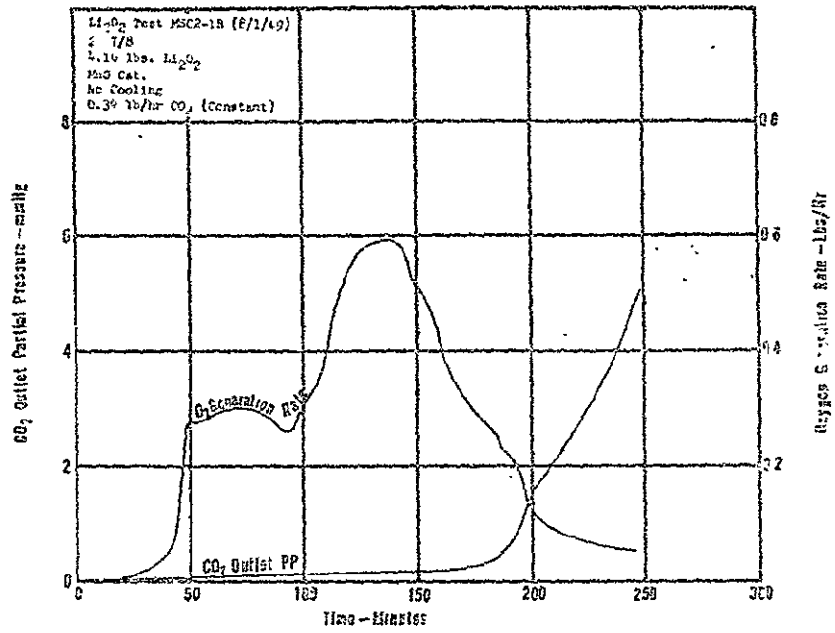


Figure 1-2

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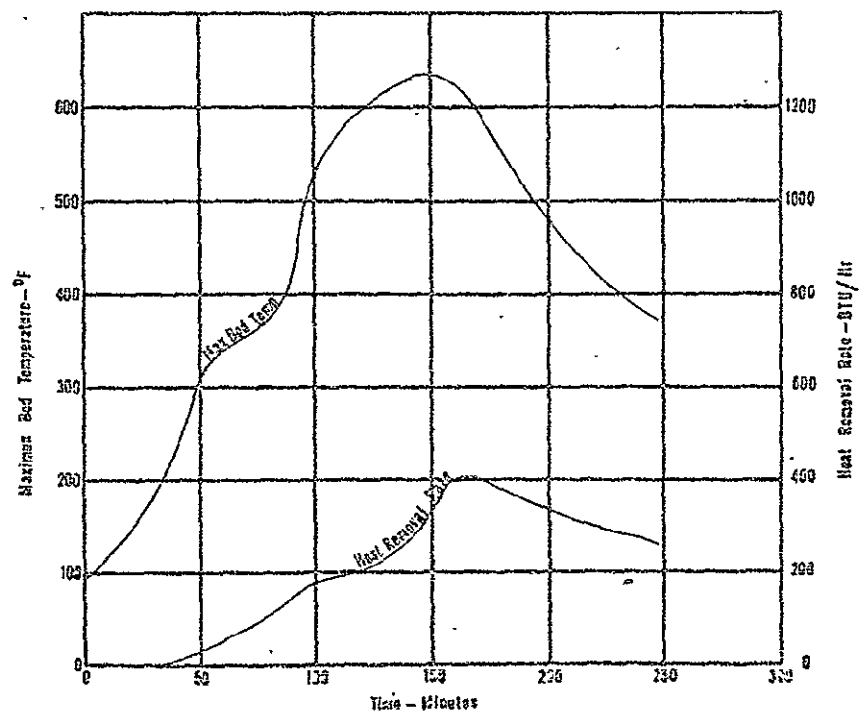
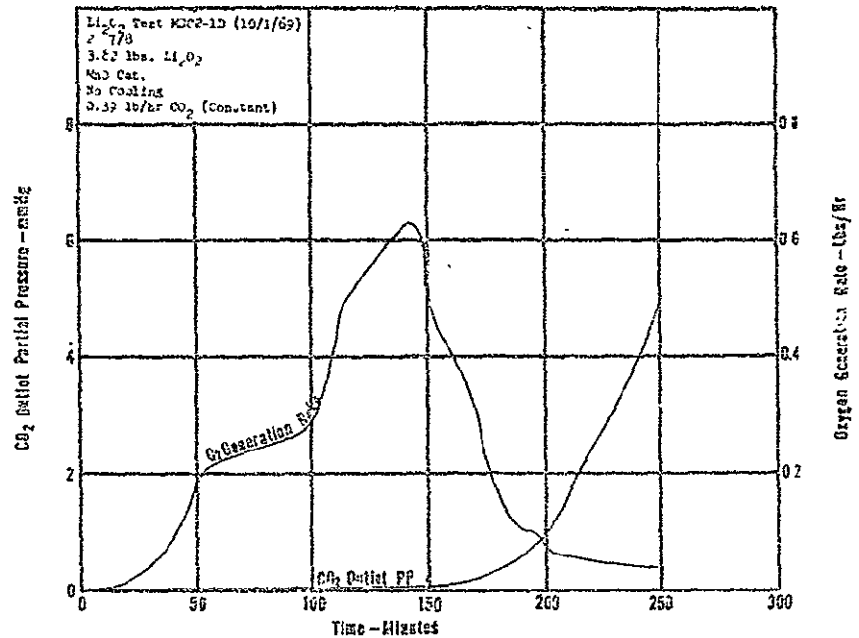


Figure 1-4

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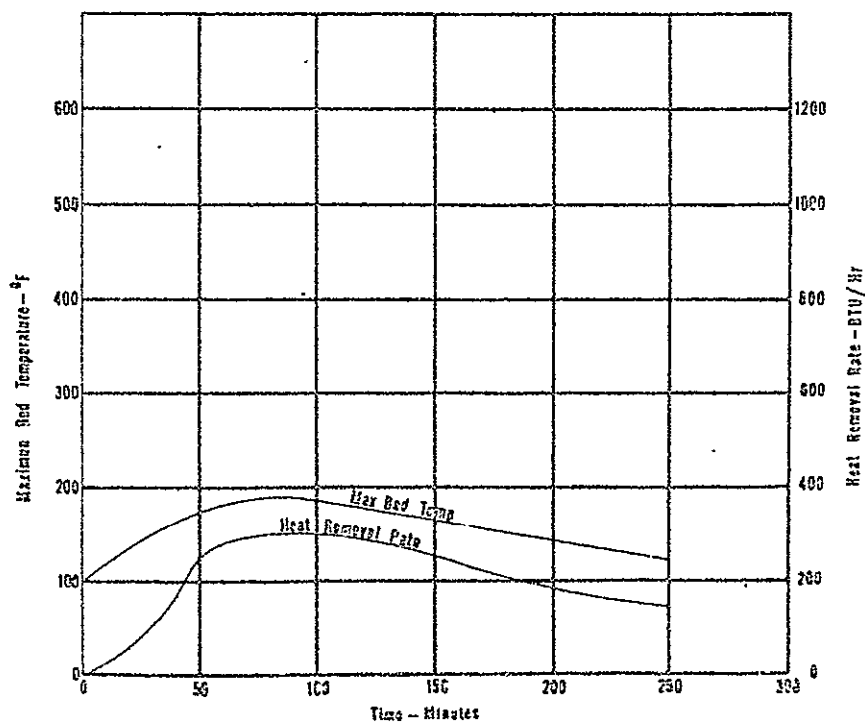
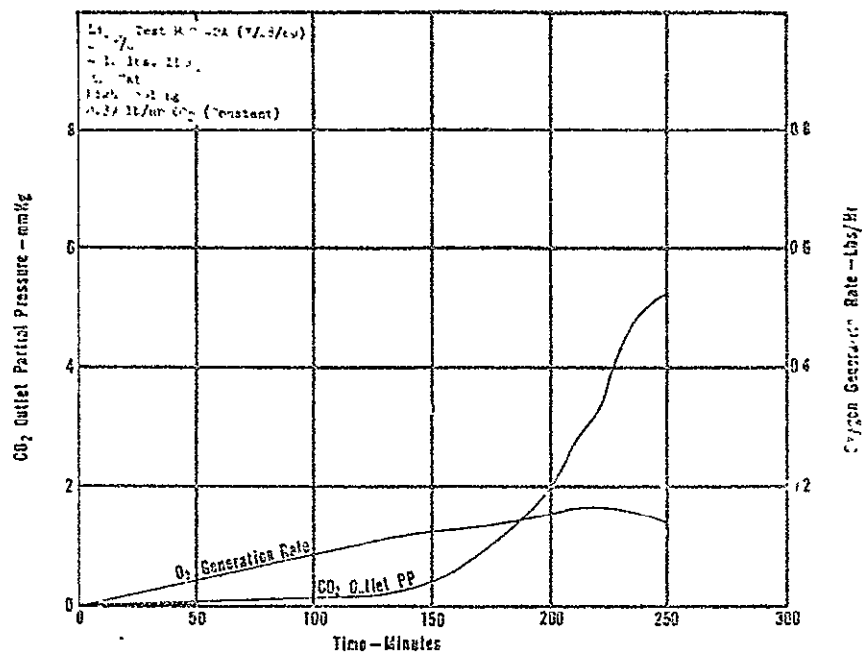


Figure 4-5

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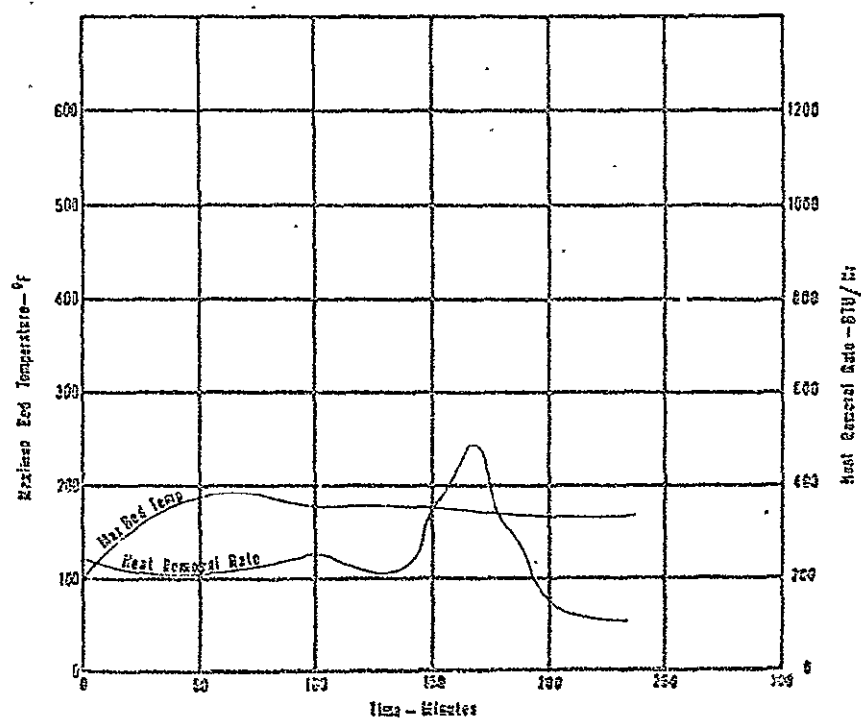
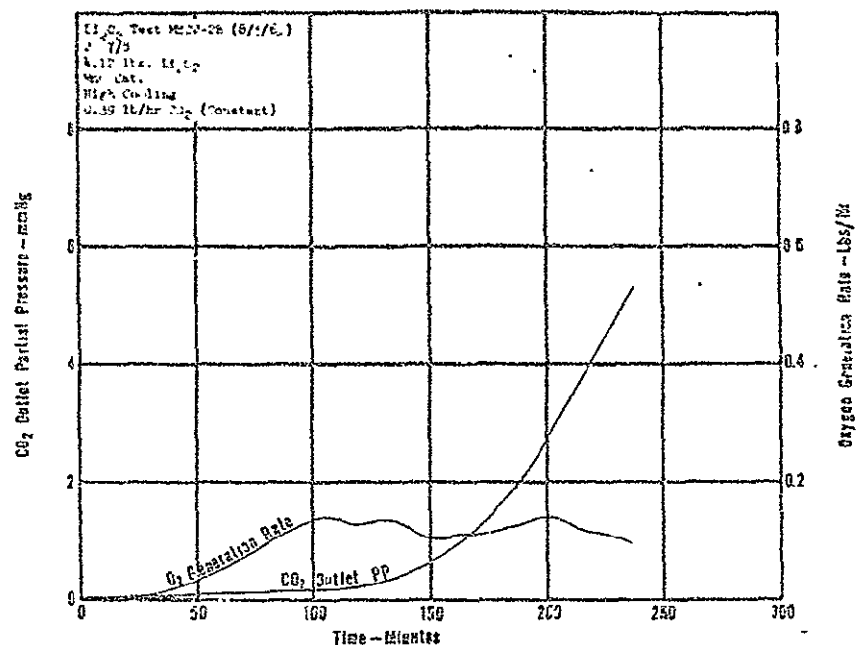


Figure L-6

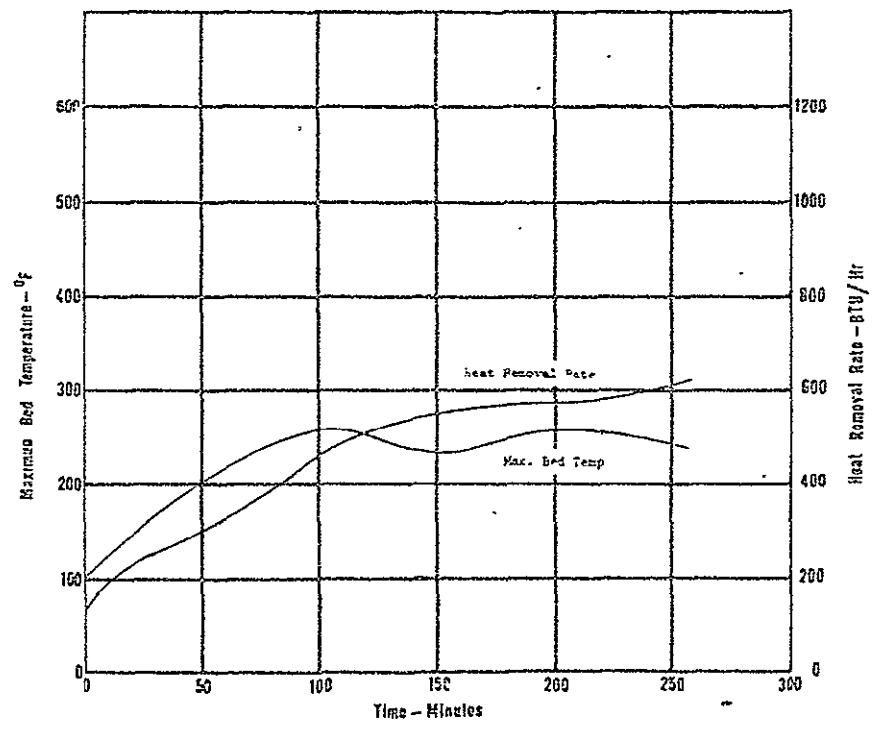
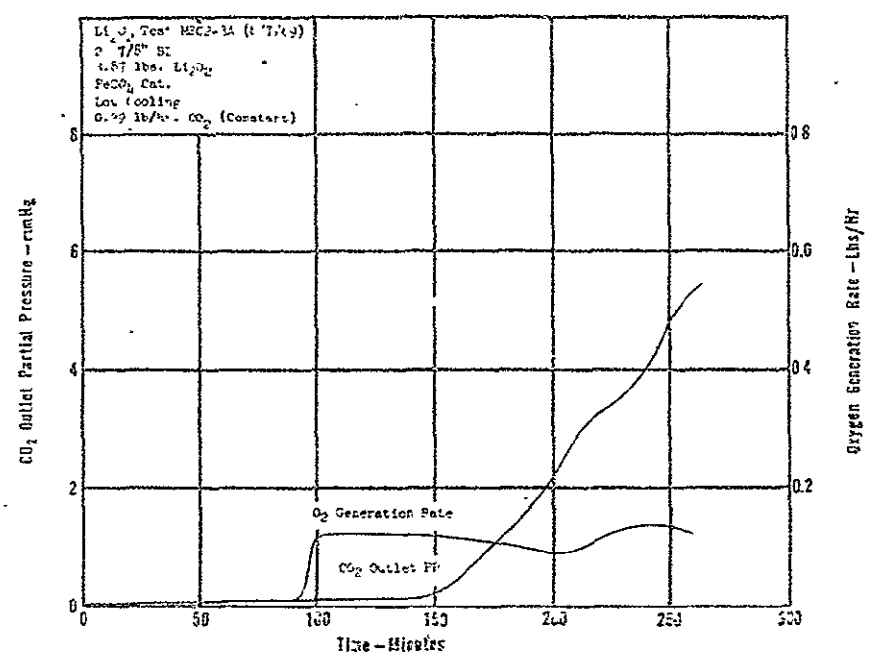


Figure 4-7

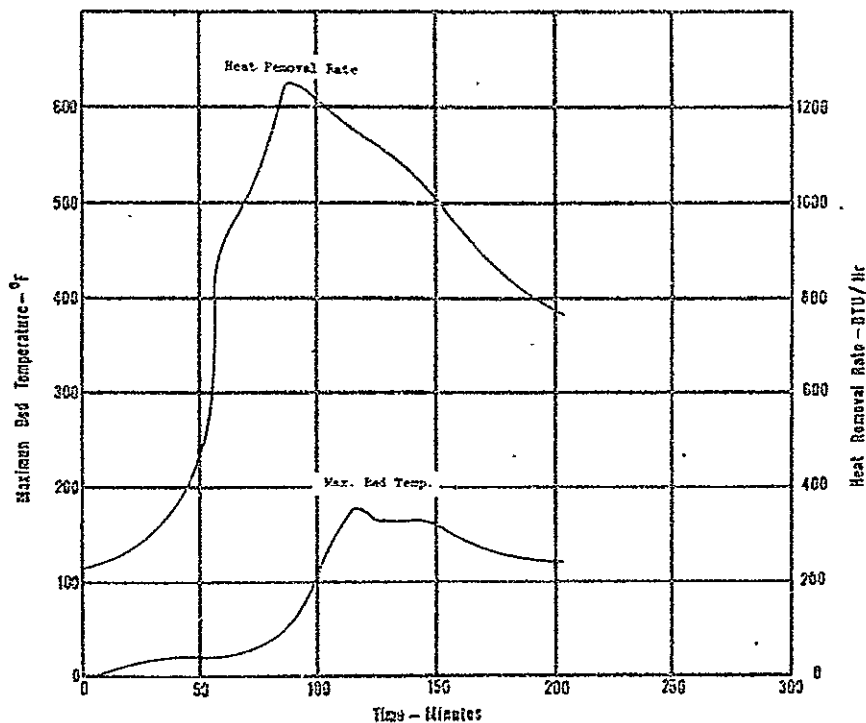
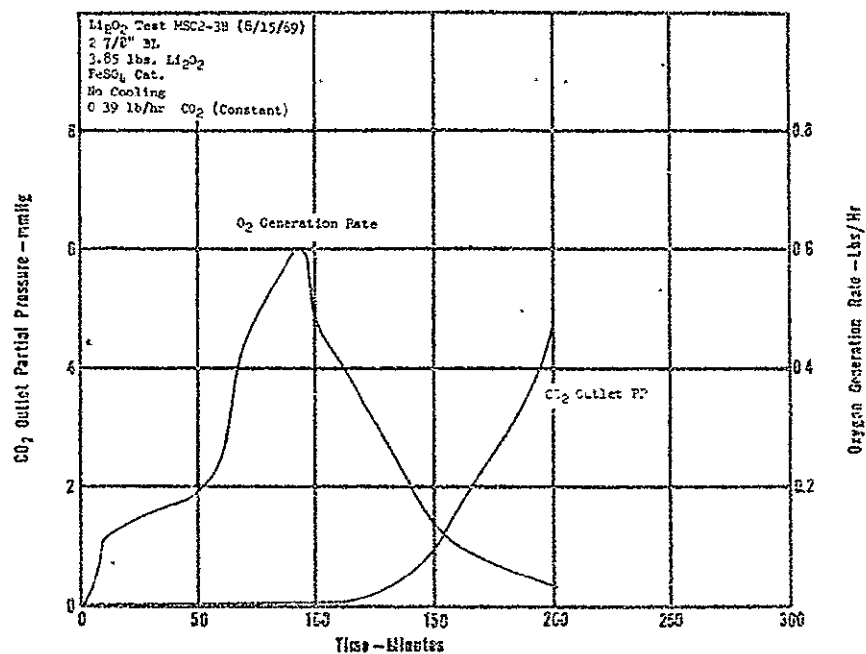


Figure 1-6

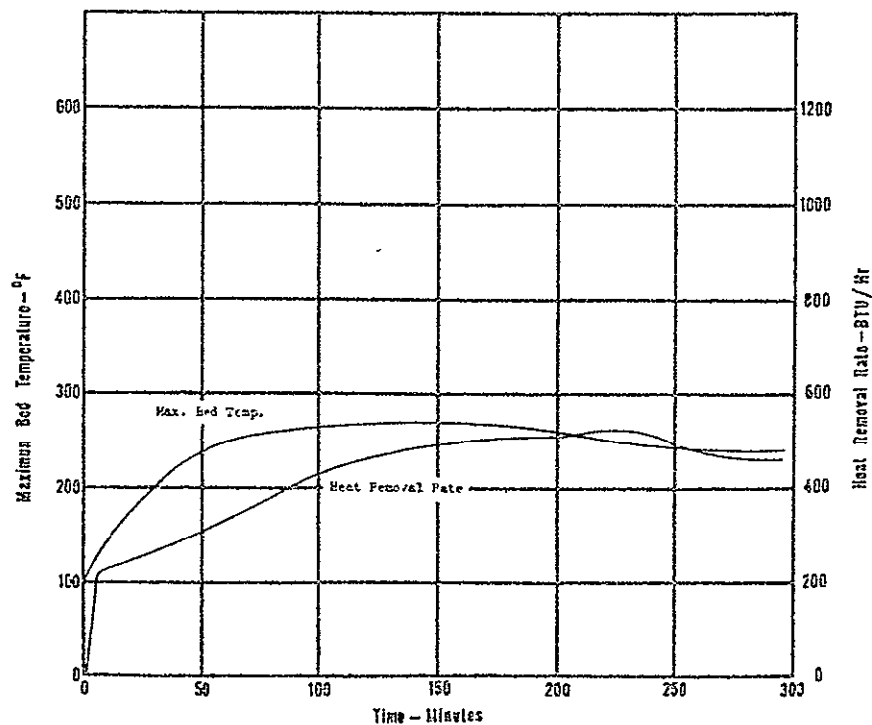
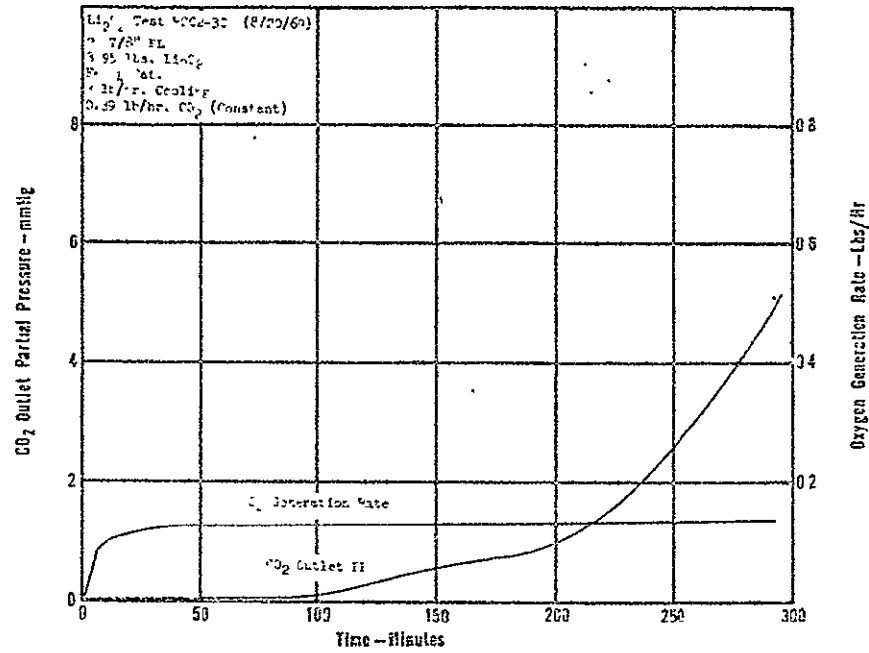


Figure 4-9

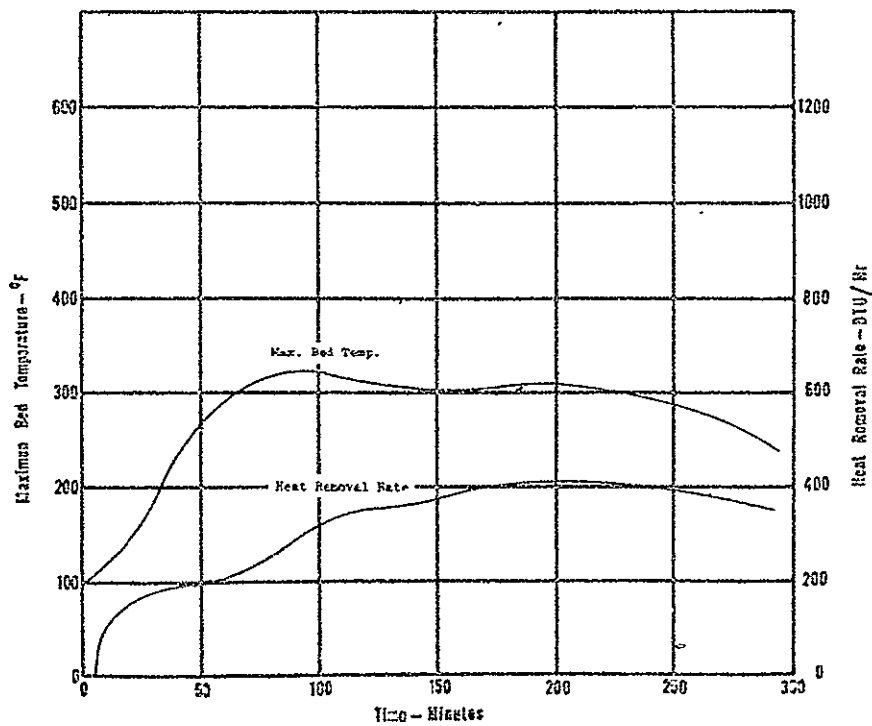
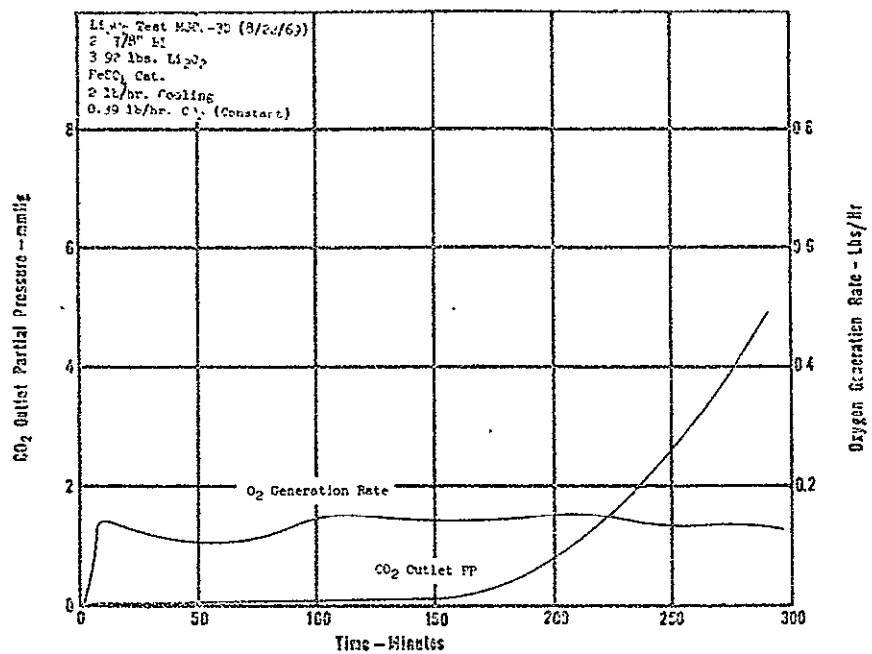


Figure A-10

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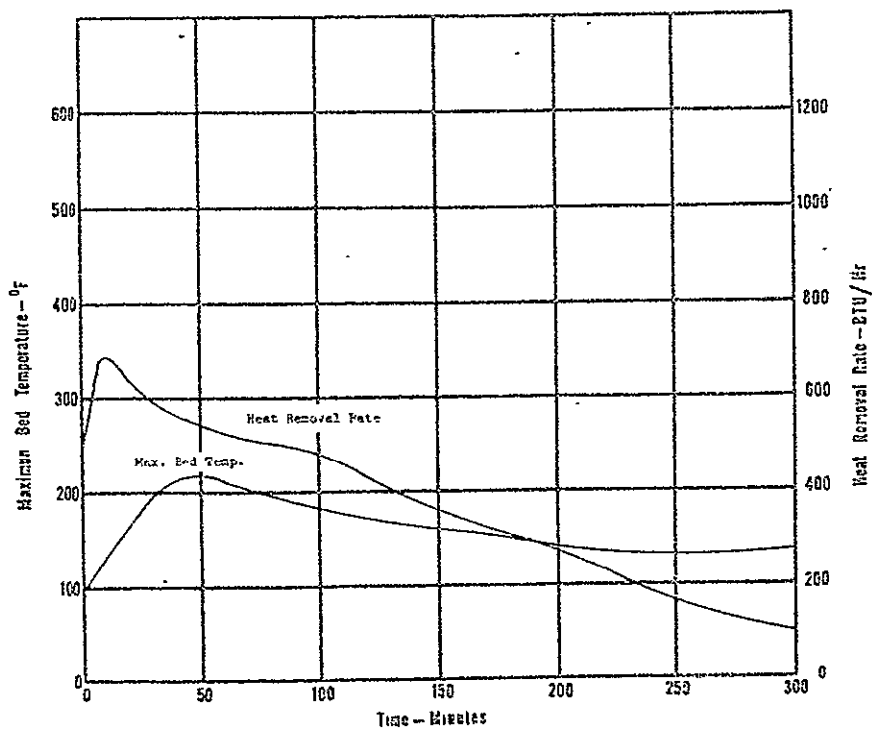
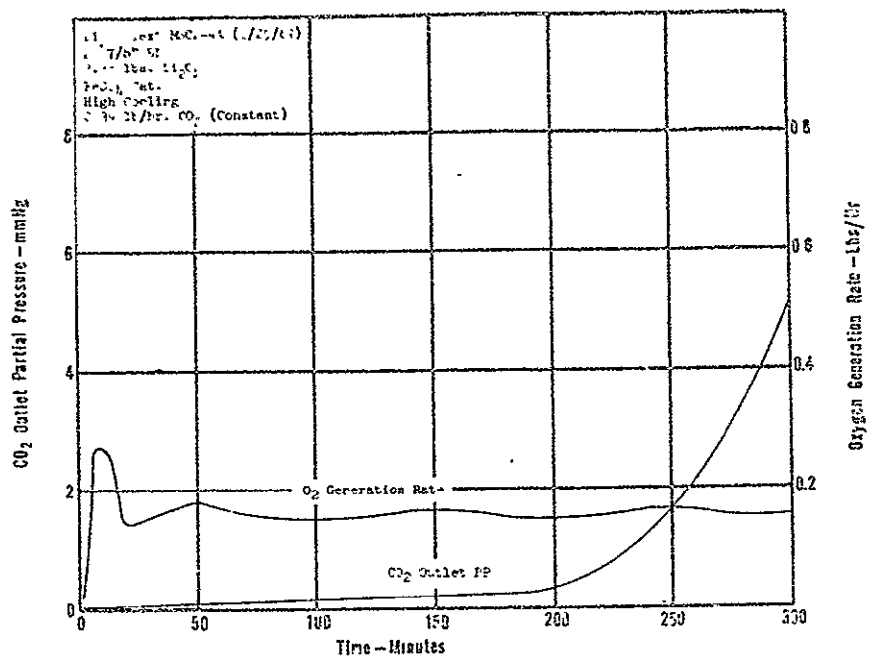


Figure 4-11

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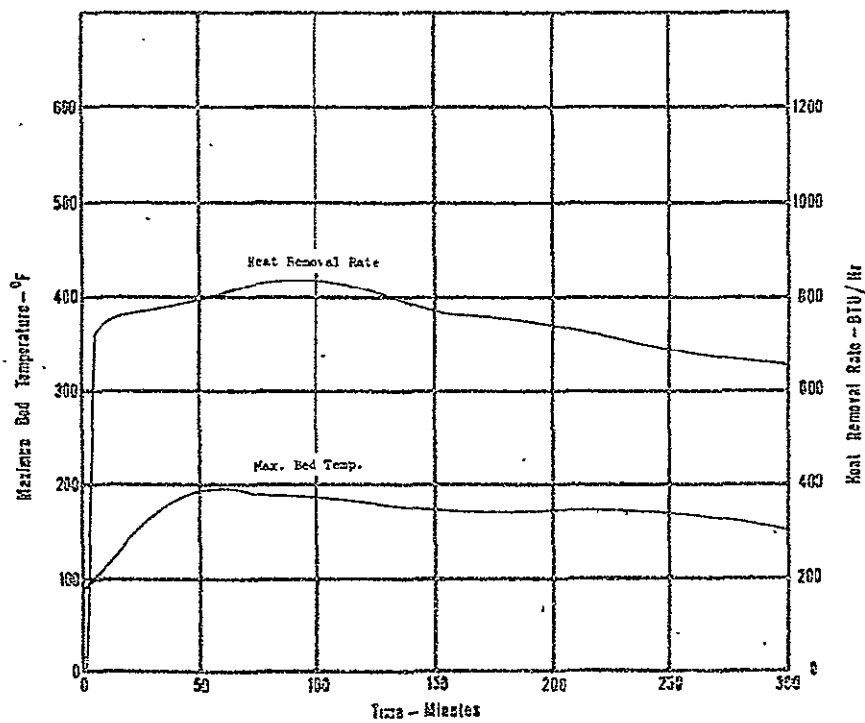
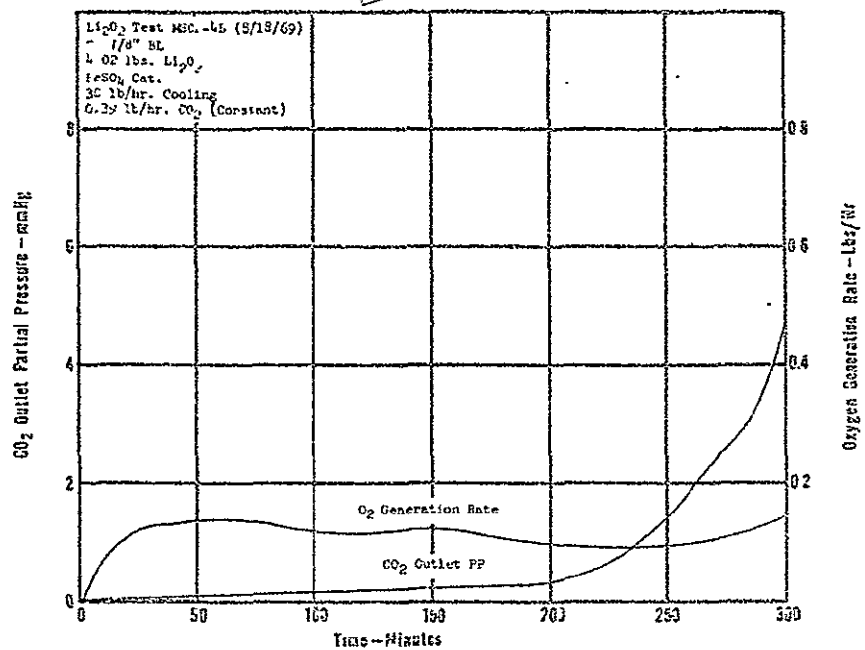


Figure 4-12

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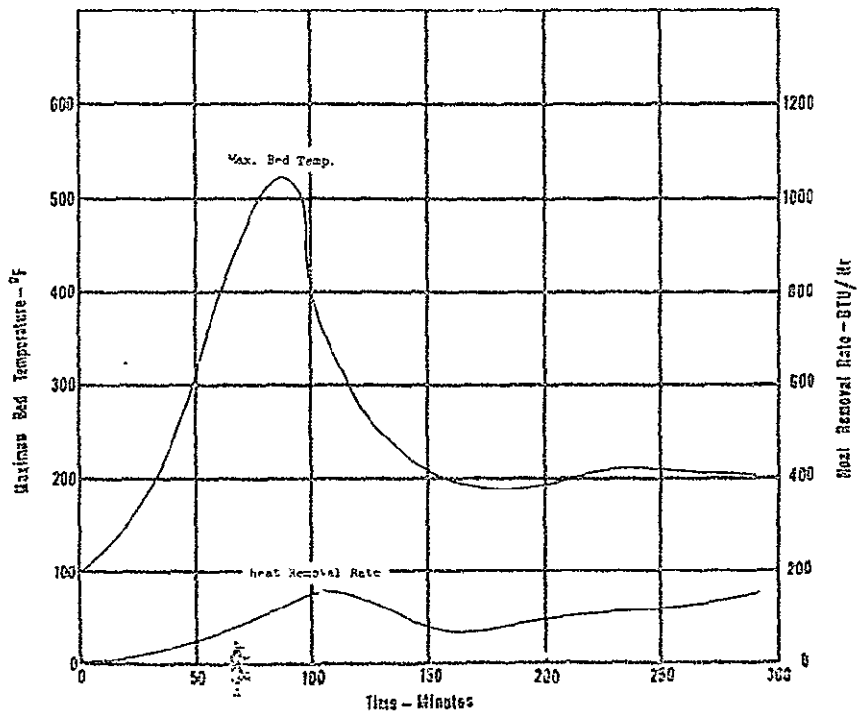
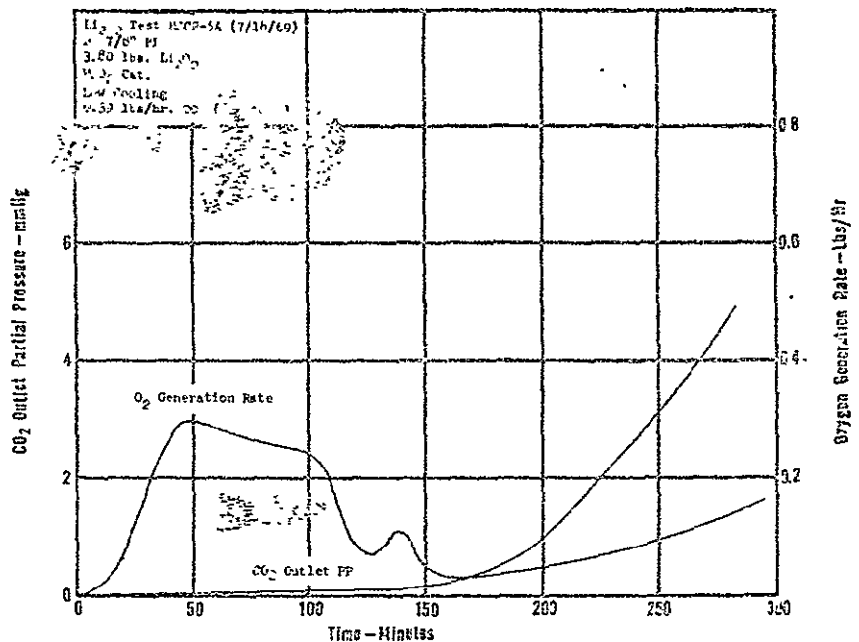


Figure 4-13

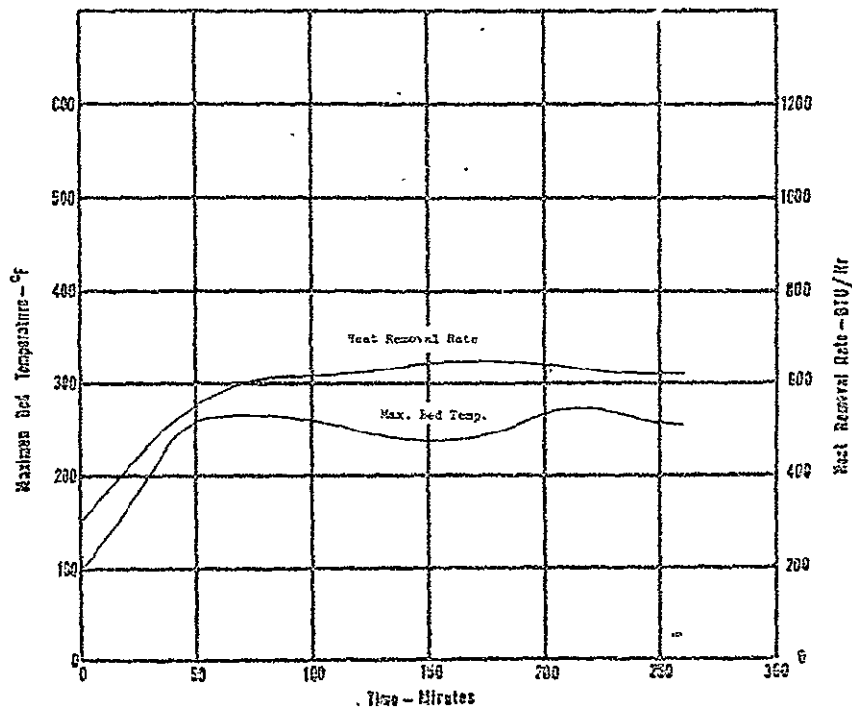
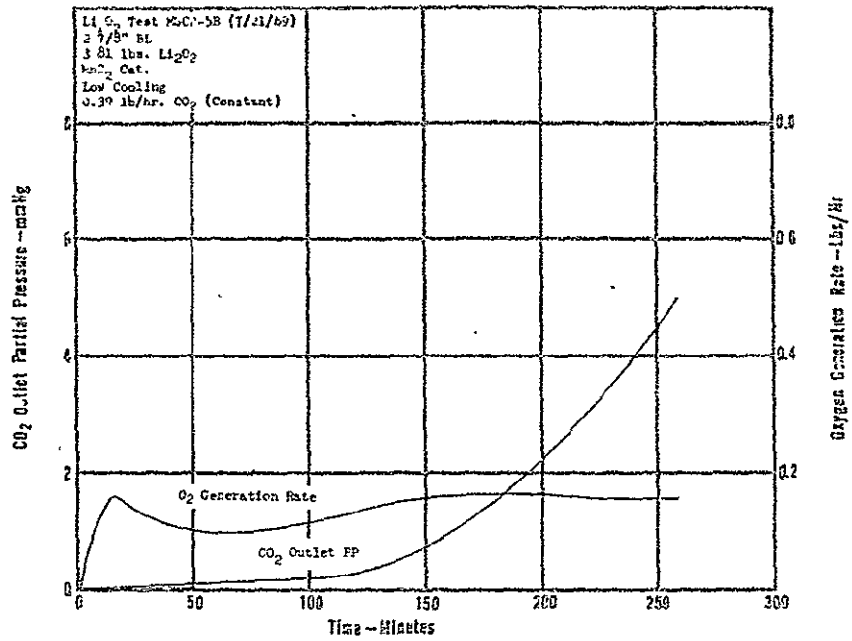


Figure 4-11

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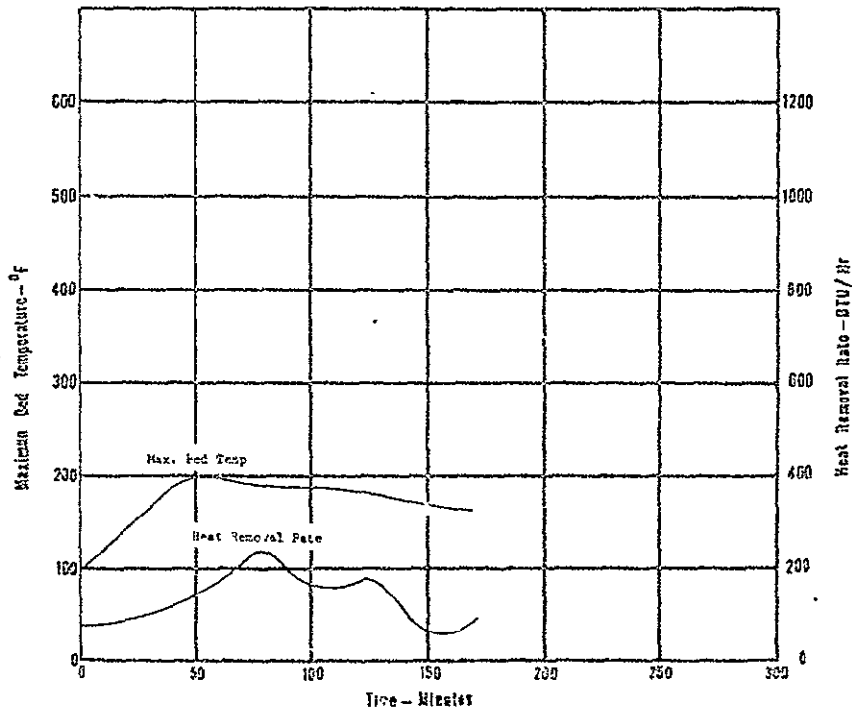
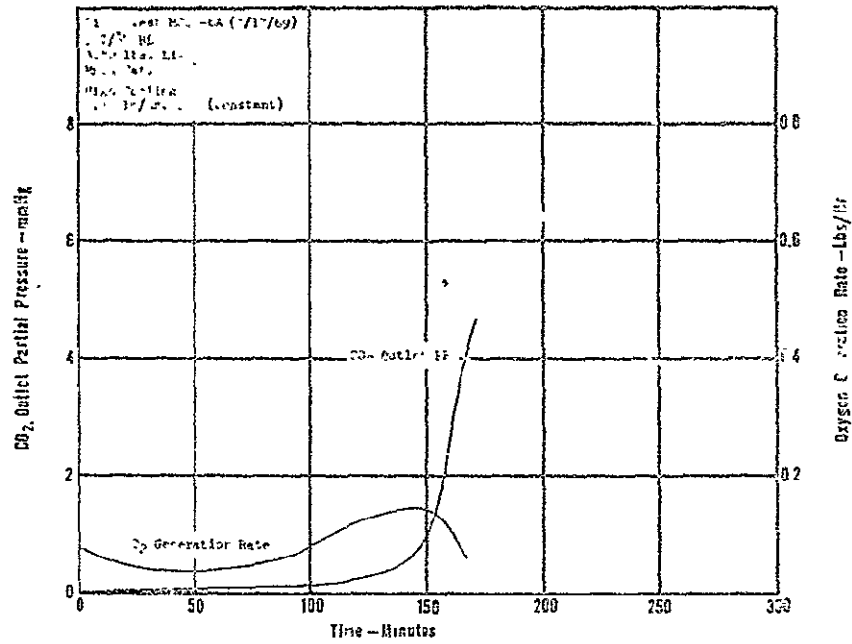


Figure 4-15

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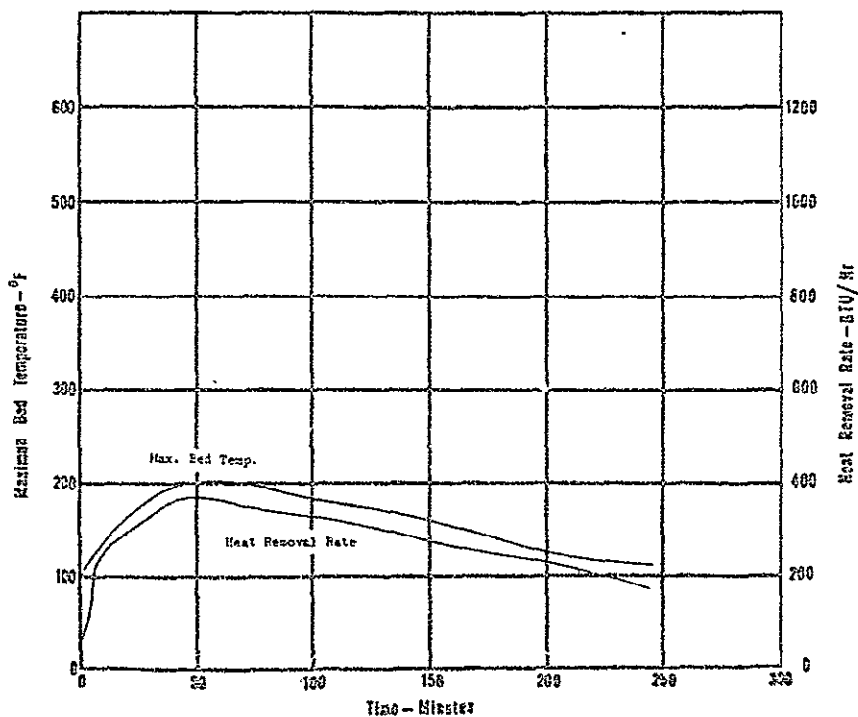
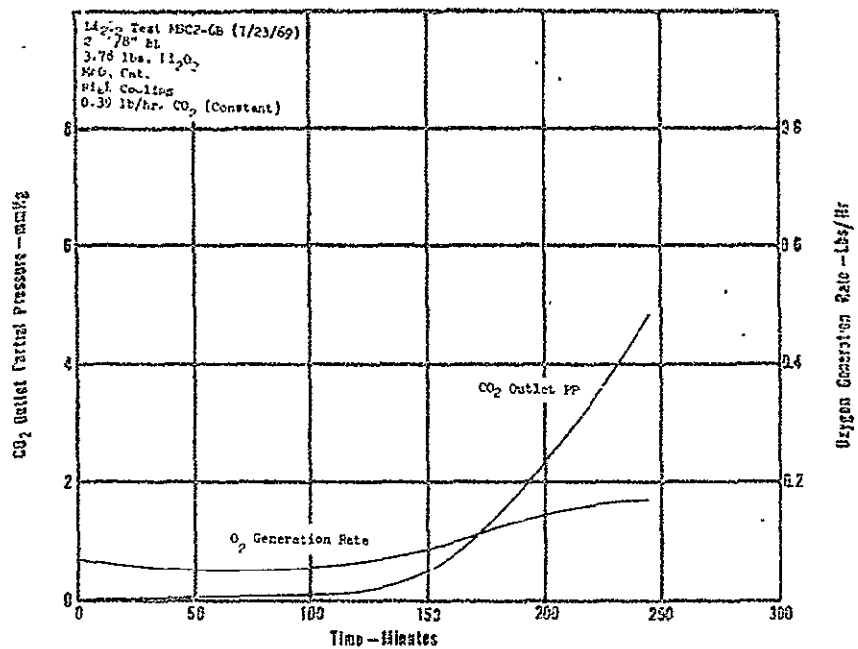


Figure 4-16

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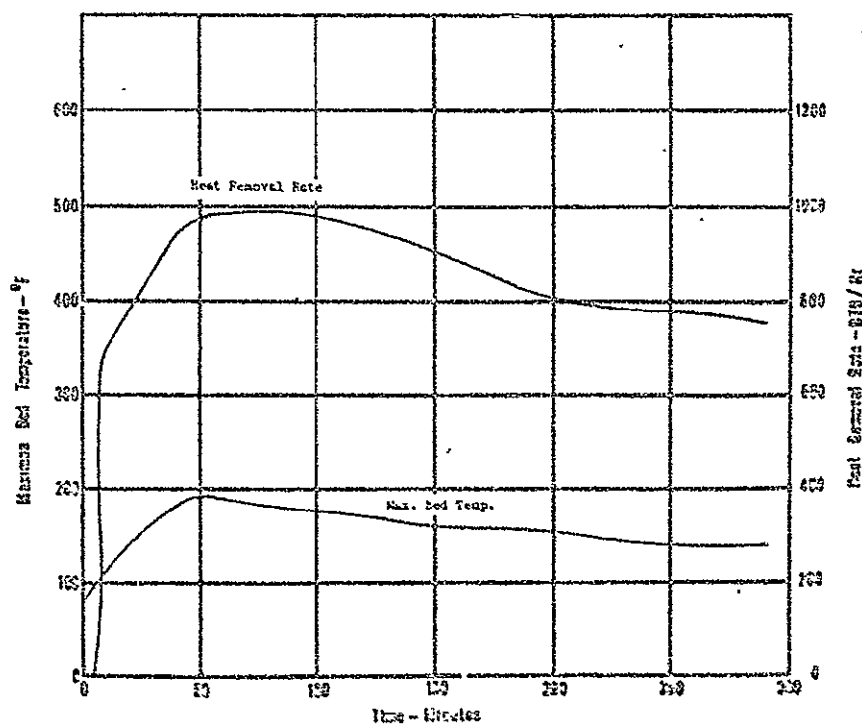
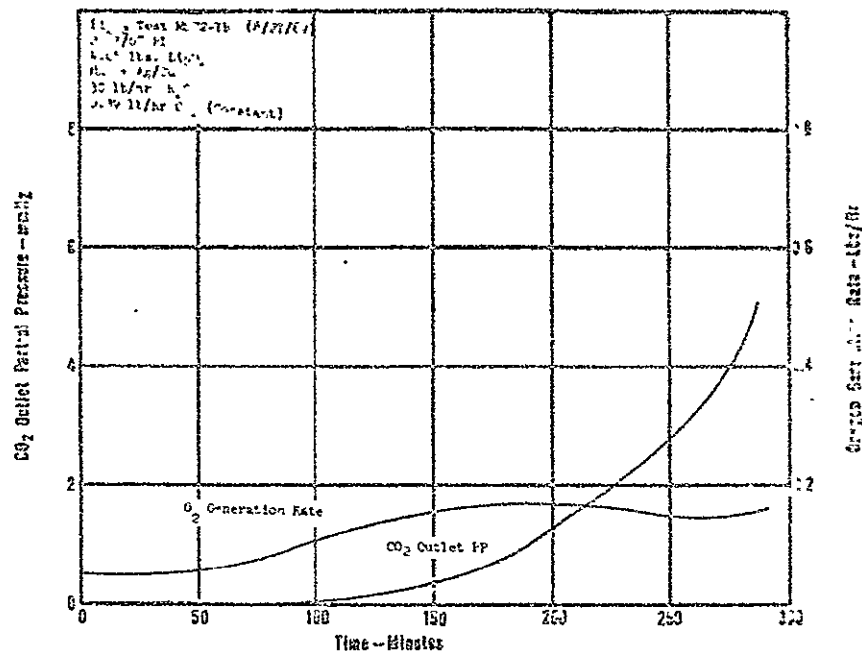


Figure 4-16

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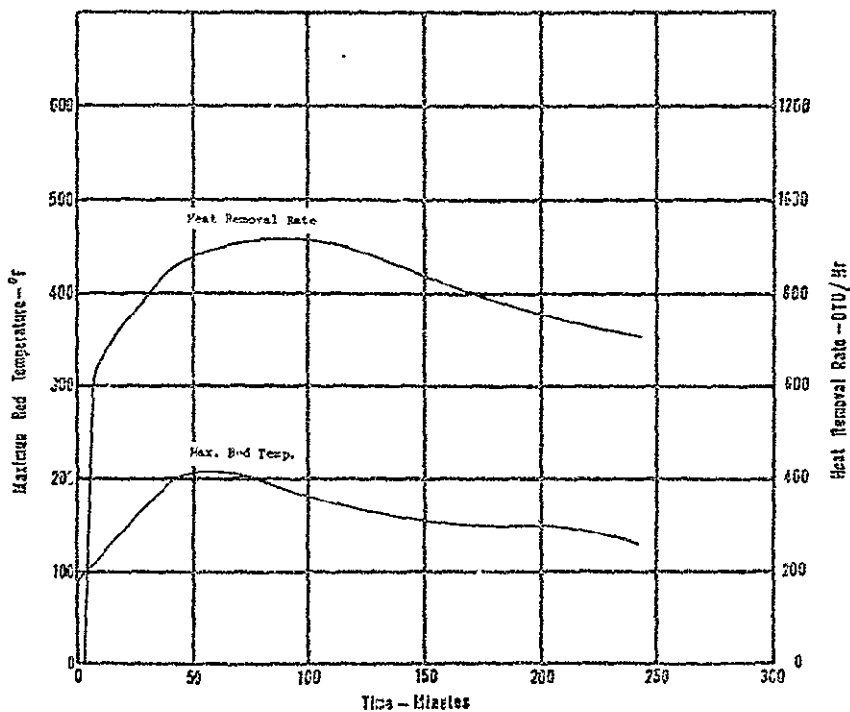
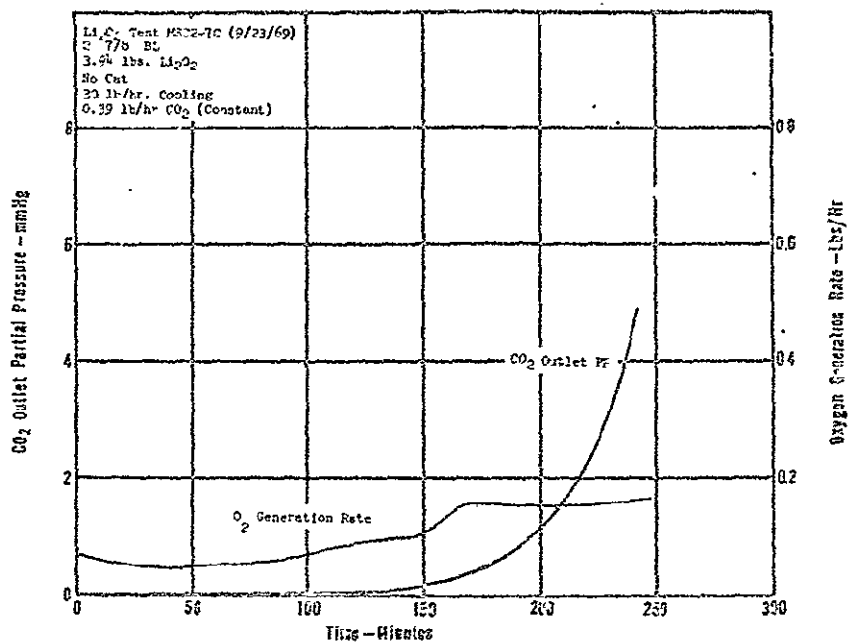


Figure 4-19

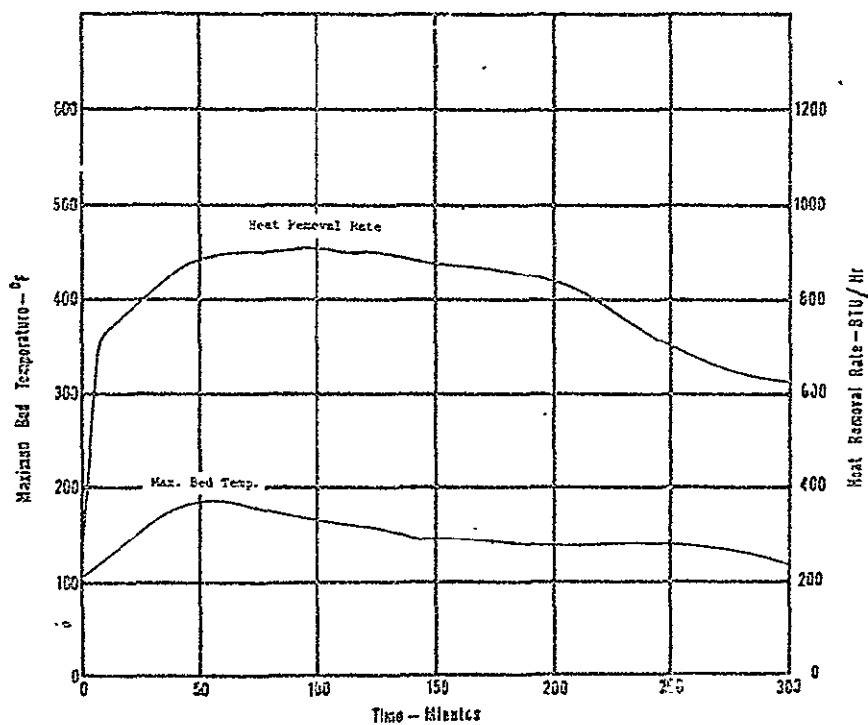
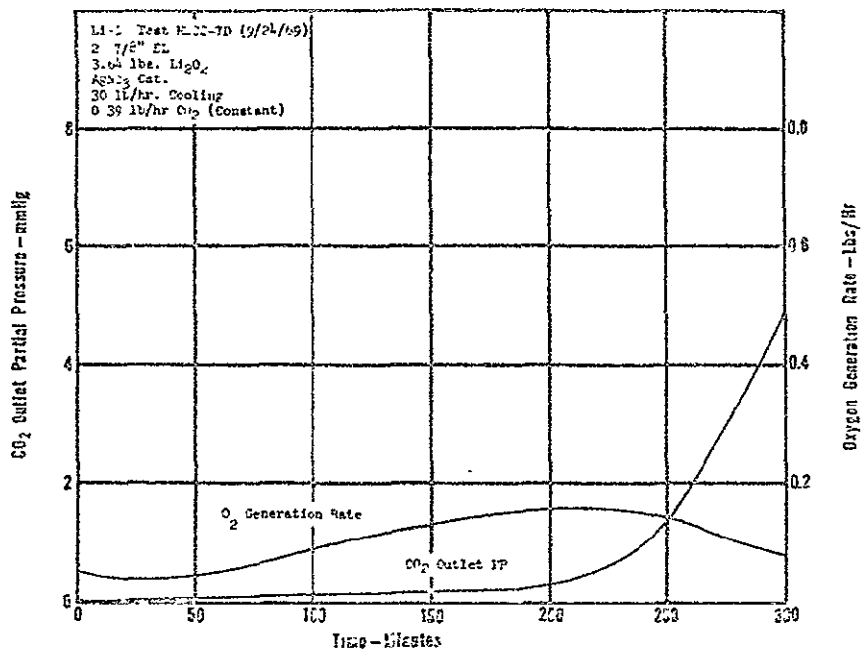


Figure 4-20

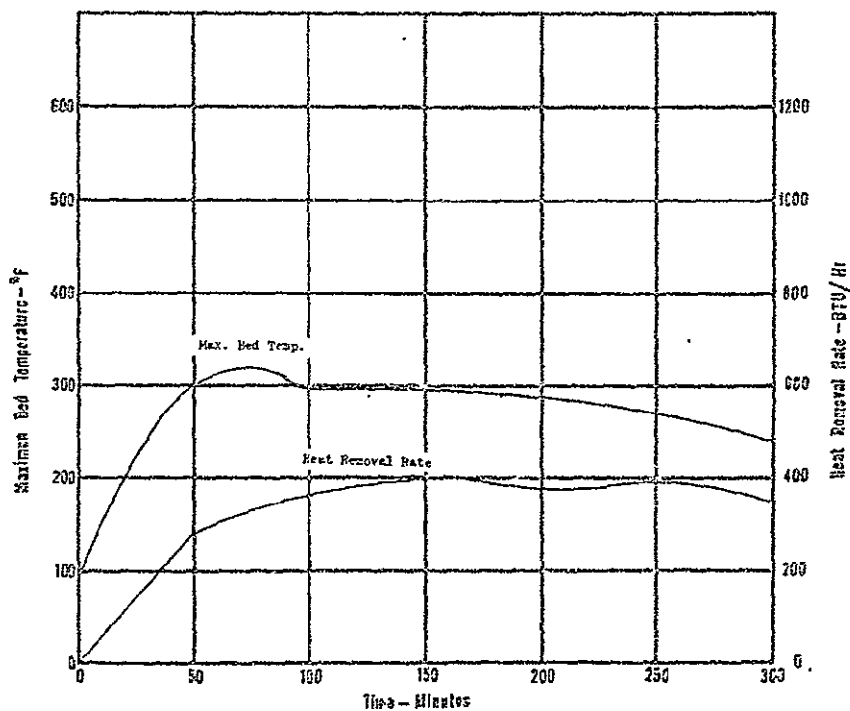
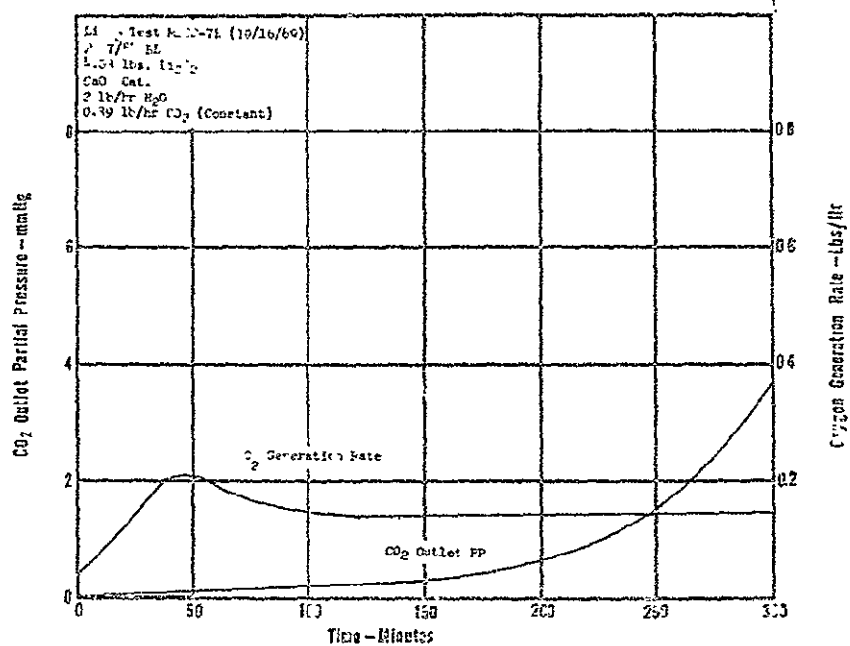


Figure 4-21

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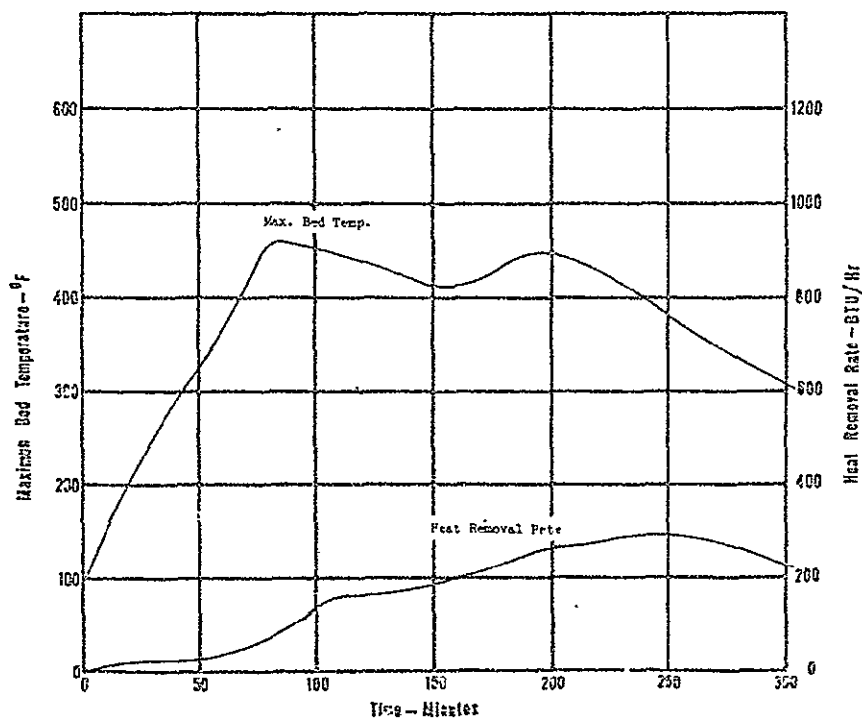
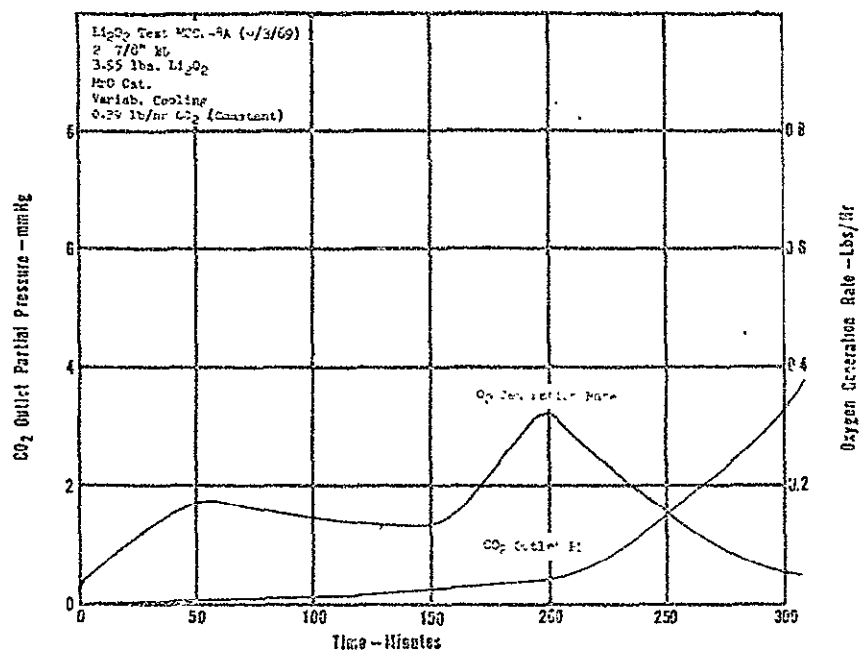


Figure 4-22

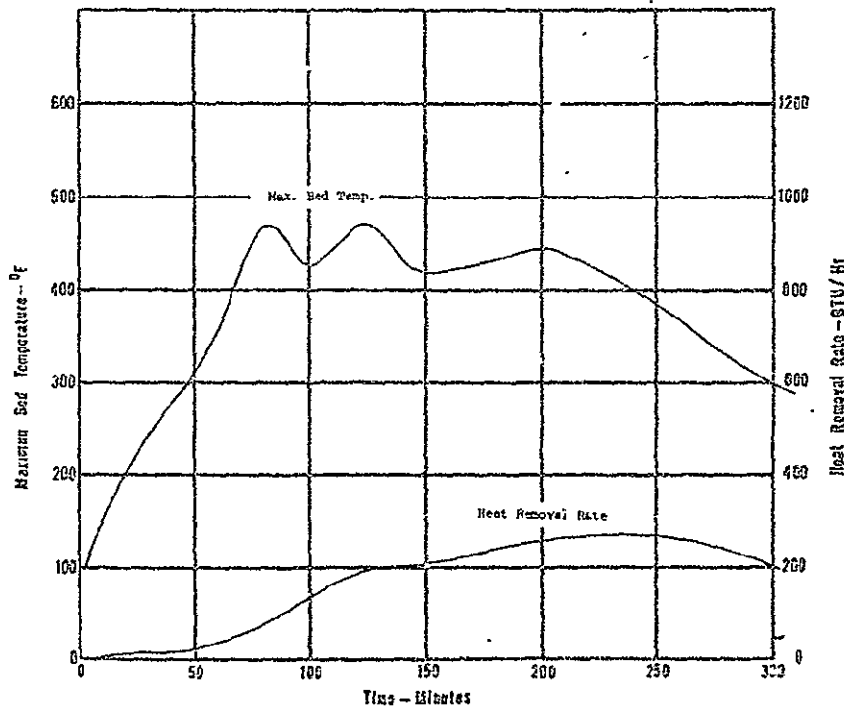
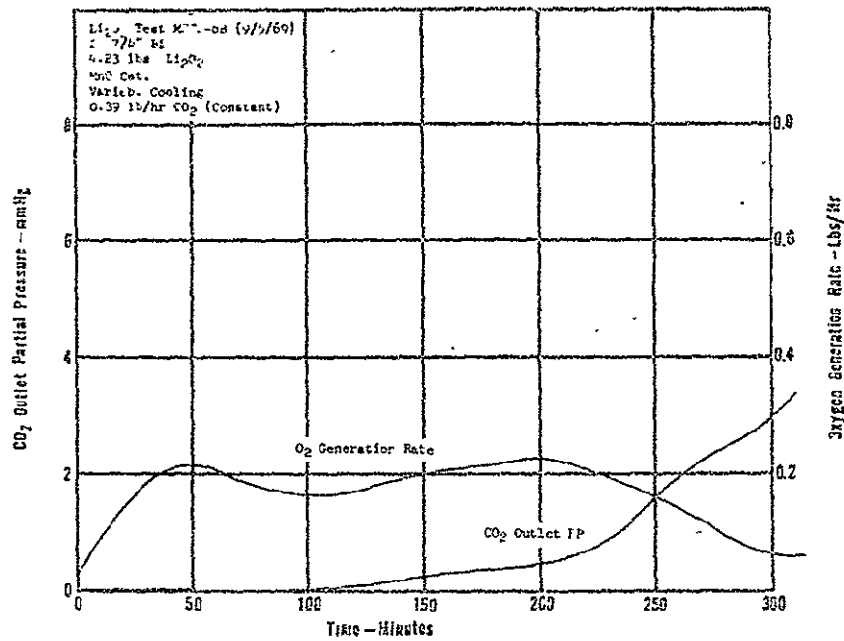


Figure 4-23

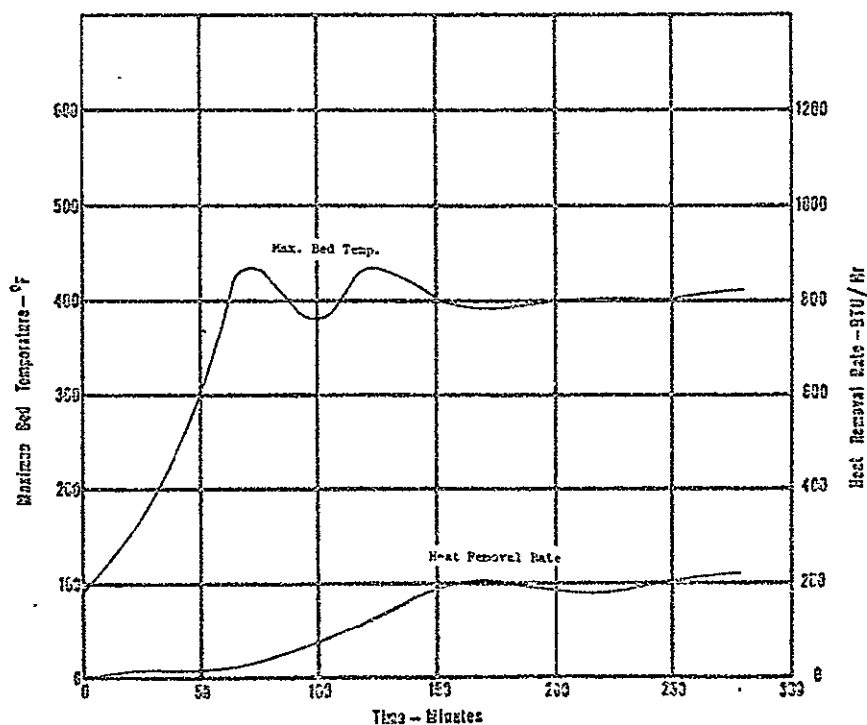
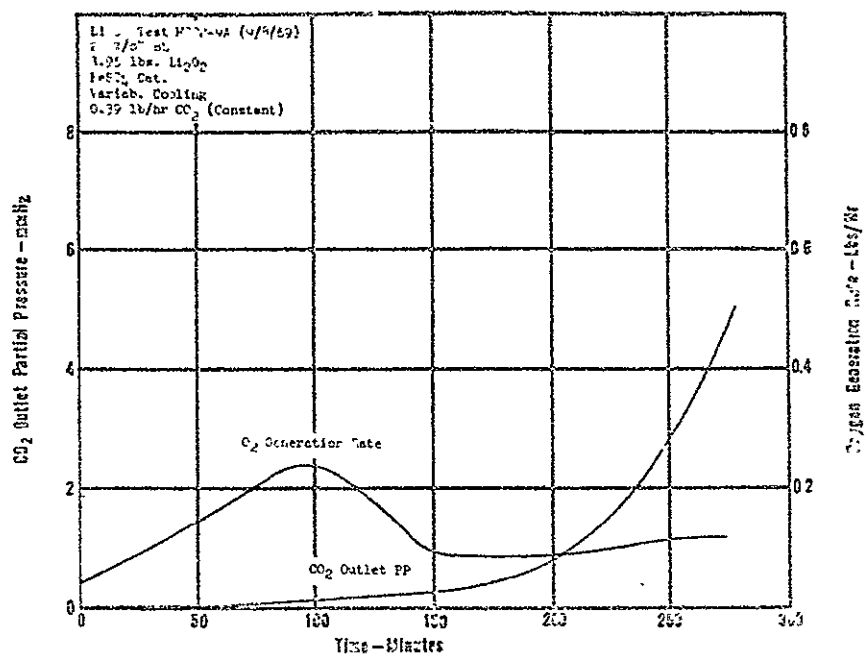


Figure 4-24

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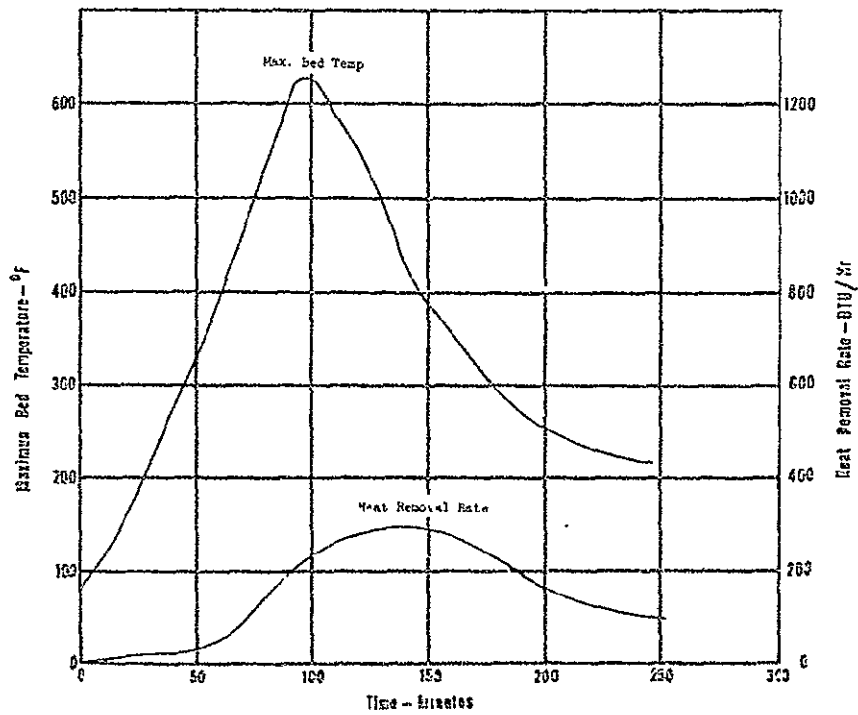
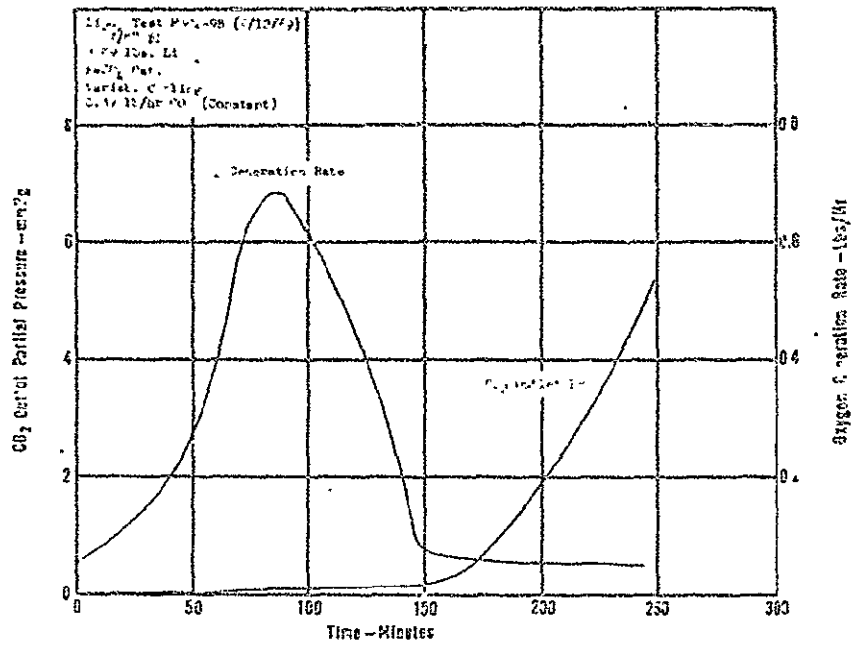


Figure b-25

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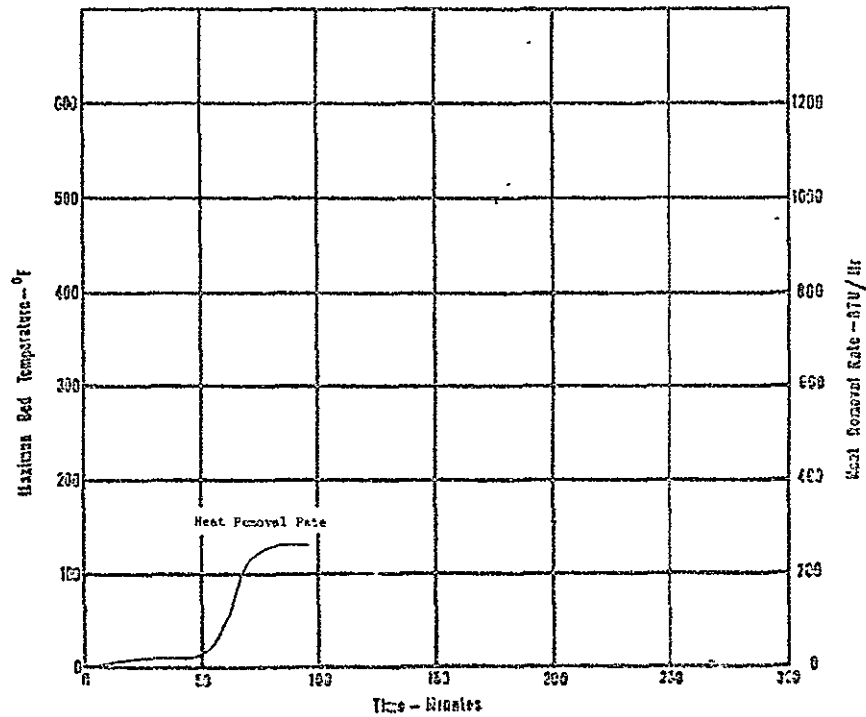
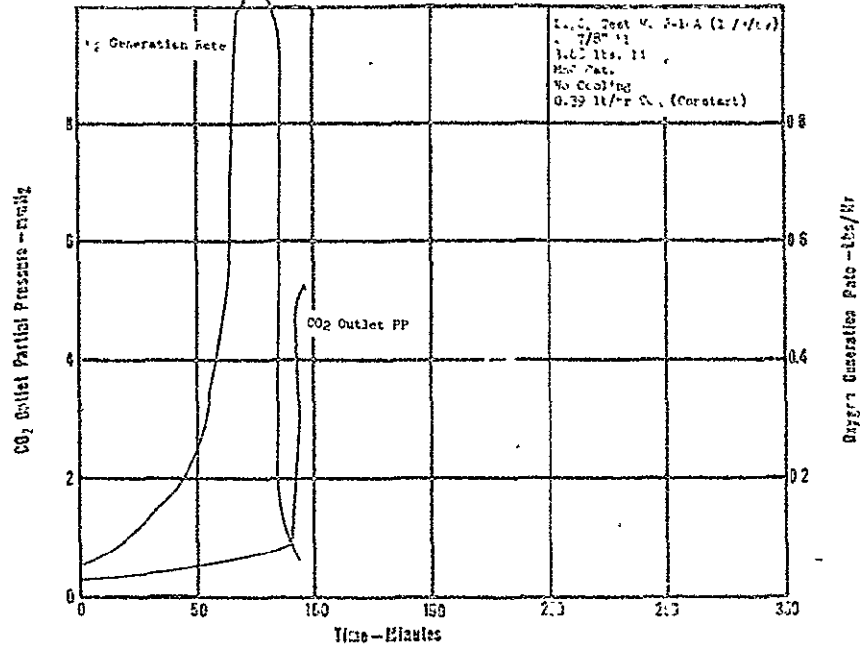


Figure 4-26

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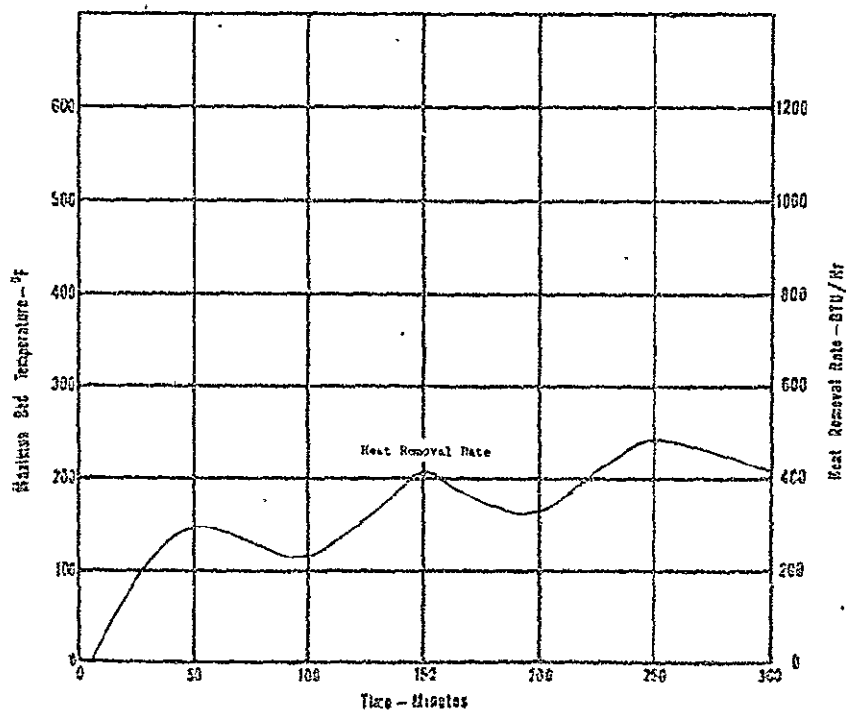
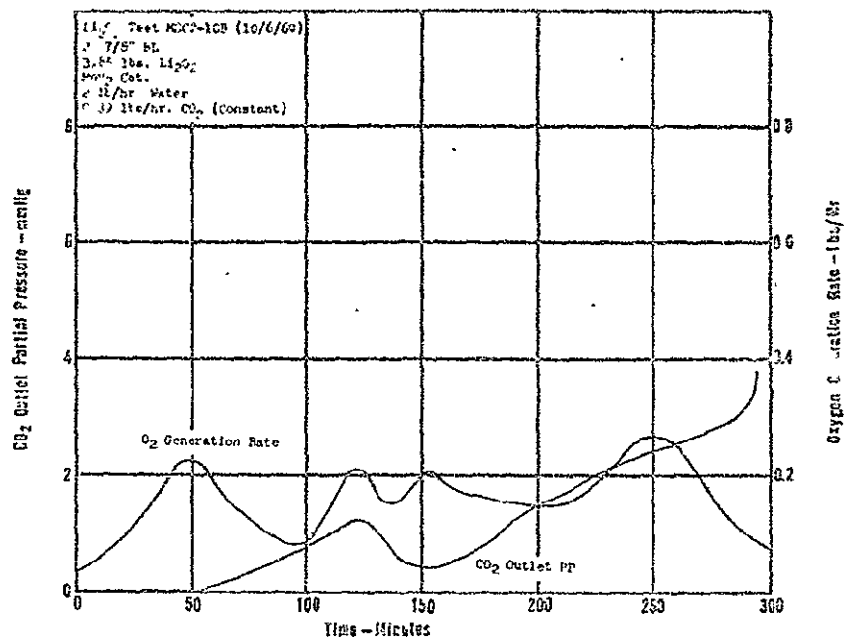


Figure 4-27

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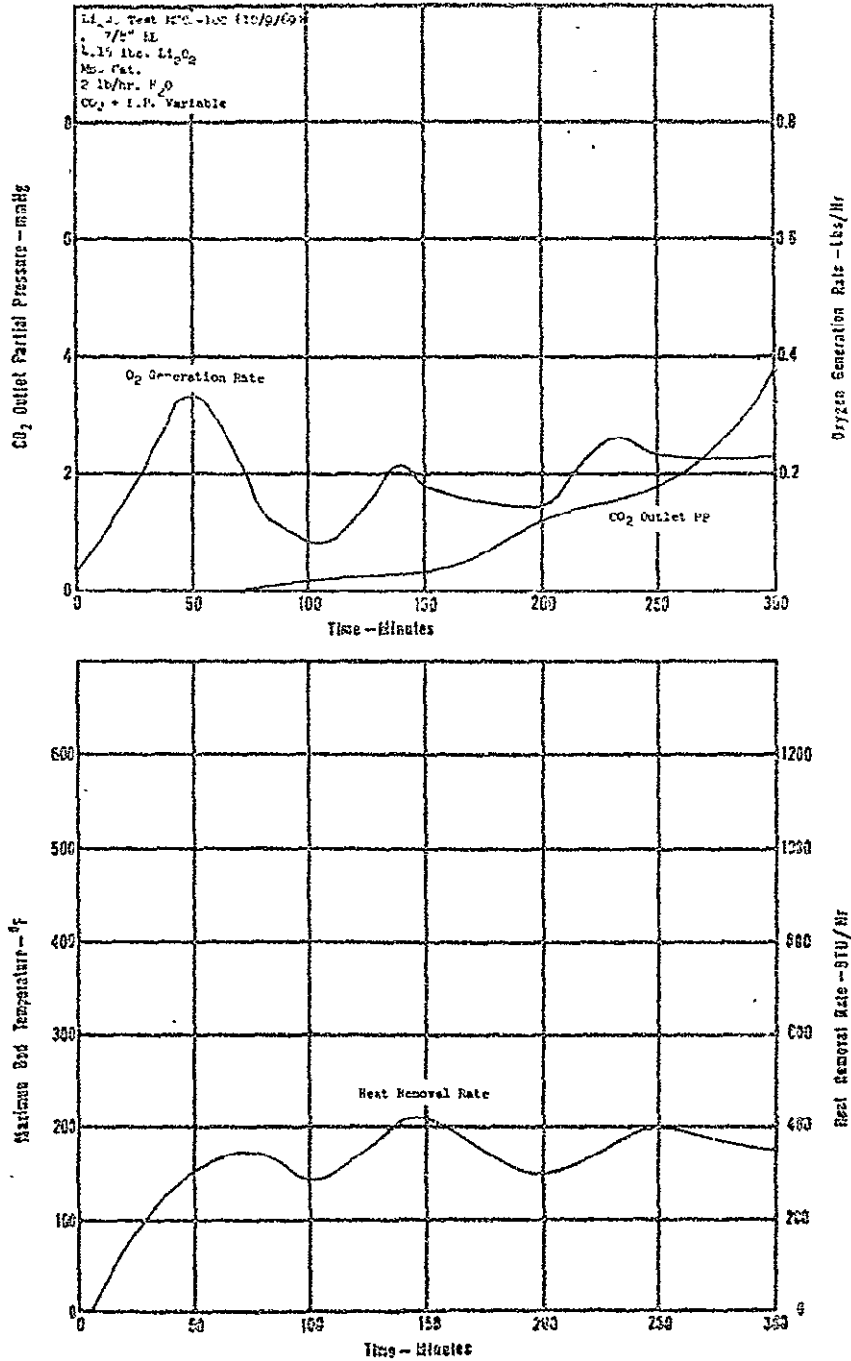


Figure 4-10

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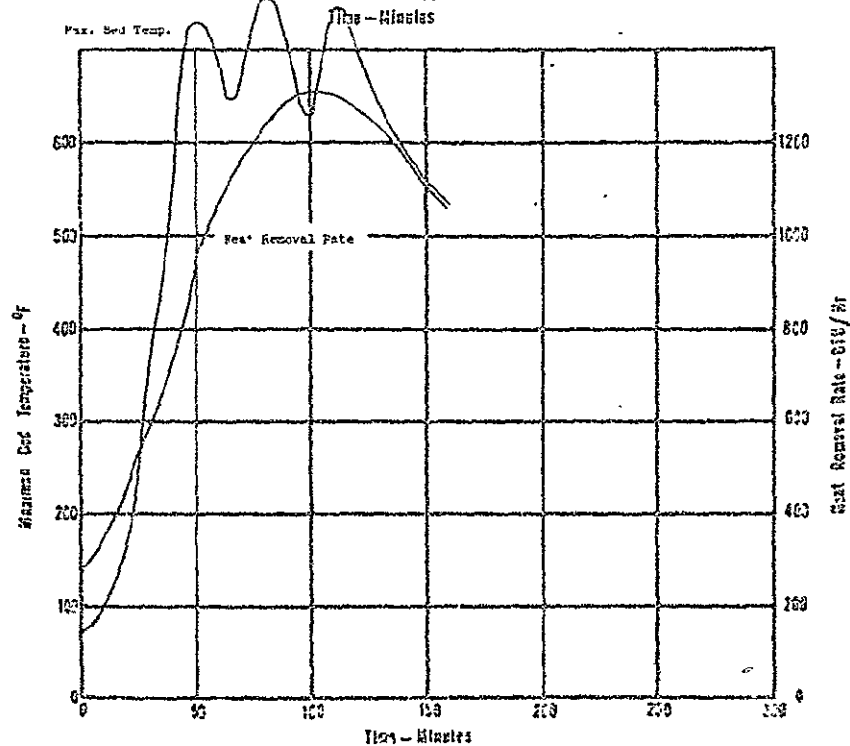
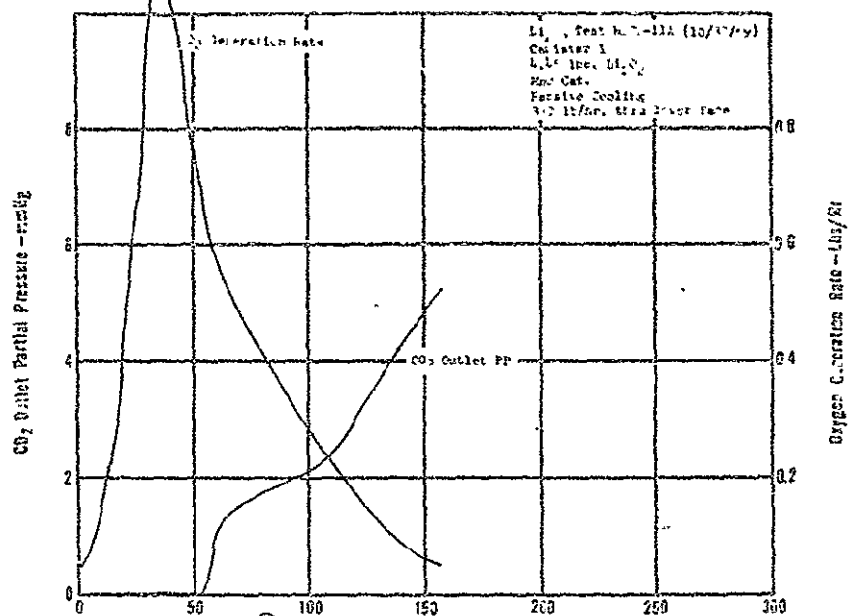


Figure 1-29

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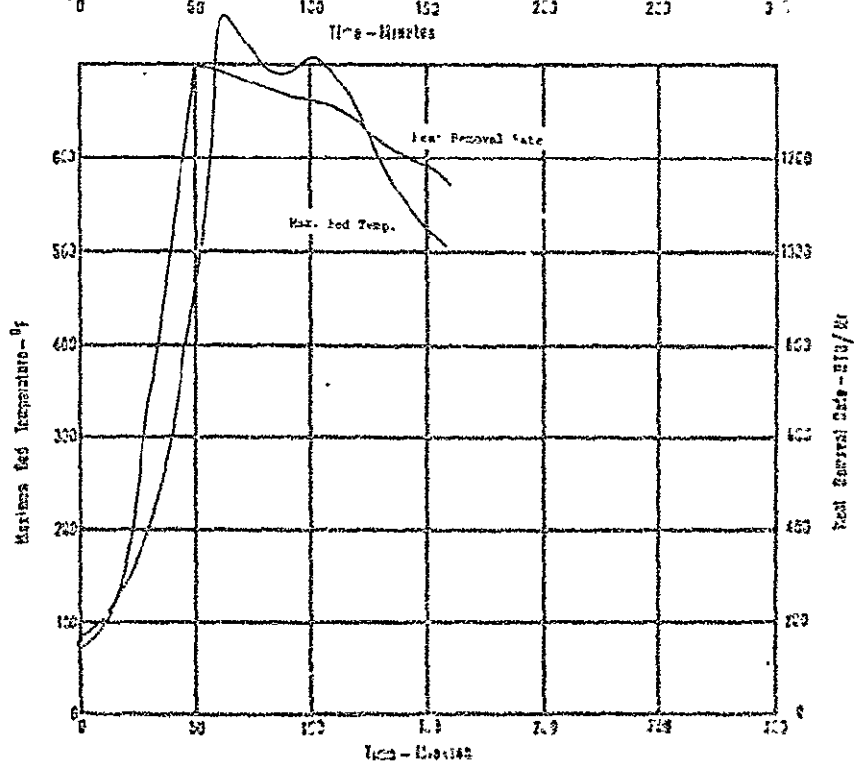
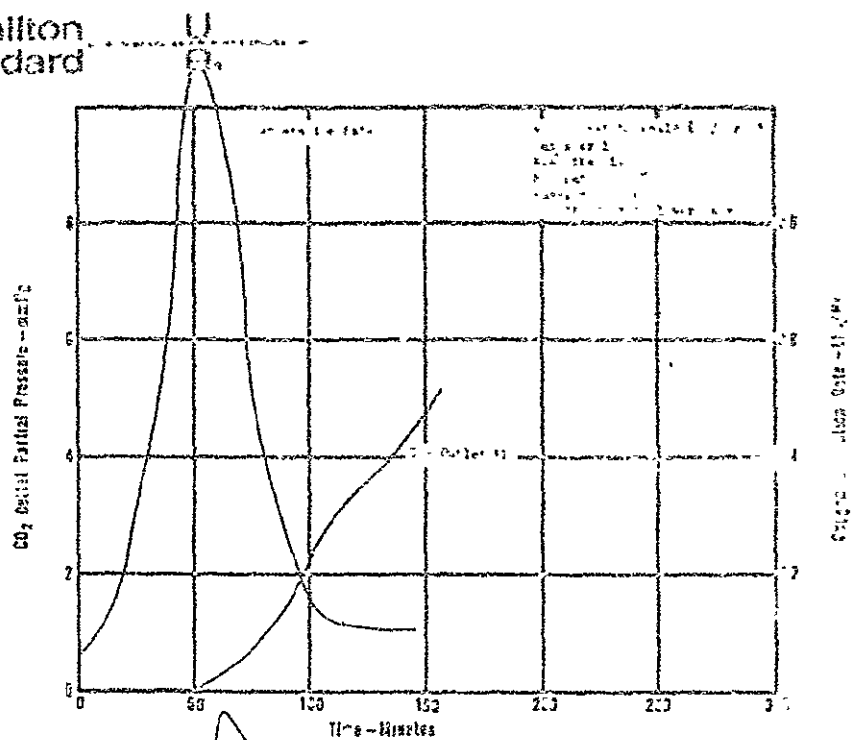


Figure 4-15

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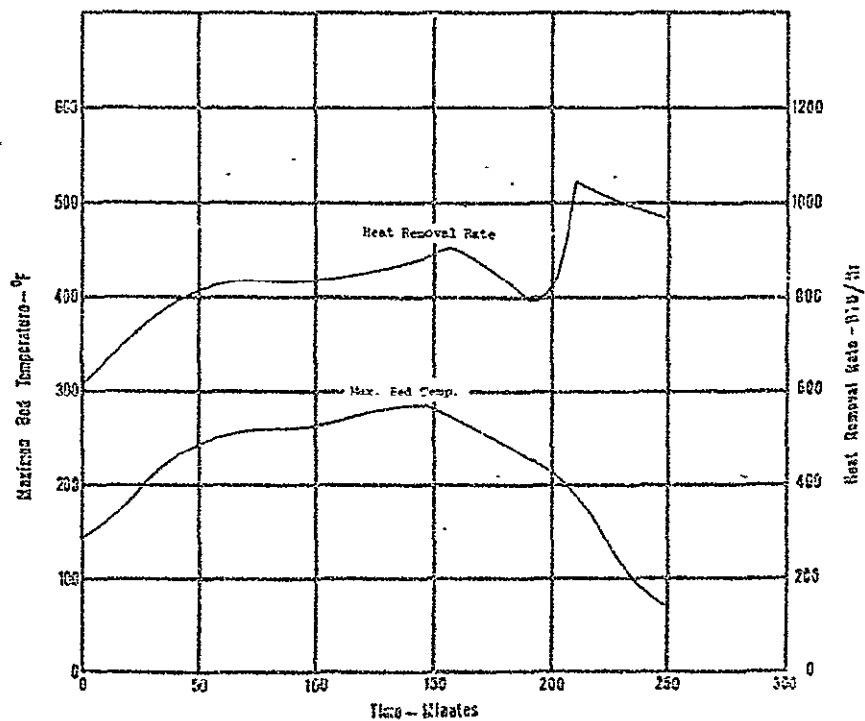
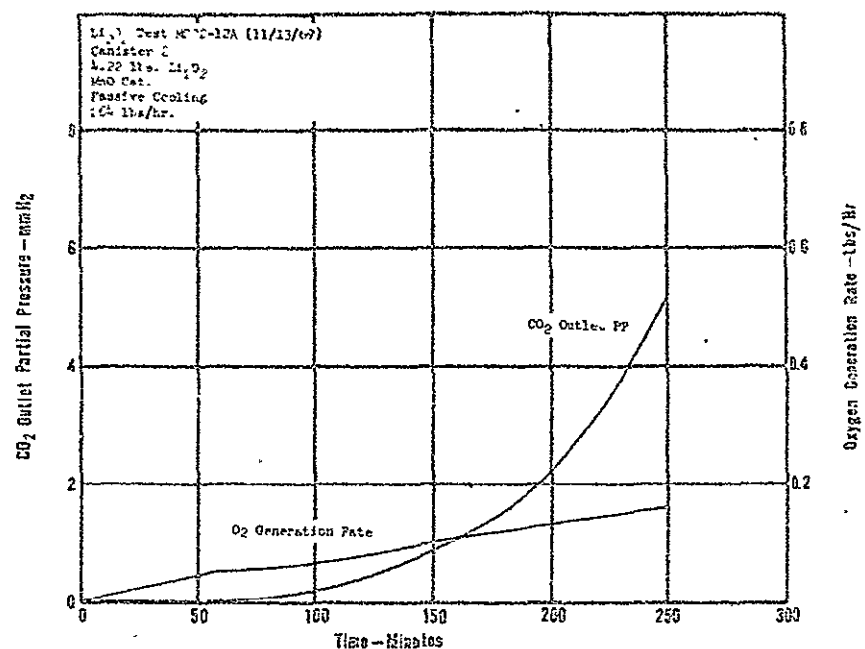


Figure 4-32

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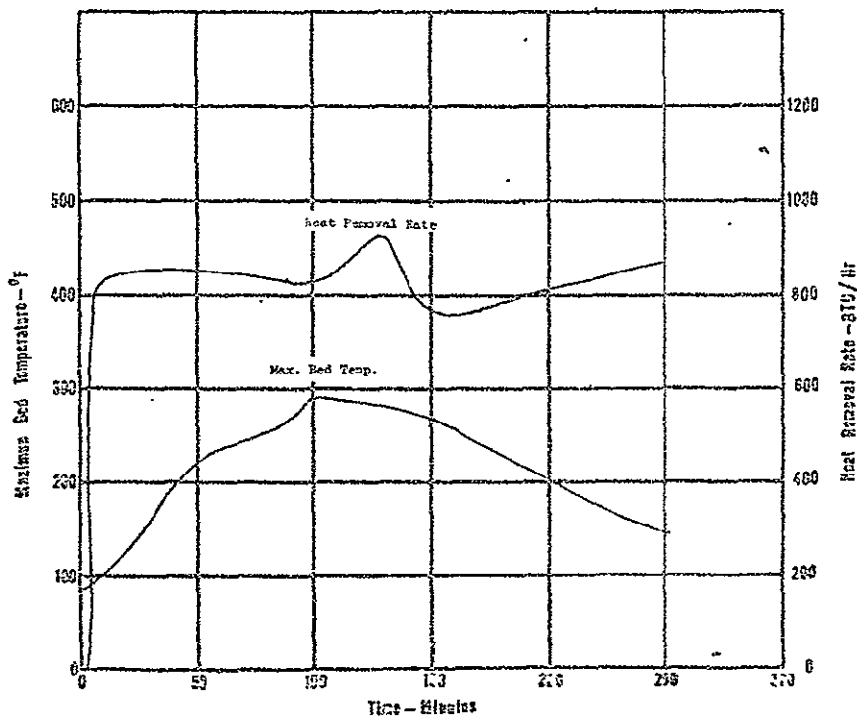
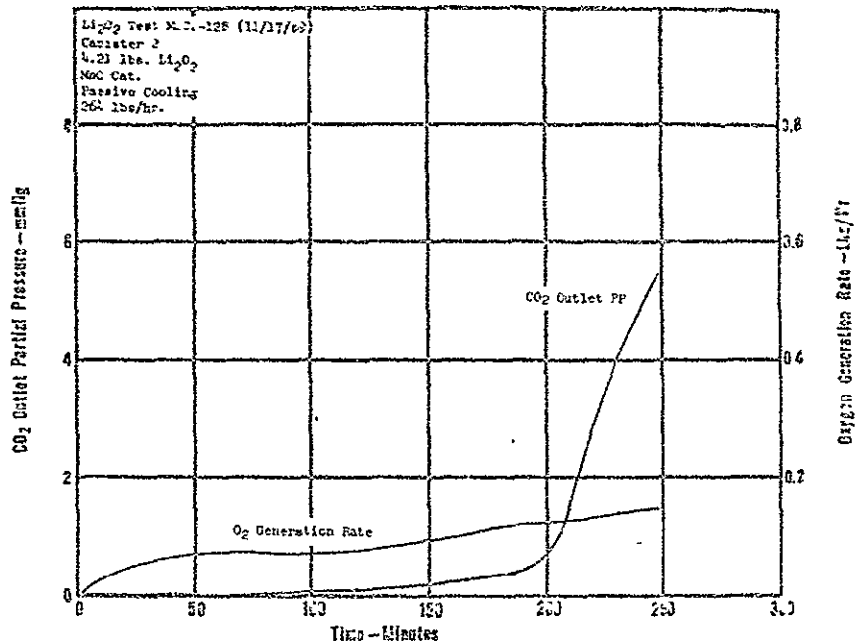


Figure 4-32

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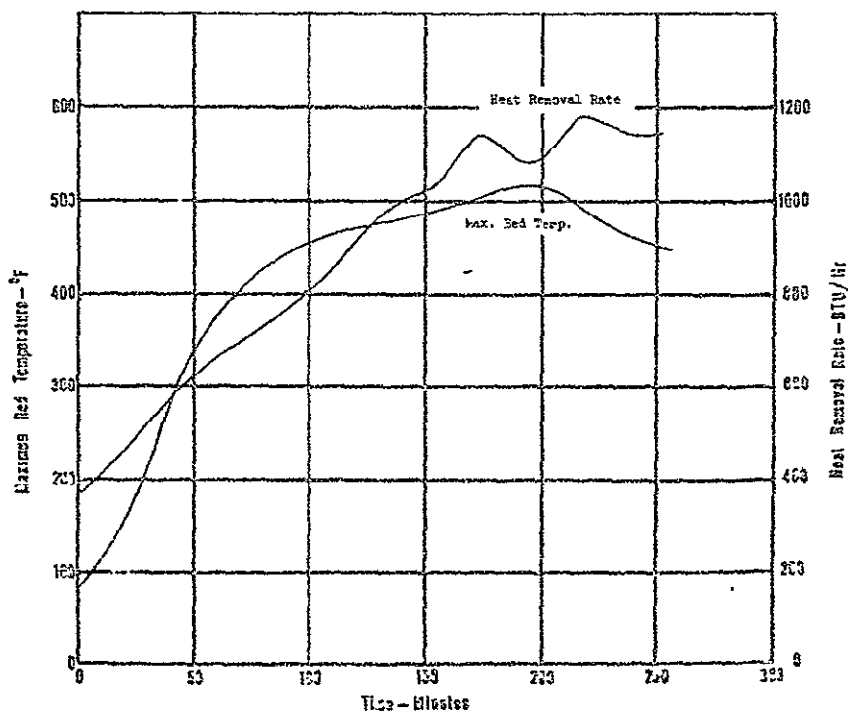
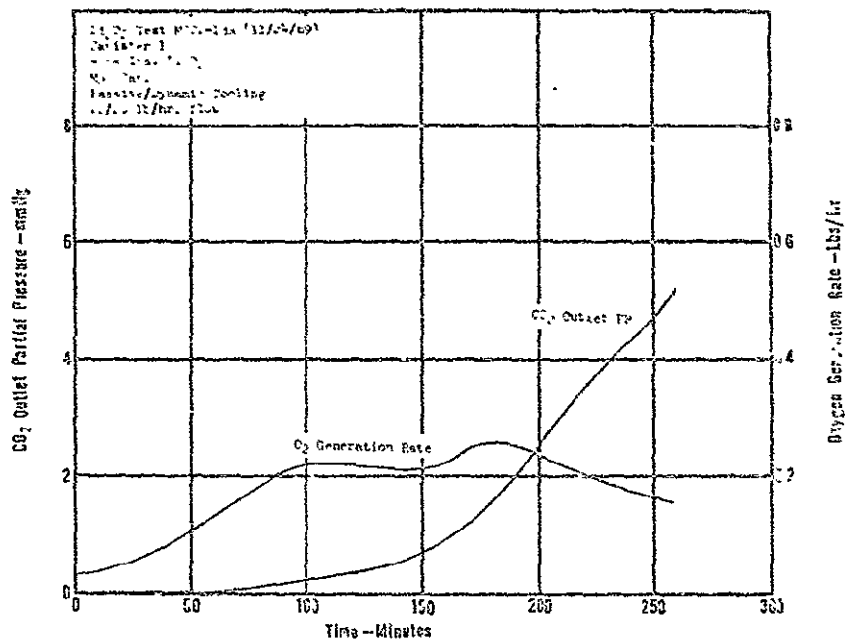


Figure 4-33

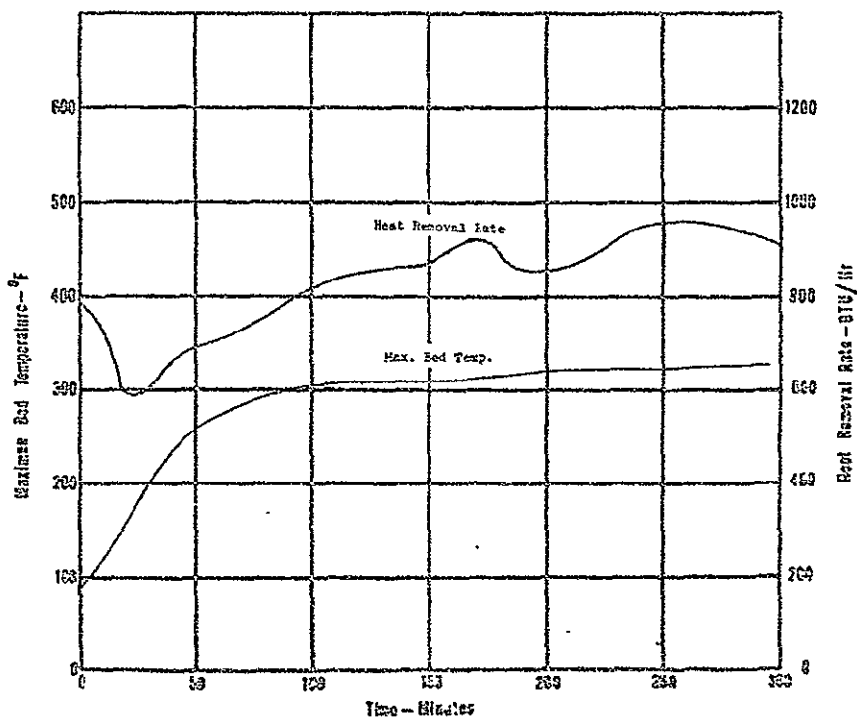
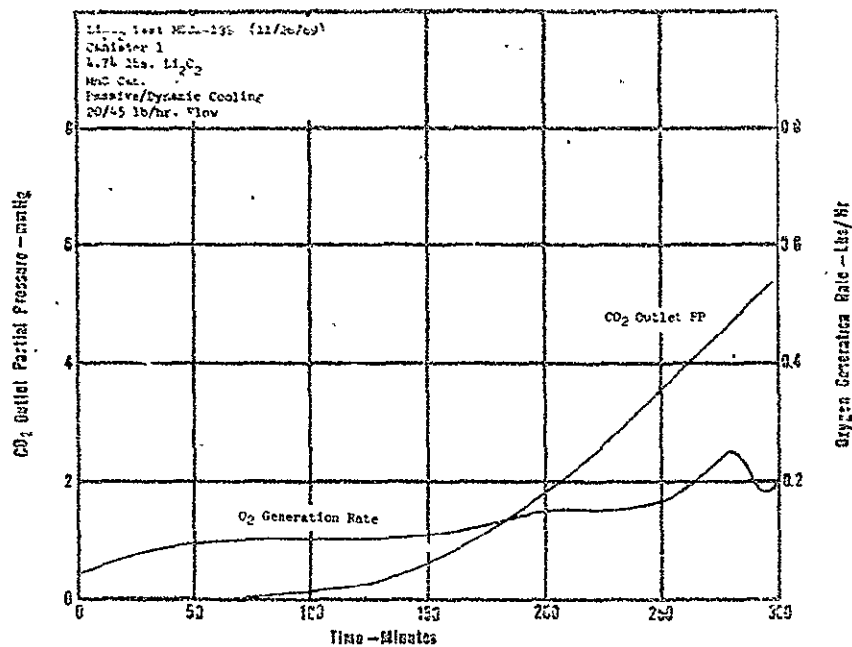


Figure 4-10

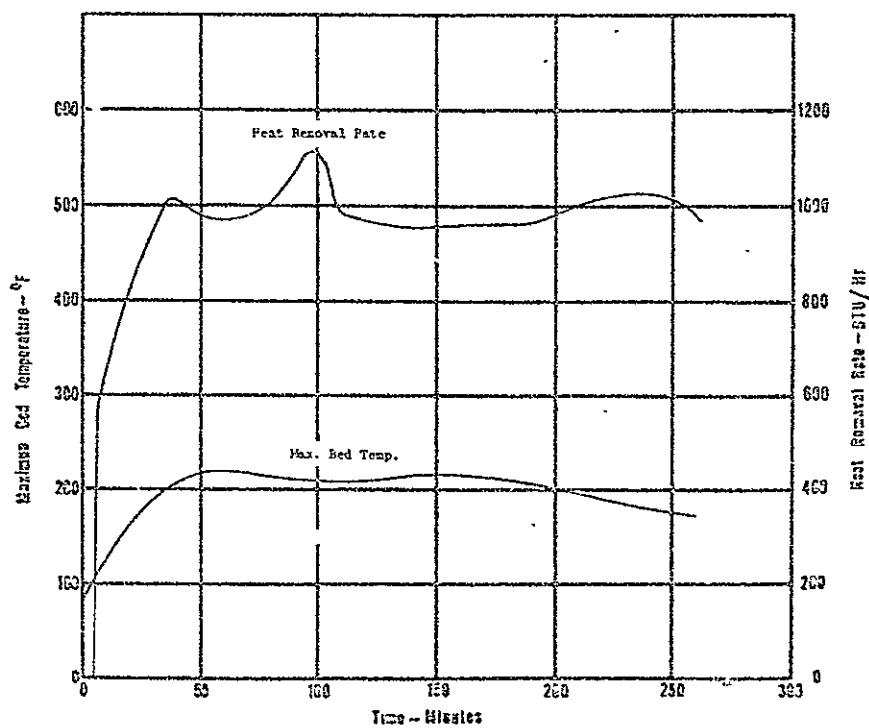
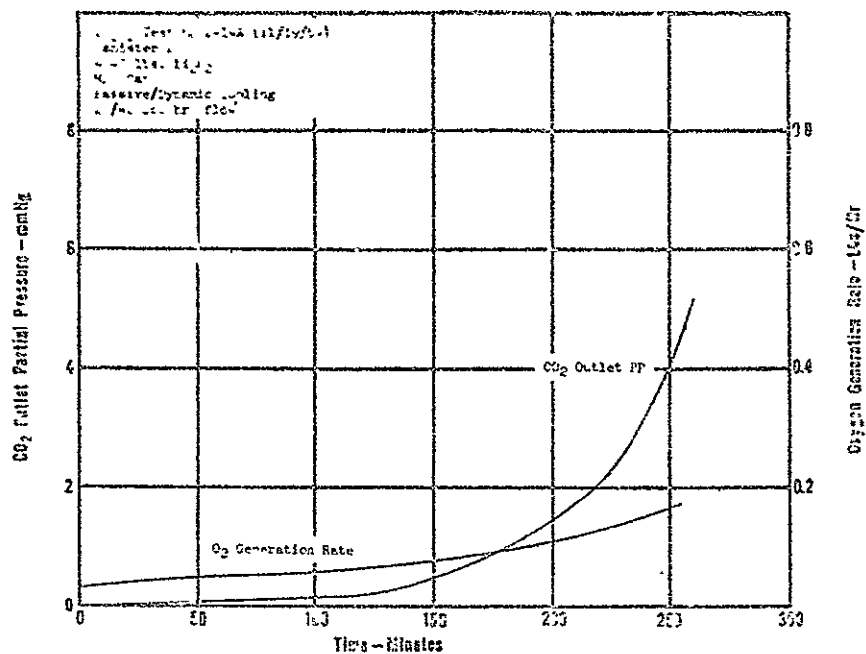
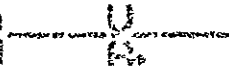


Figure 4-35

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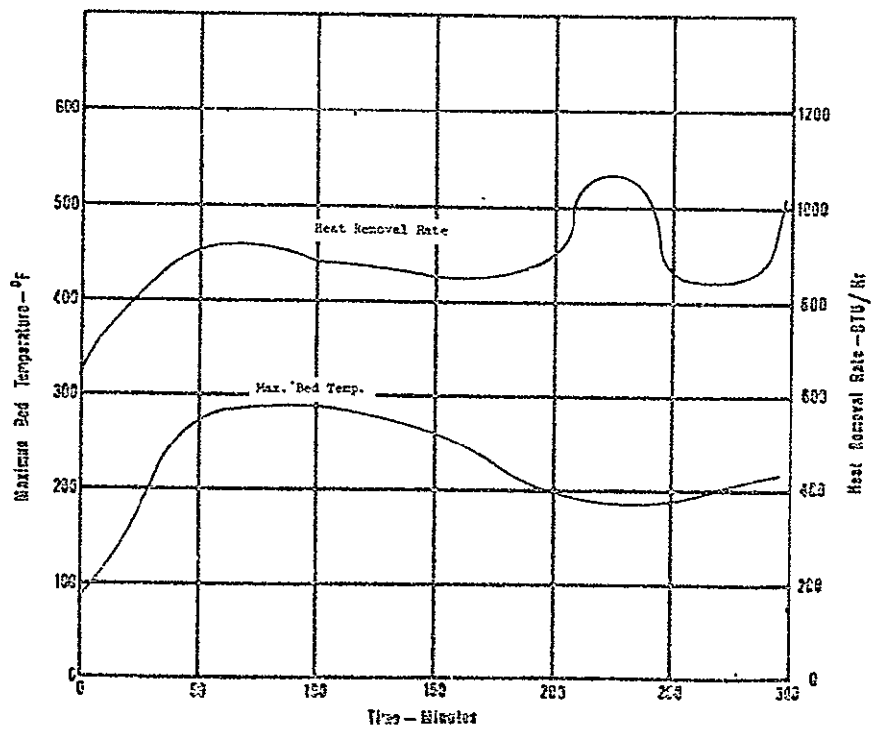
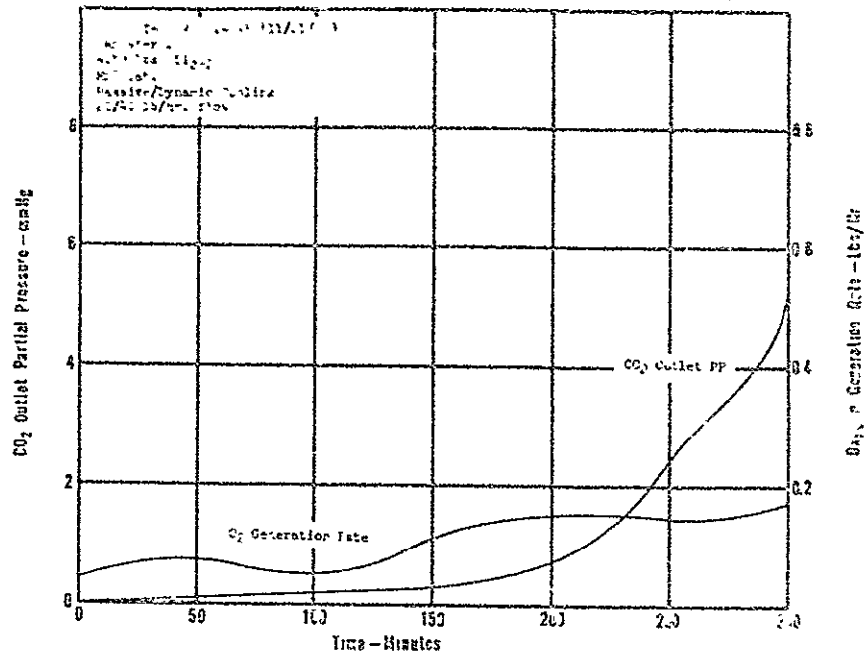


Figure 6-35

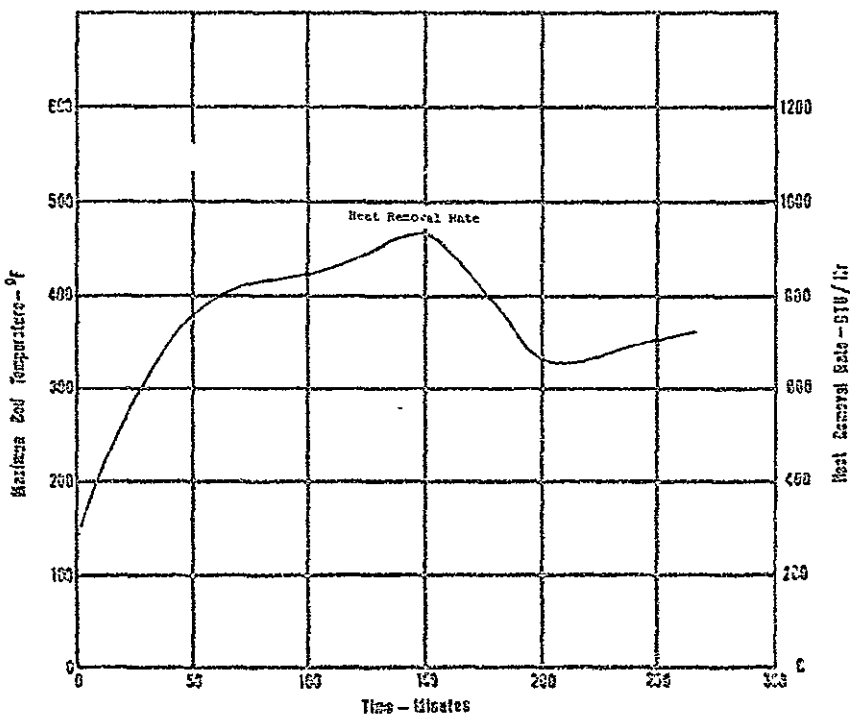
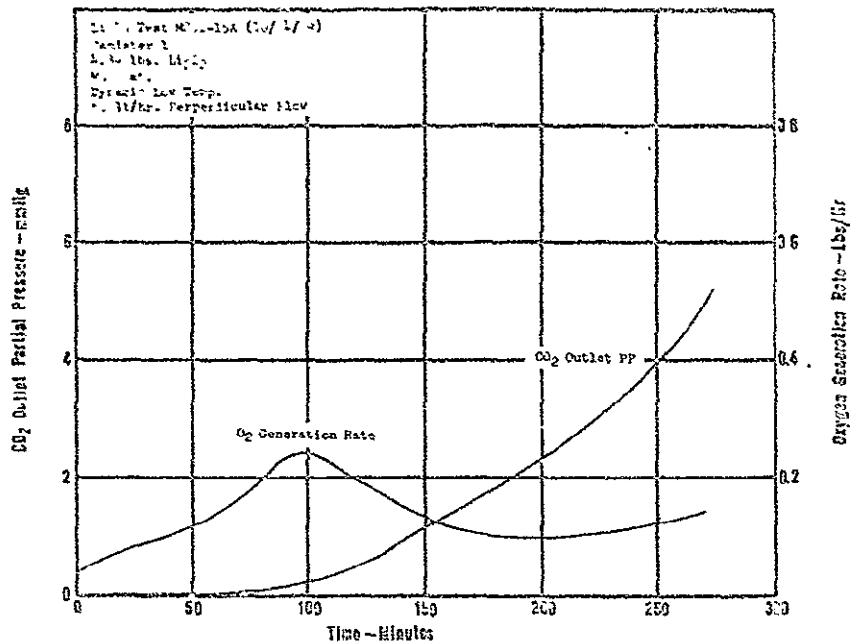


Figure 4-37

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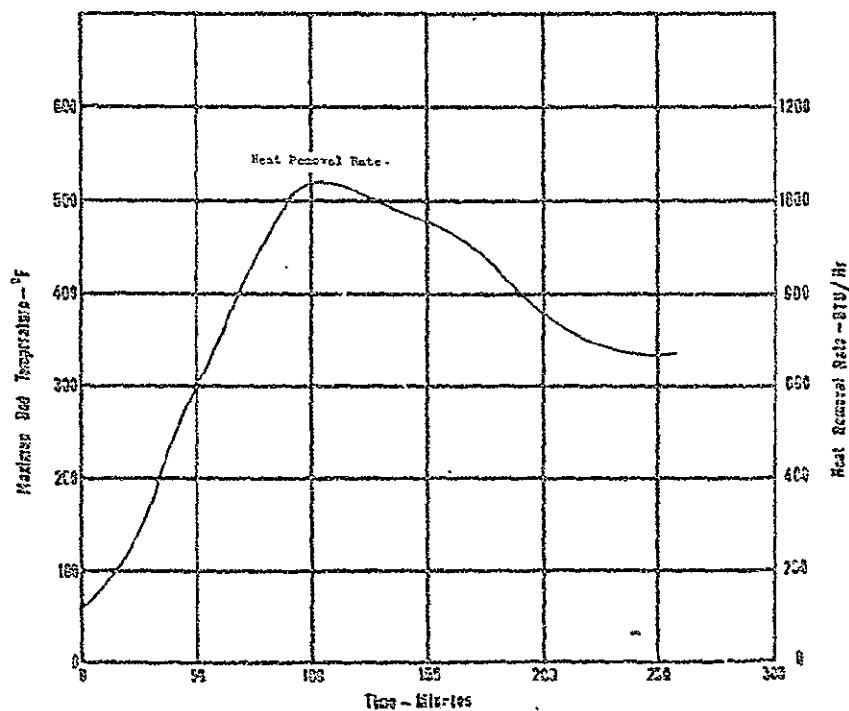
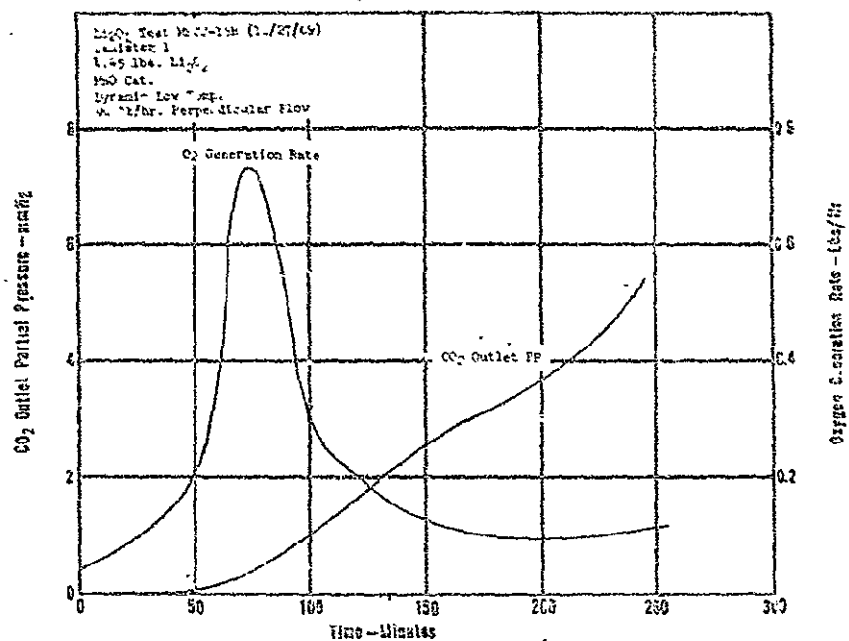


Figure 4-35

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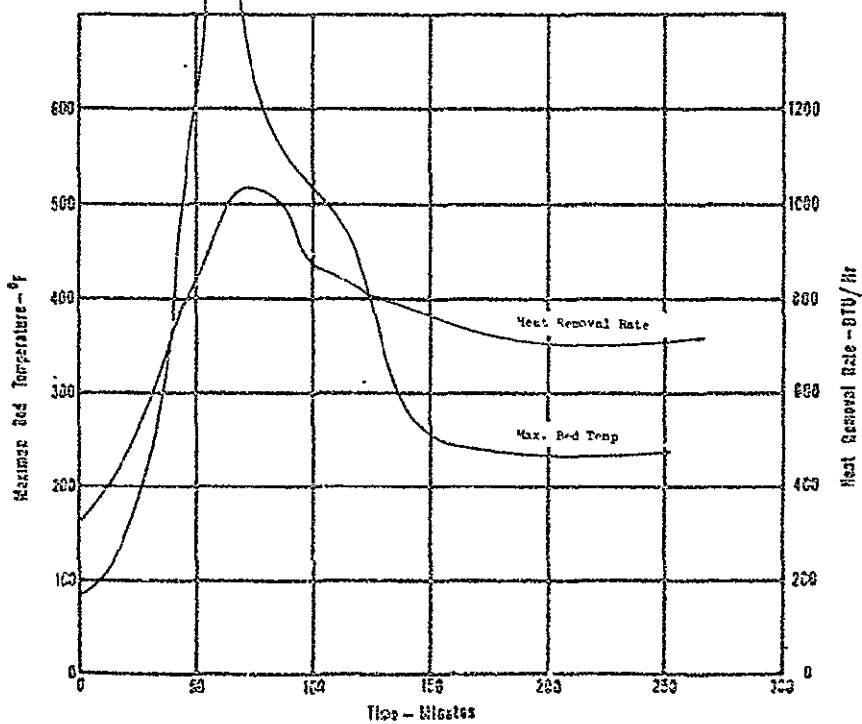
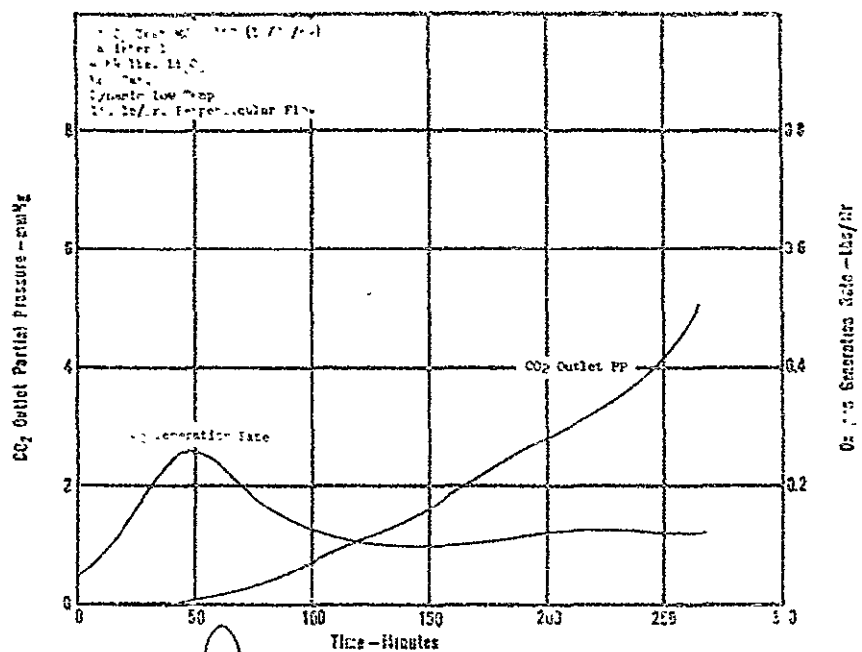


Figure L-39

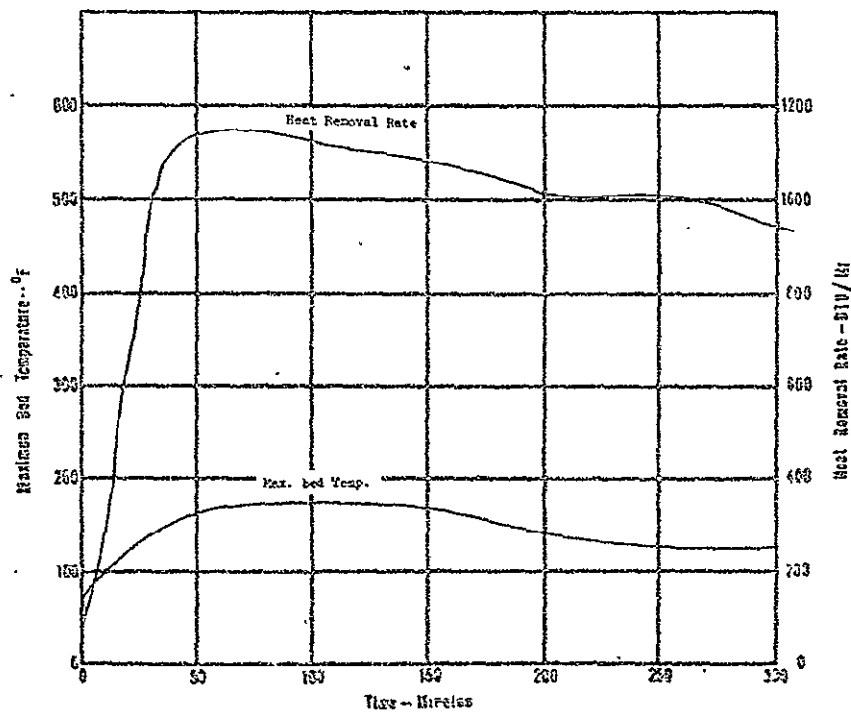
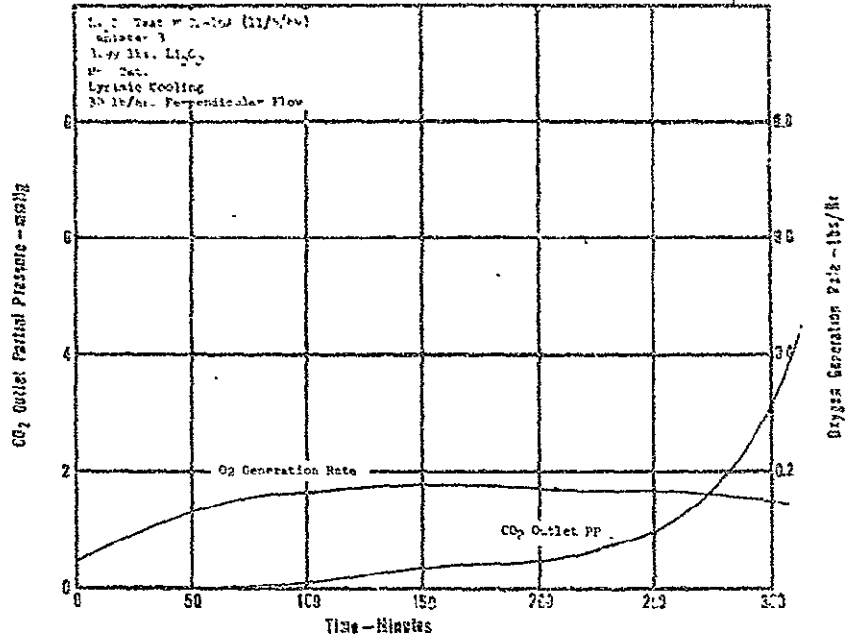


Figure 4-40

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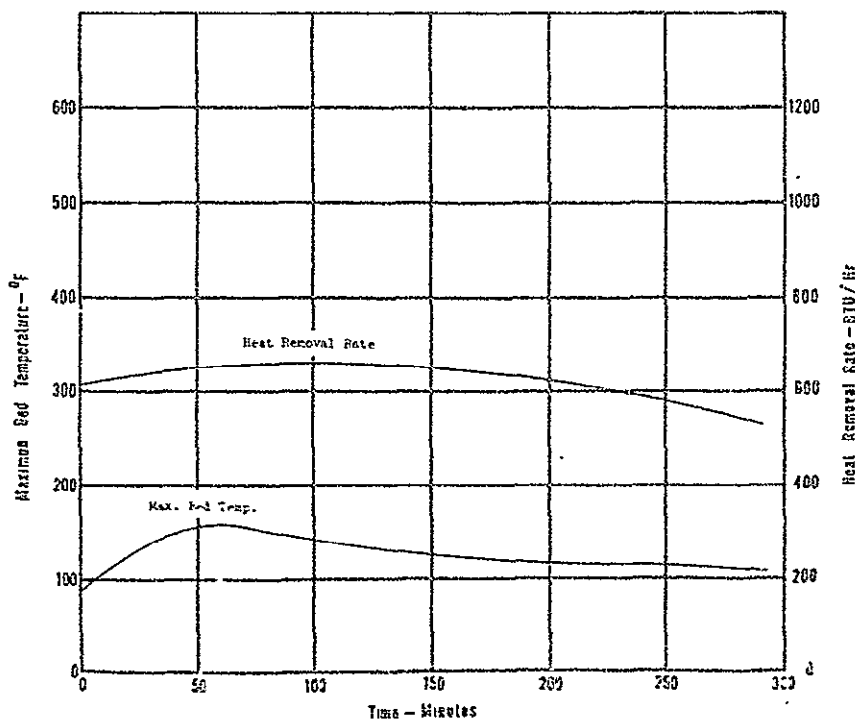
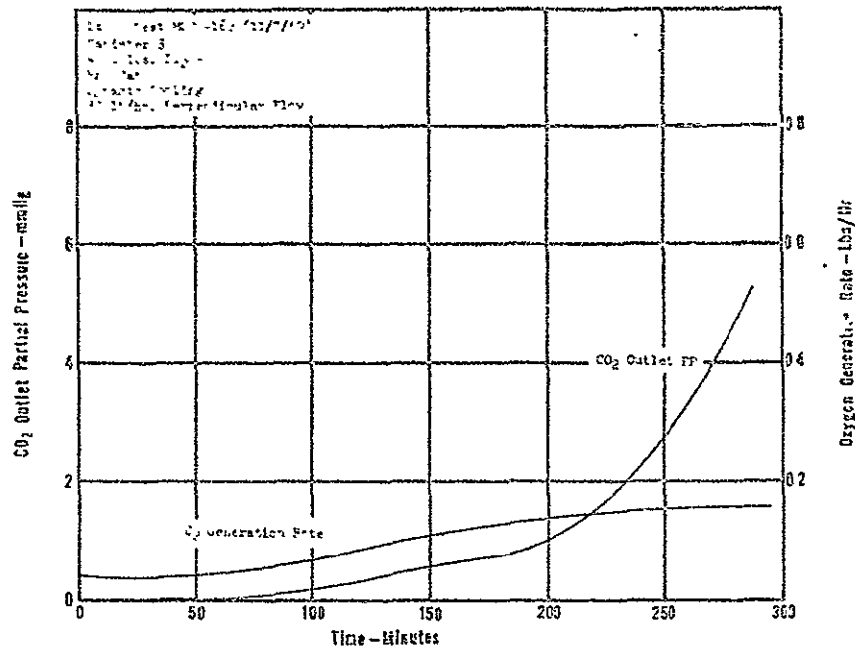


Figure 2-21

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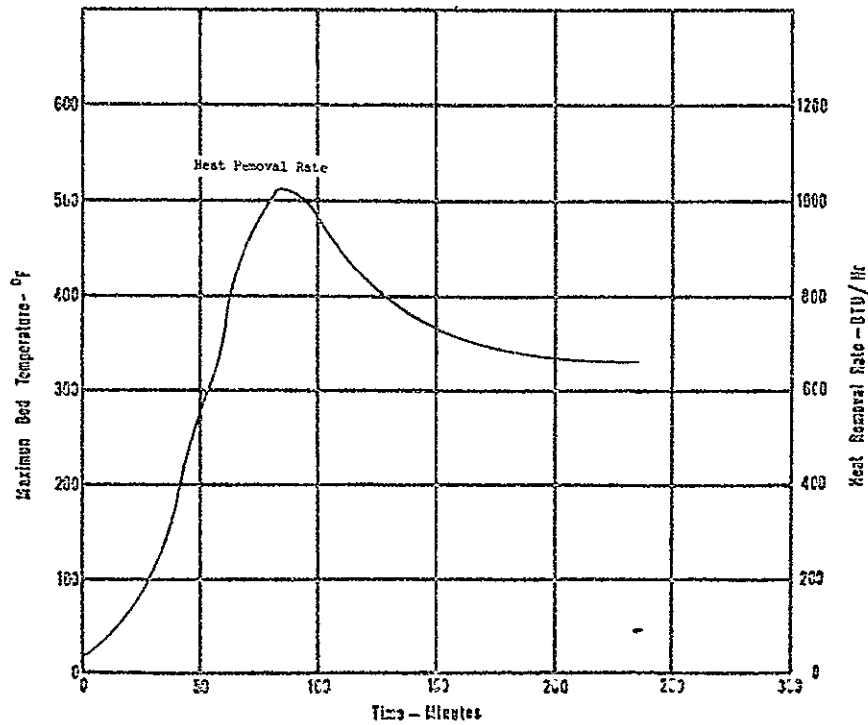
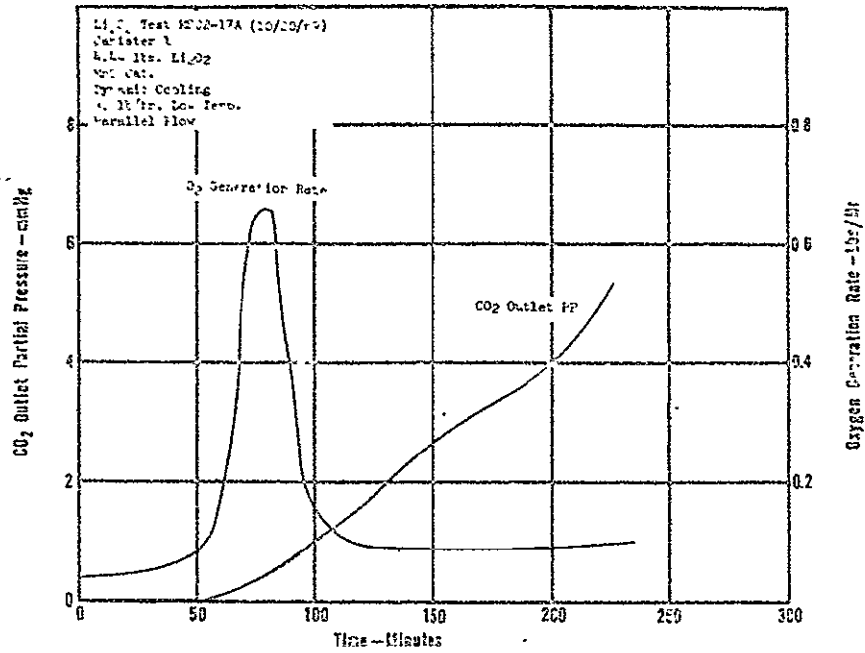


Figure 4-42

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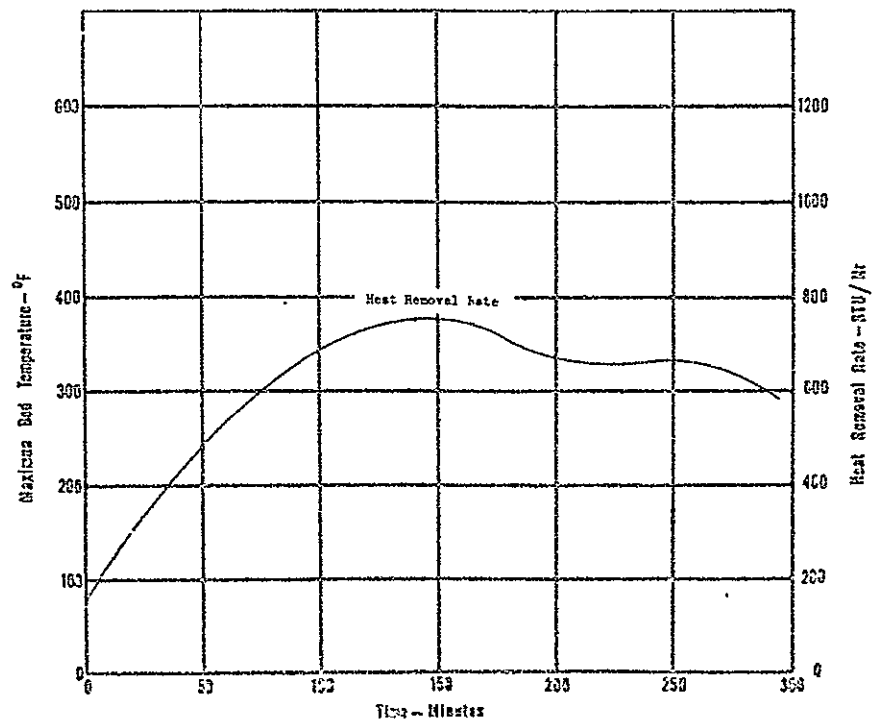
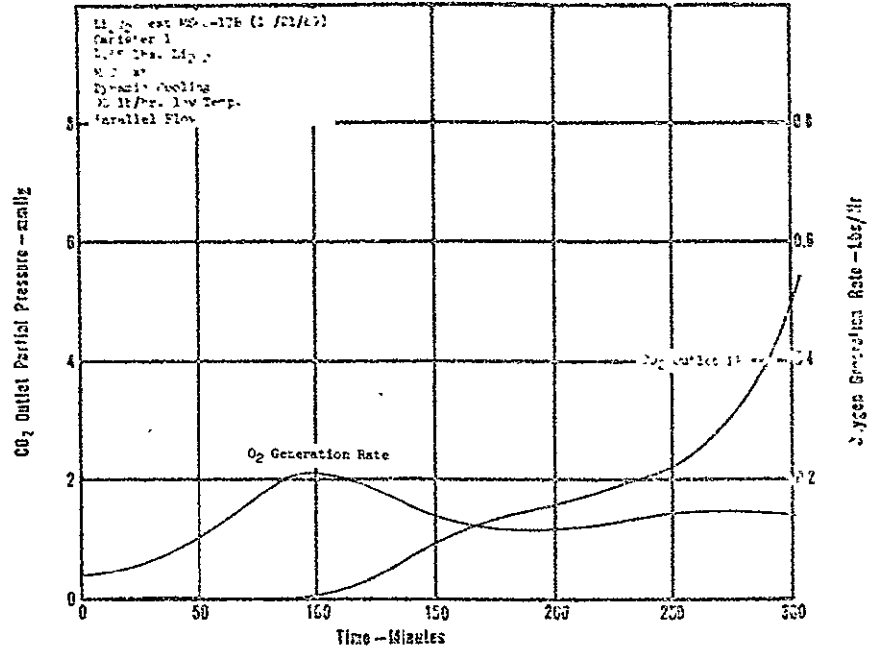


Figure 4-4.3

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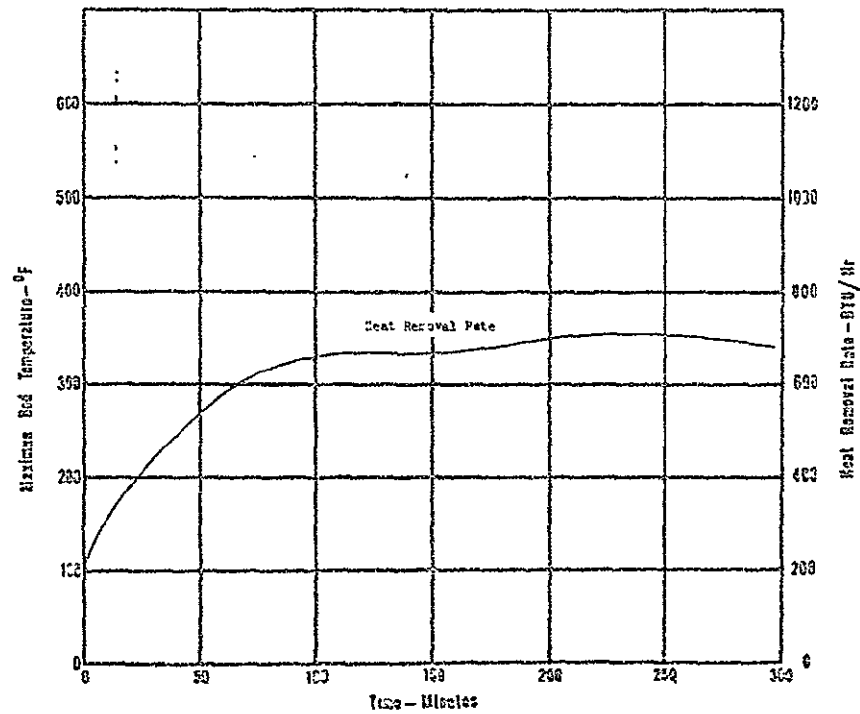
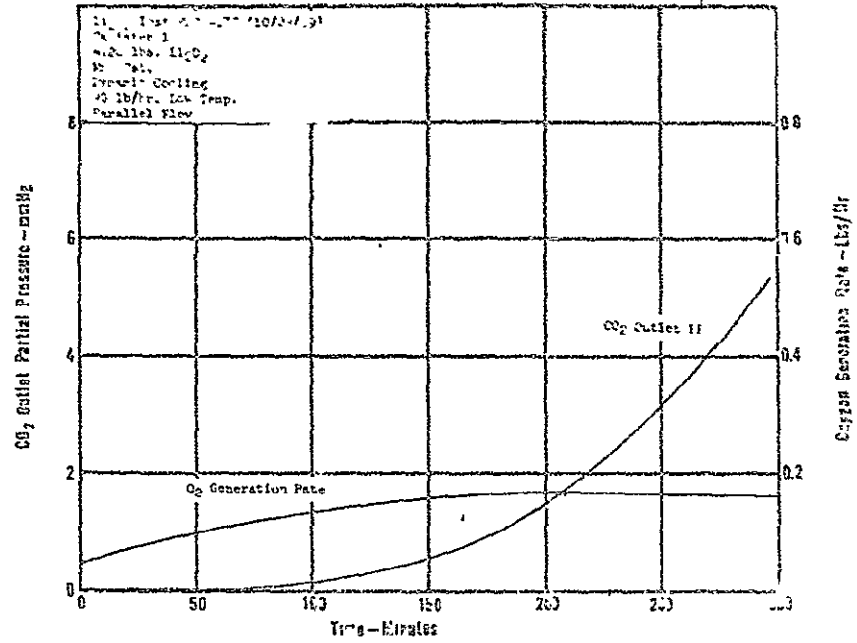


Figure 4-4b

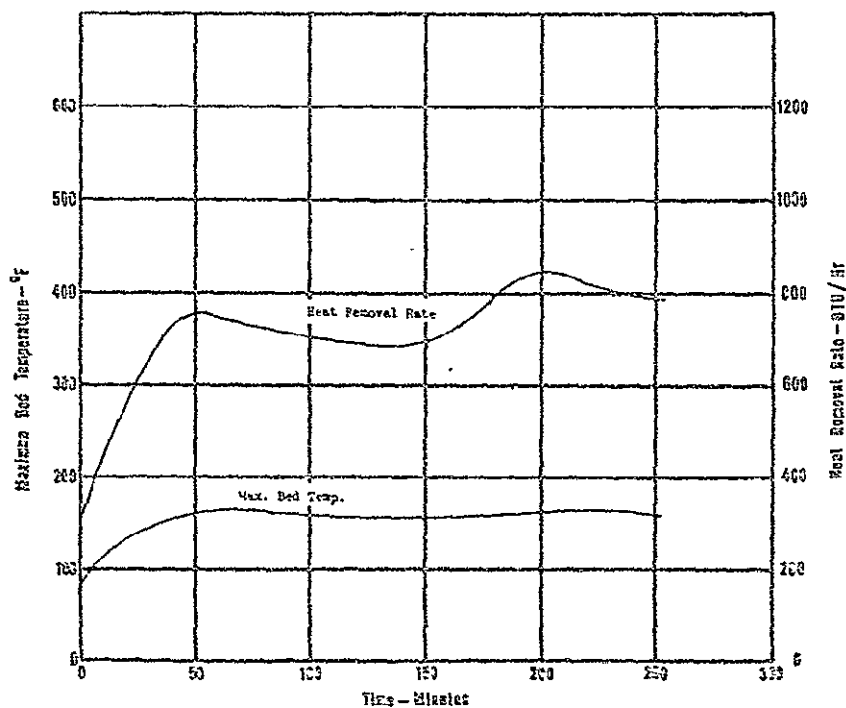
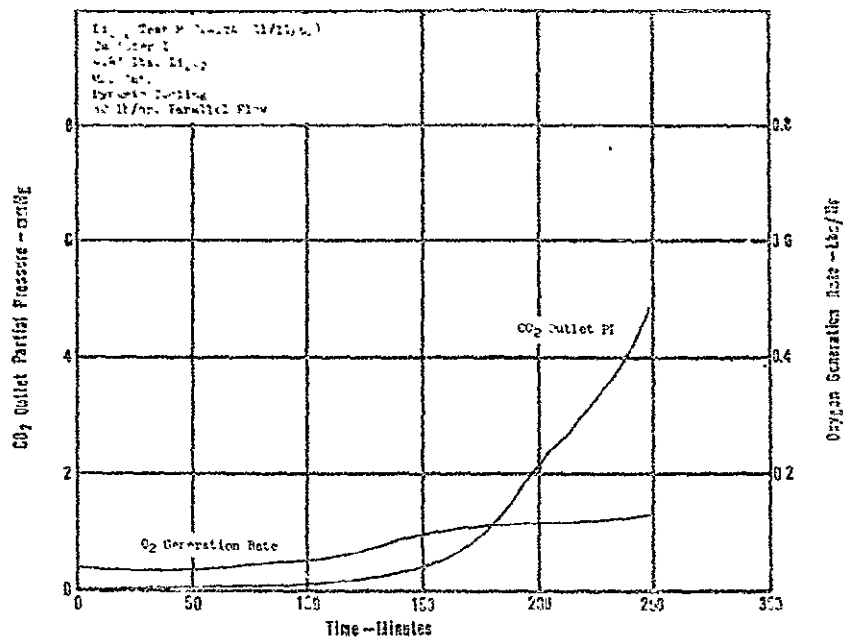


Figure 4-4.5

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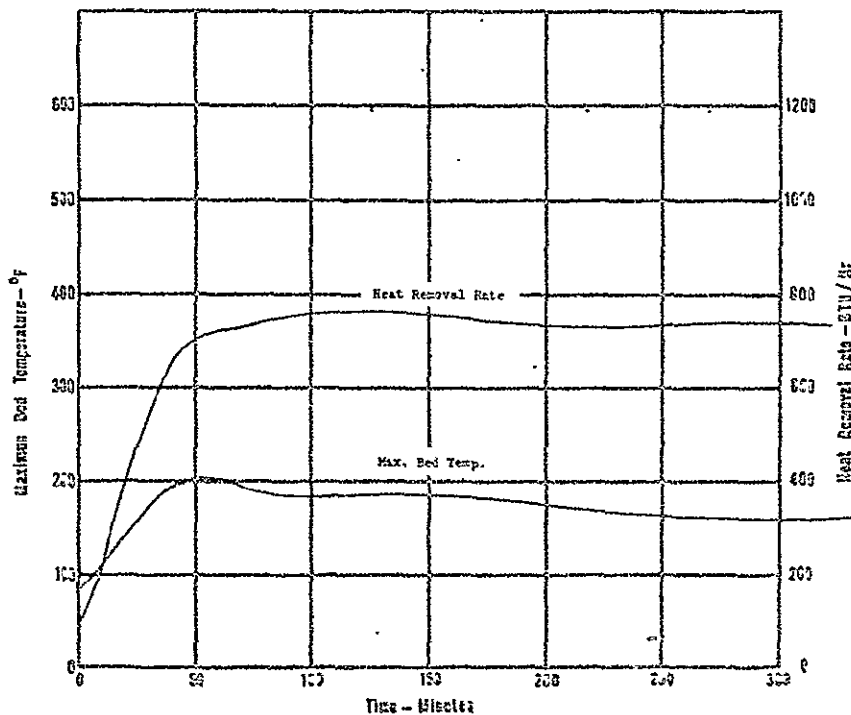
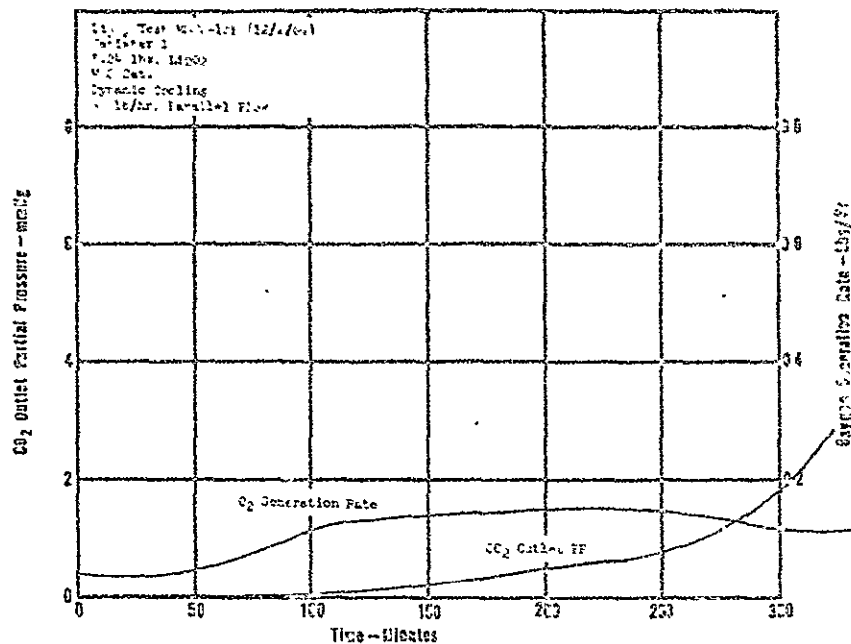


Figure 1-46

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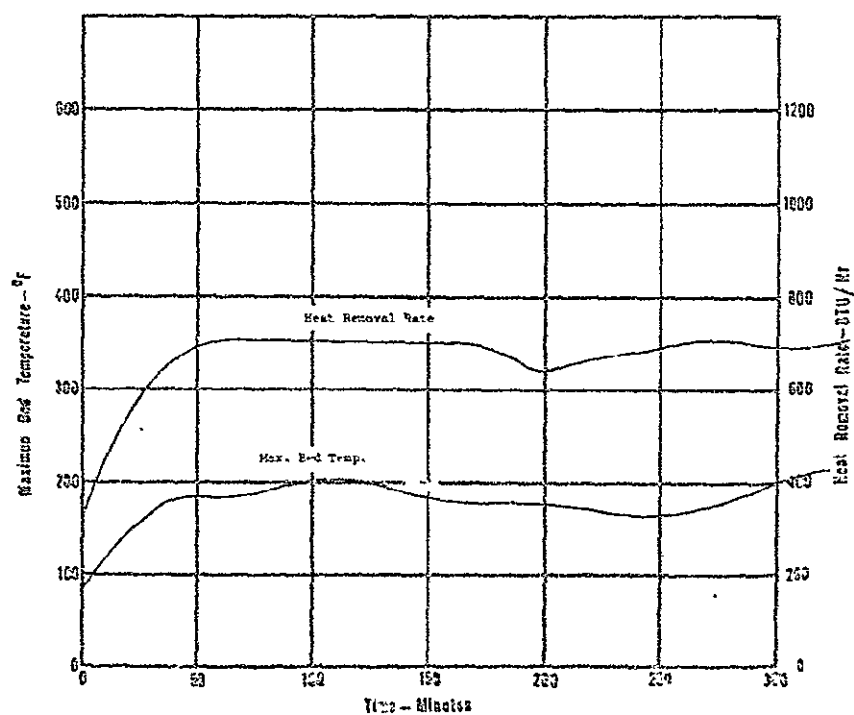
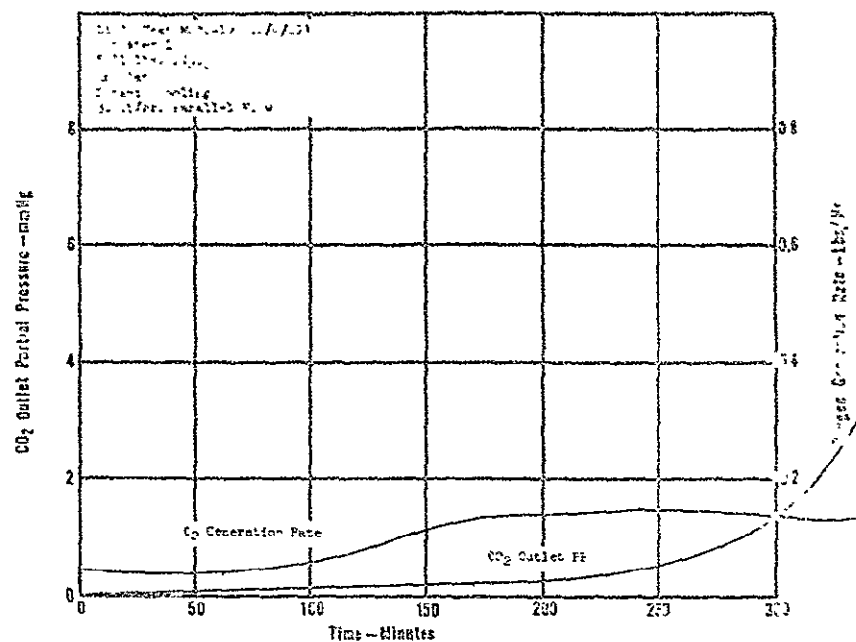


Figure 4-4?

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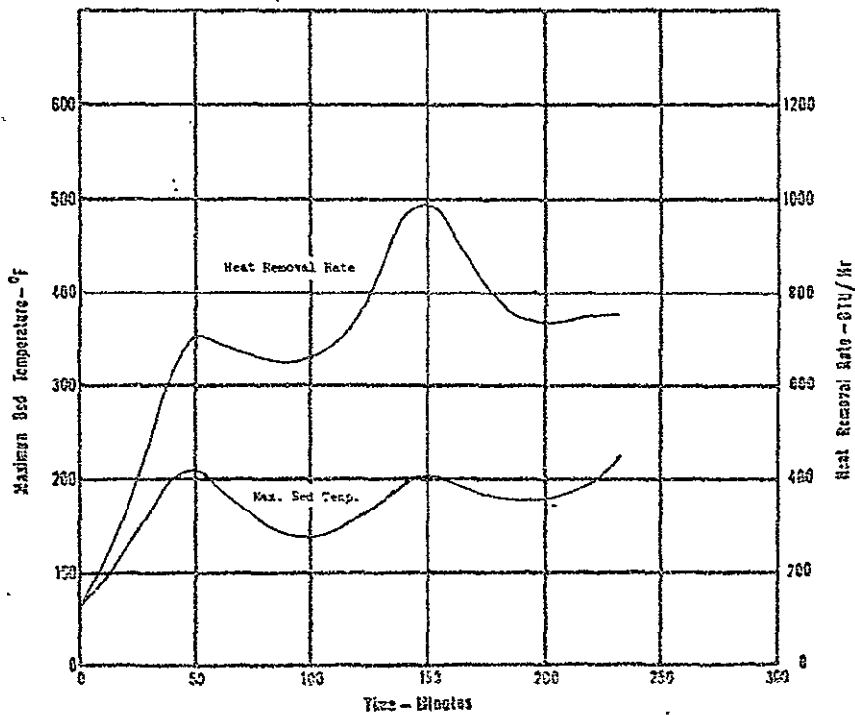
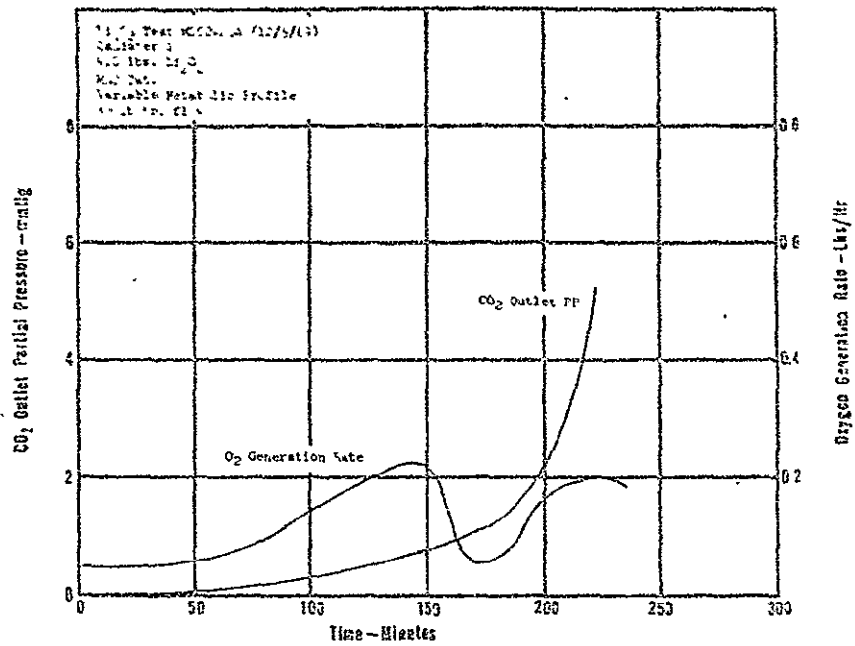


Figure 4-45

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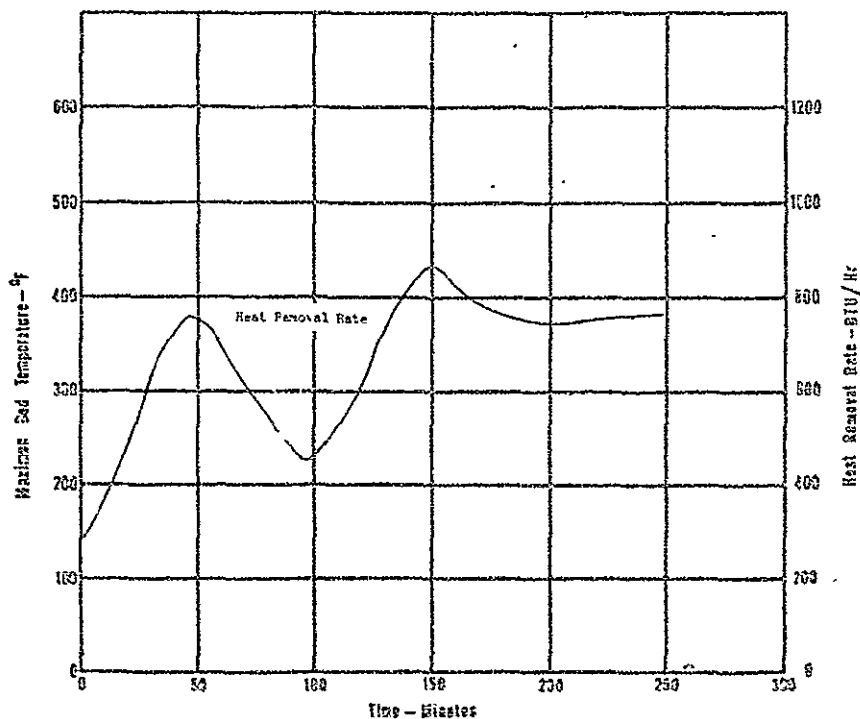
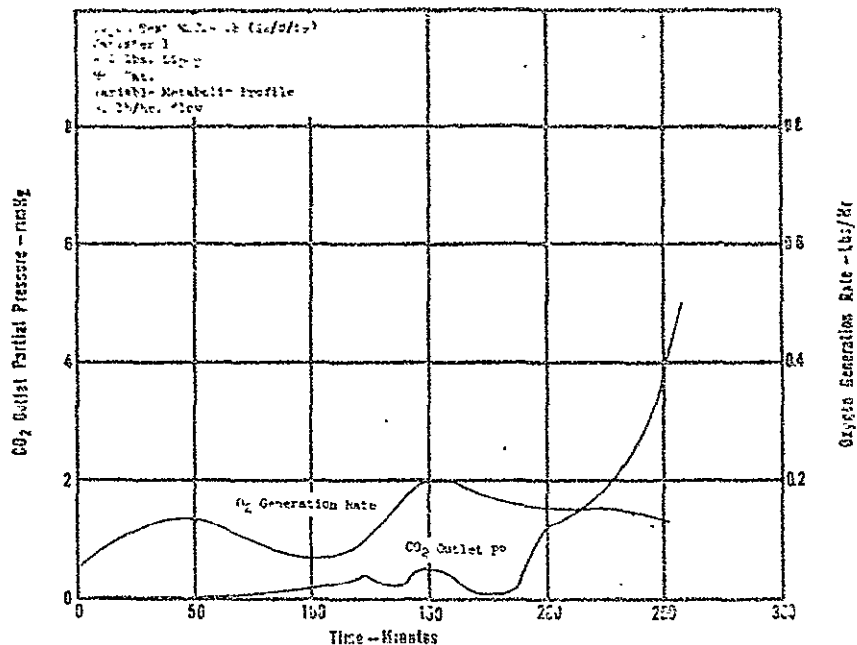


Figure 4-4.9

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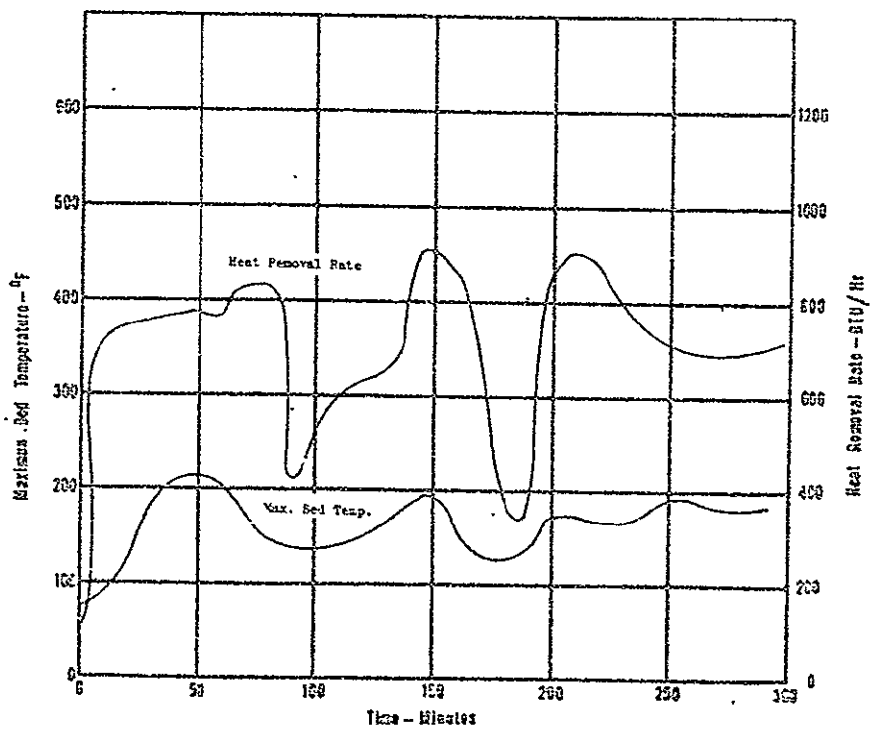
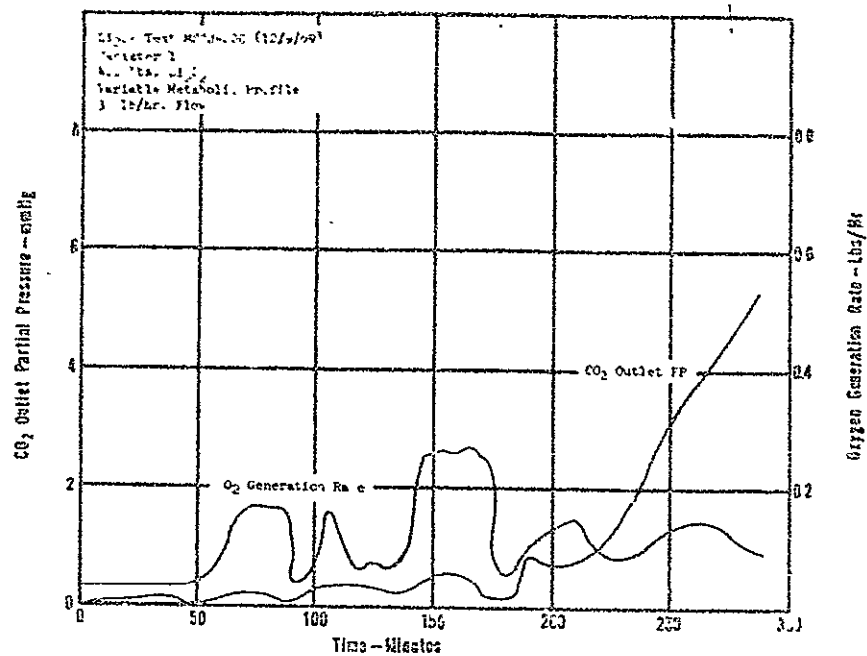


Figure 4-50

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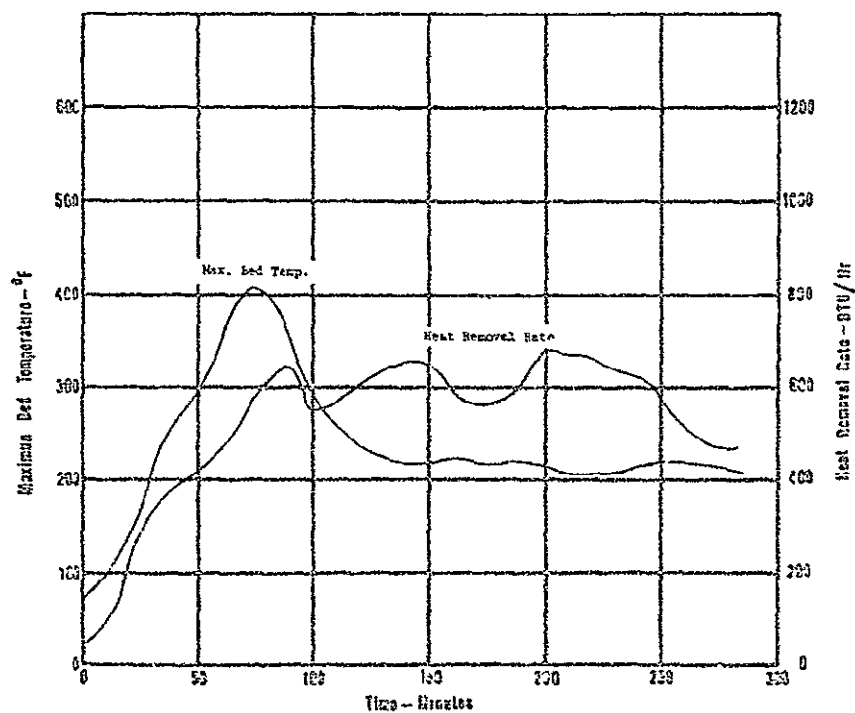
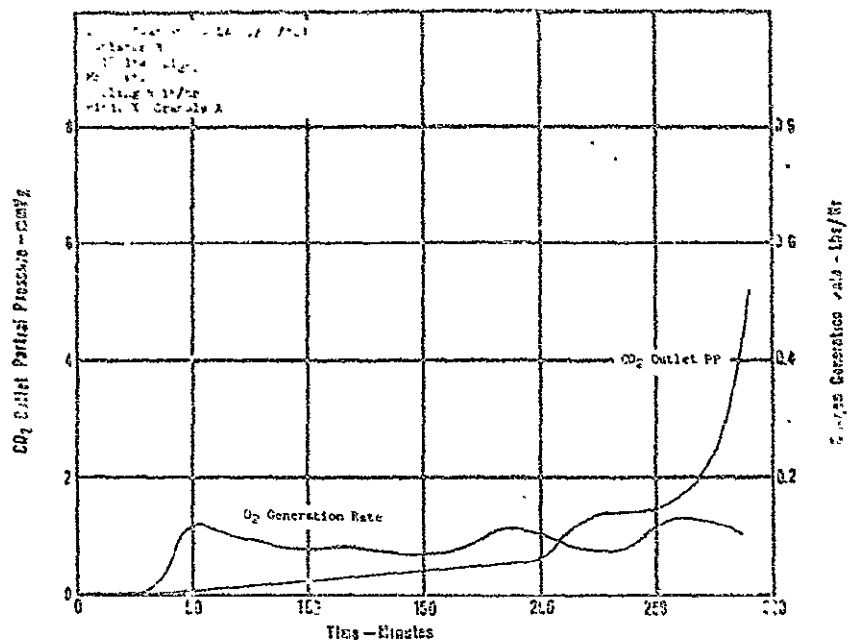


Figure 4-51

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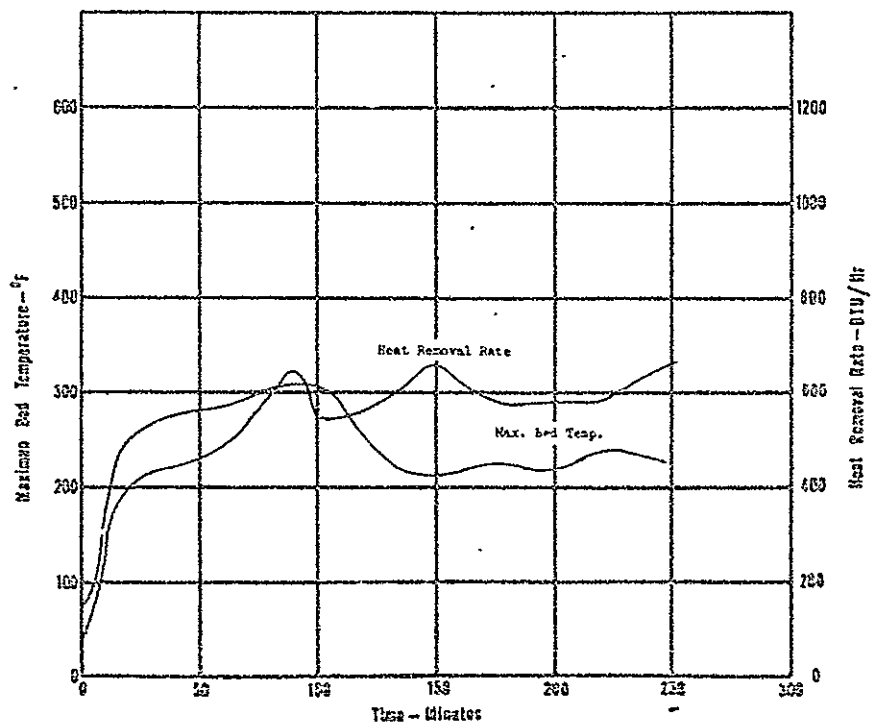
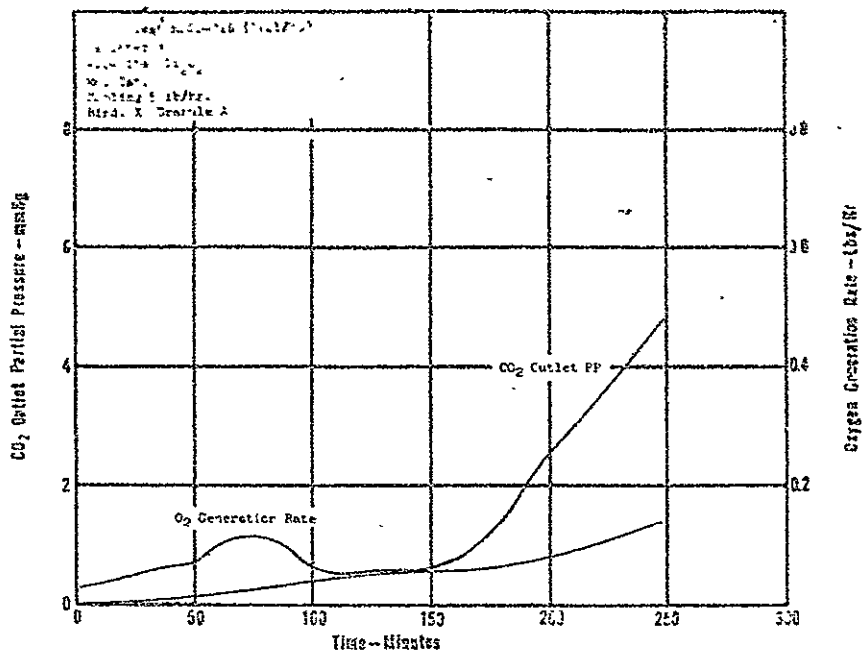


Figure 4-52

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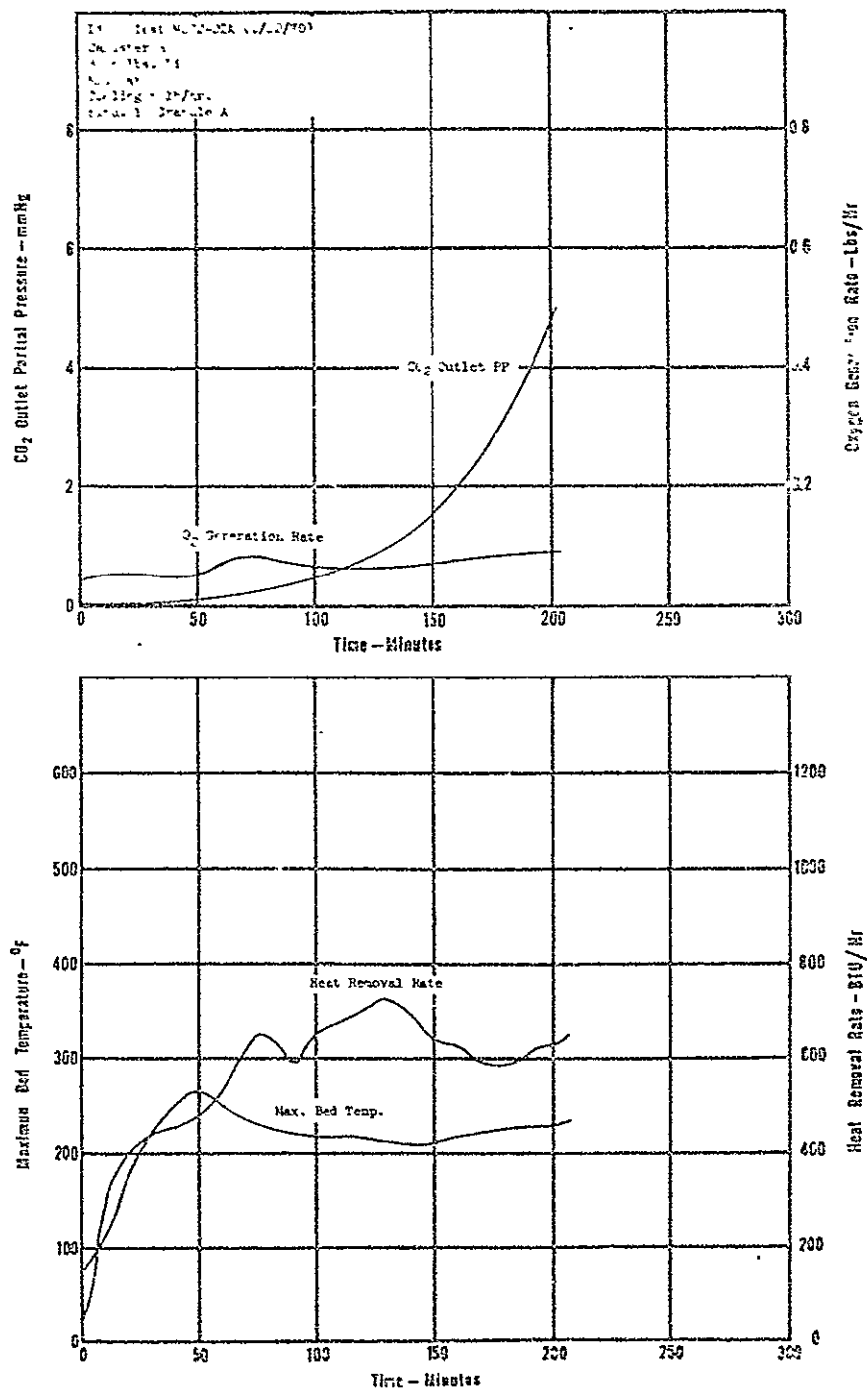


Figure 4-53

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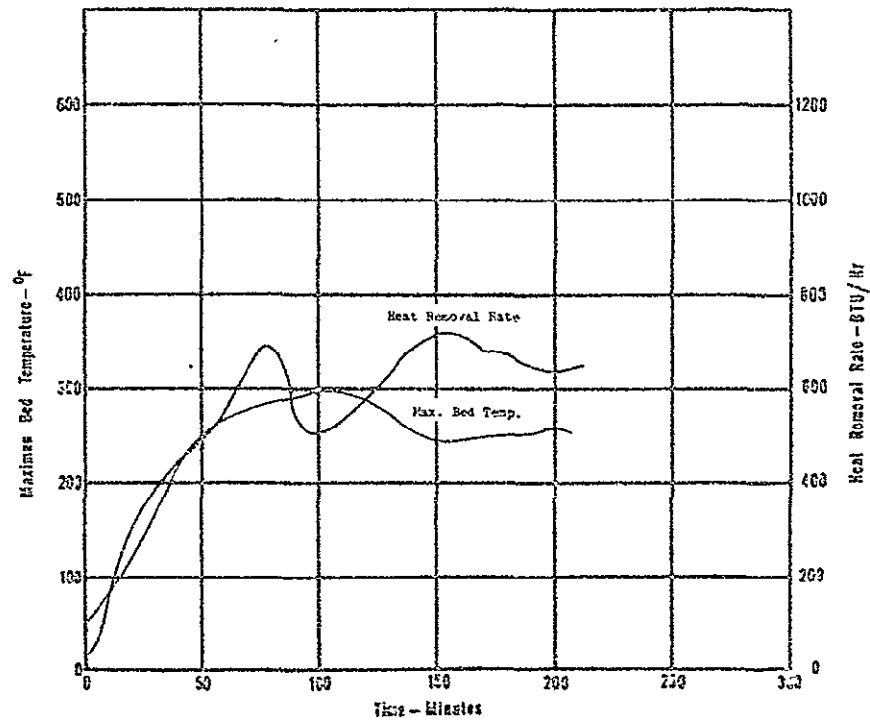
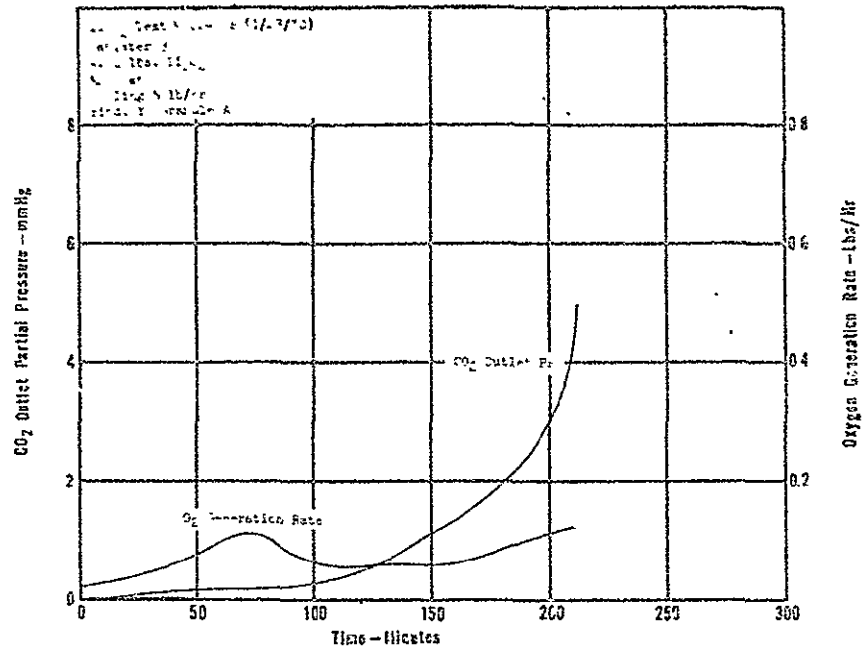


Figure 4-5a

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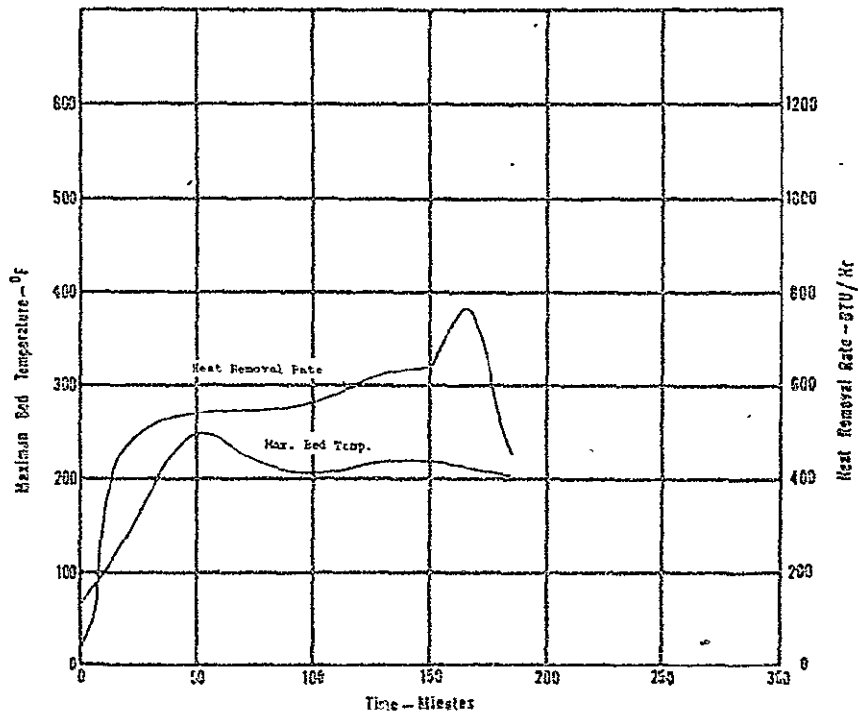
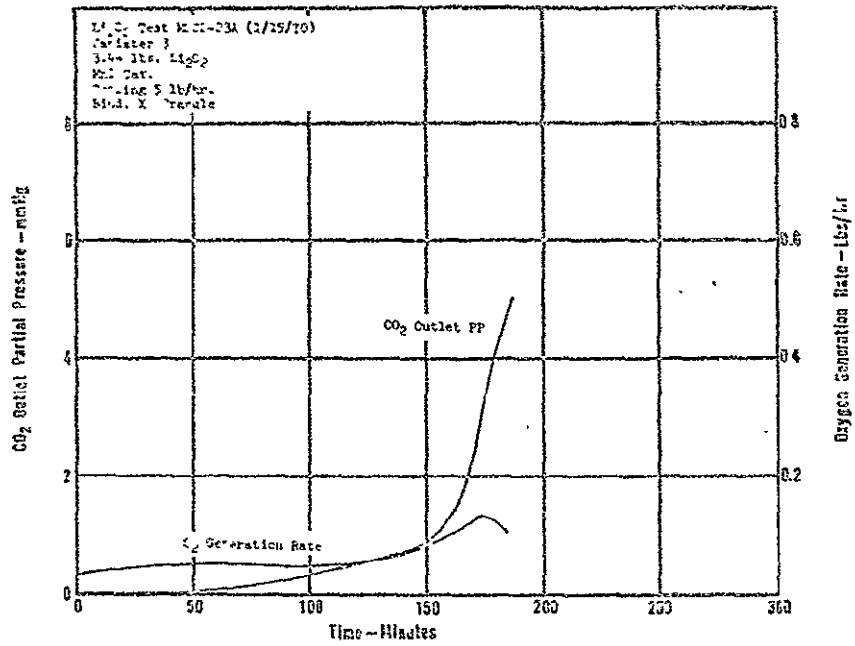


Figure 4-55

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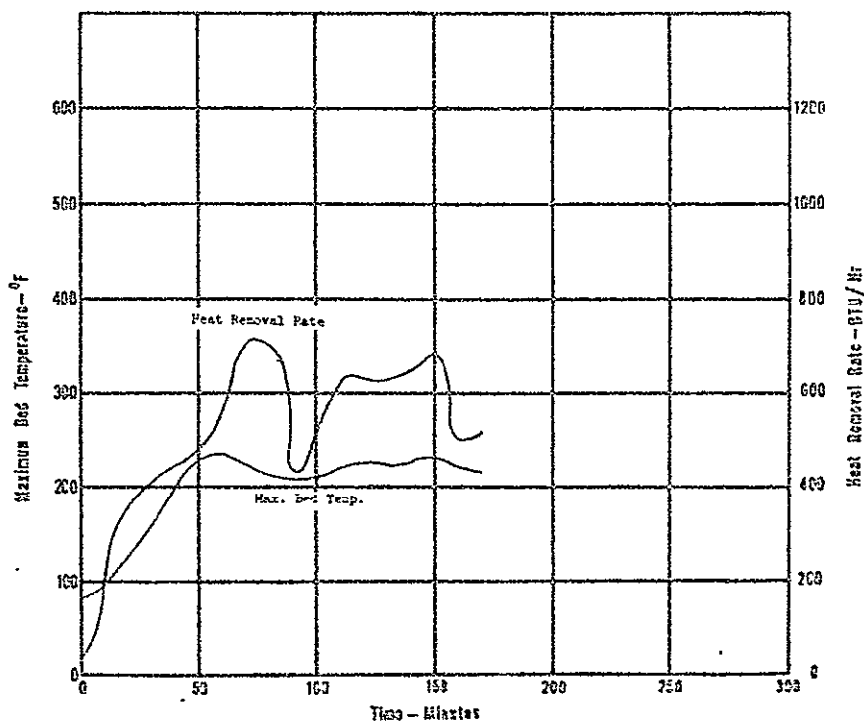
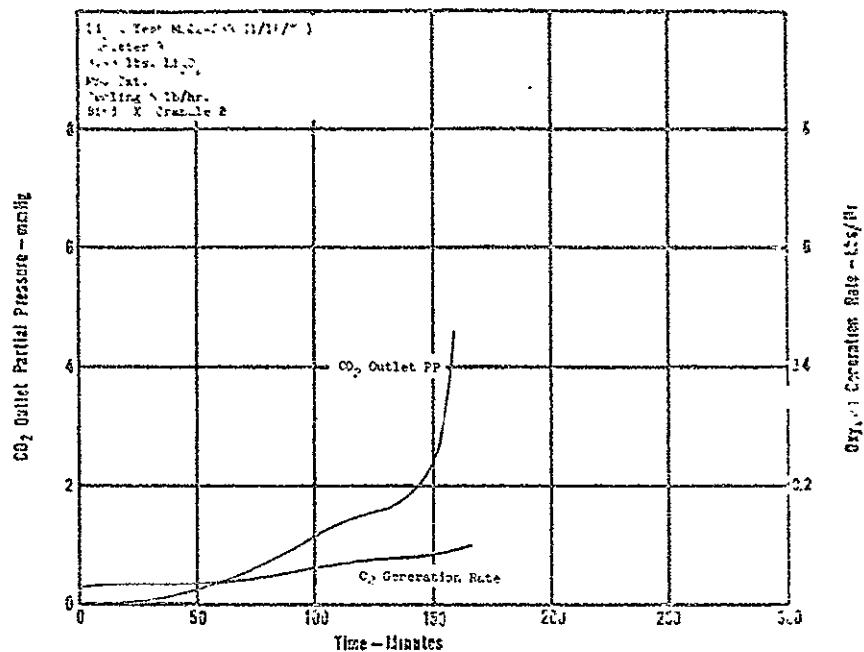


Figure L-56

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Page 5712

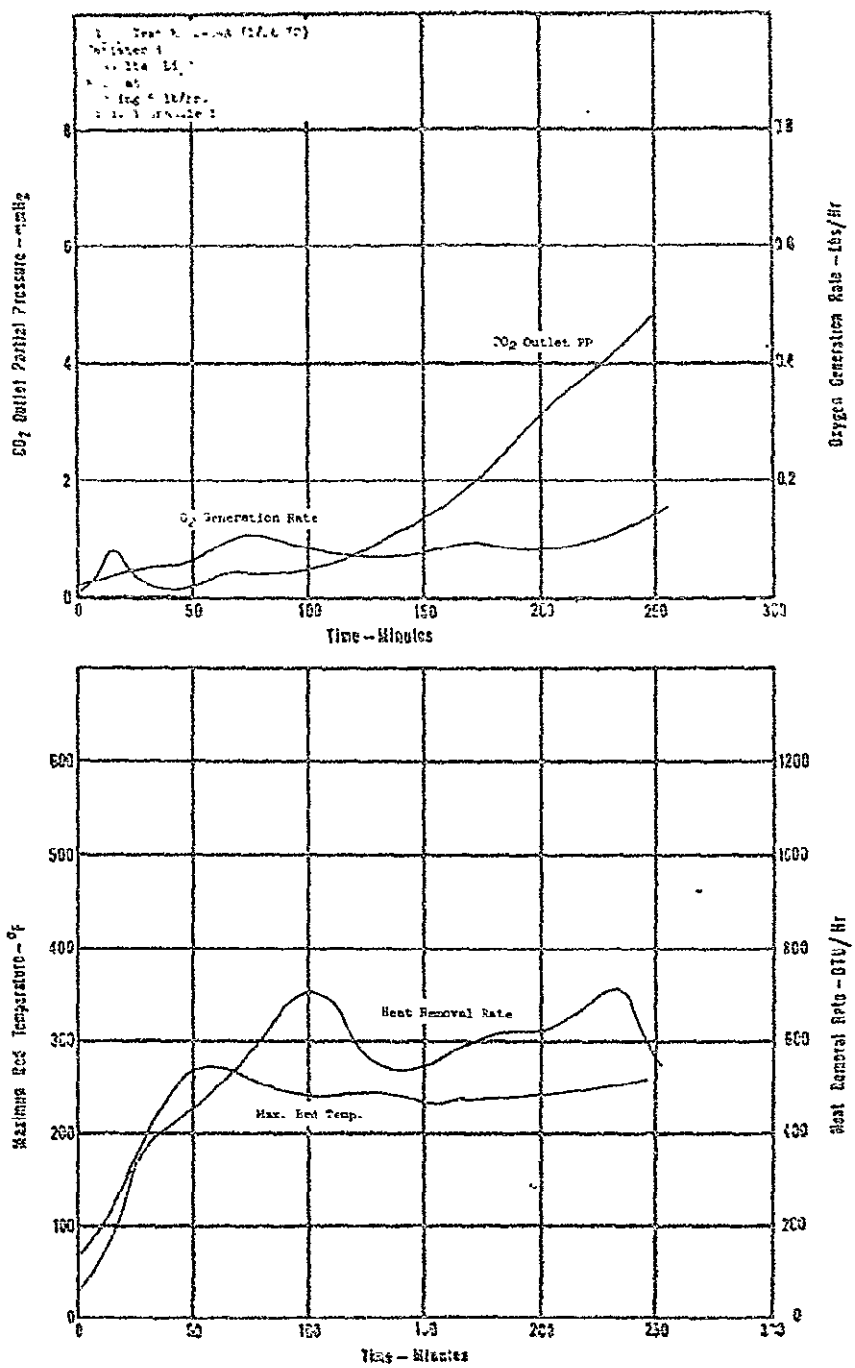


Figure 4-57

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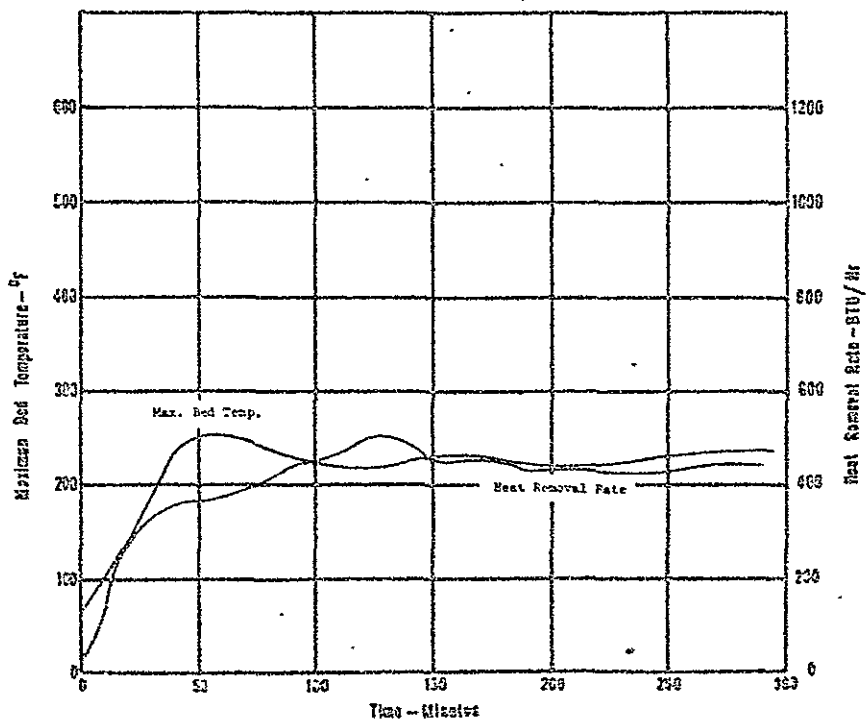
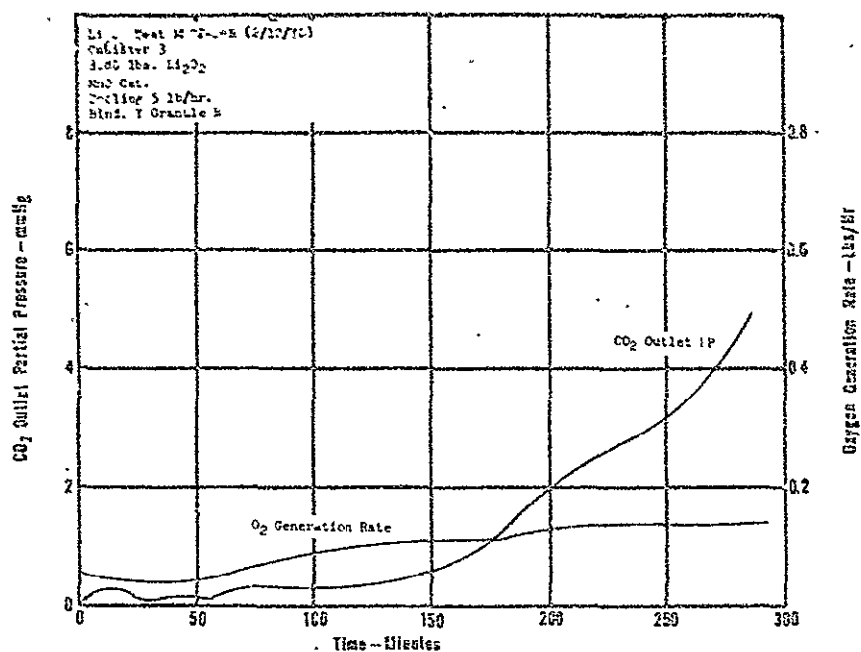


Figure 4-53

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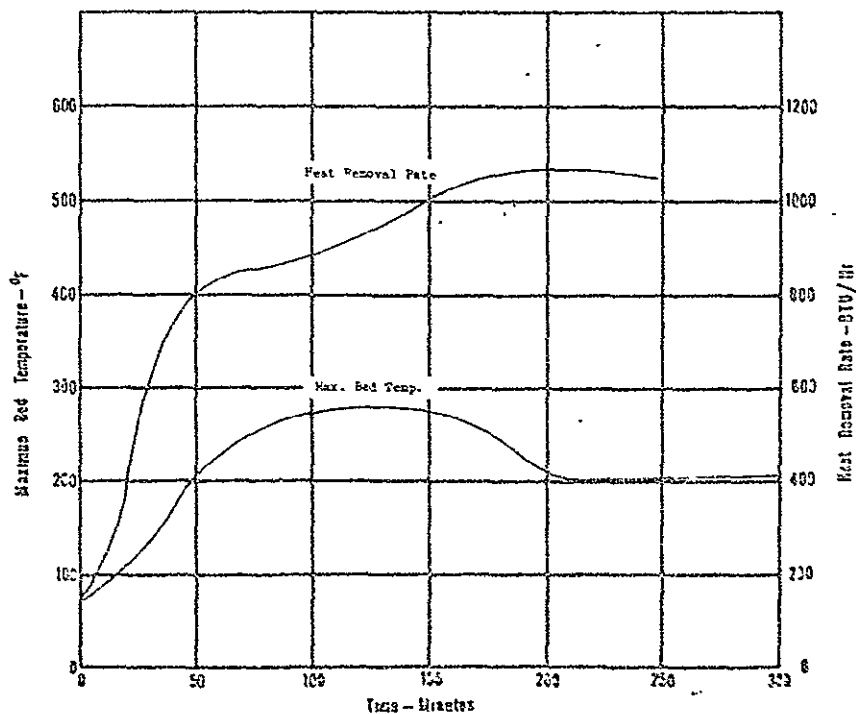
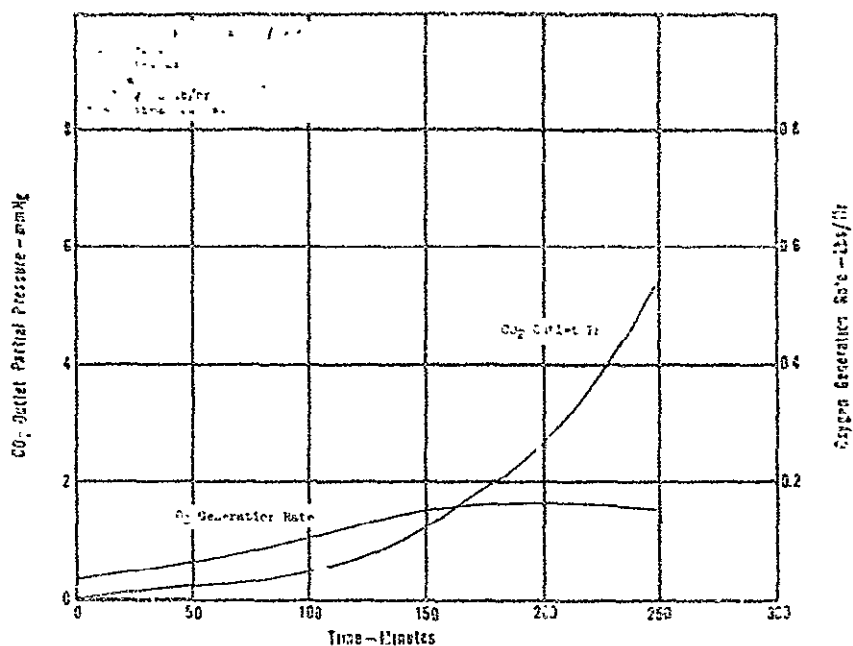


Figure 1-65

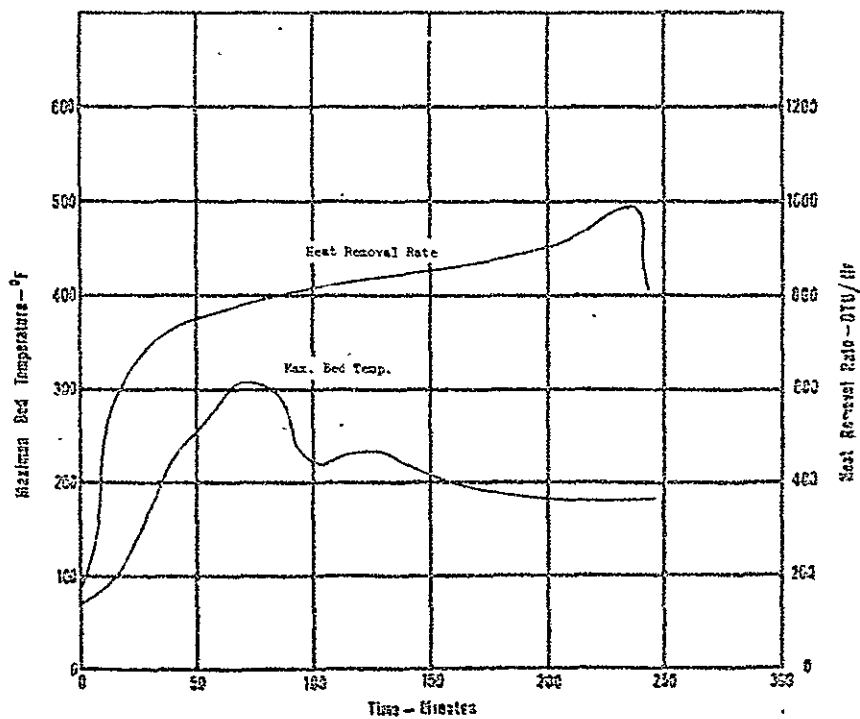
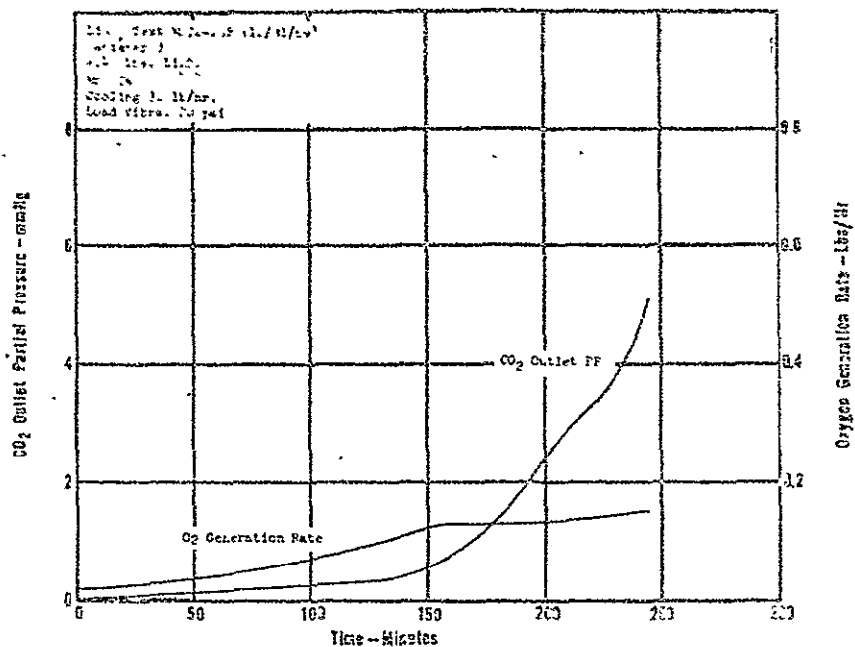


Figure 4-40

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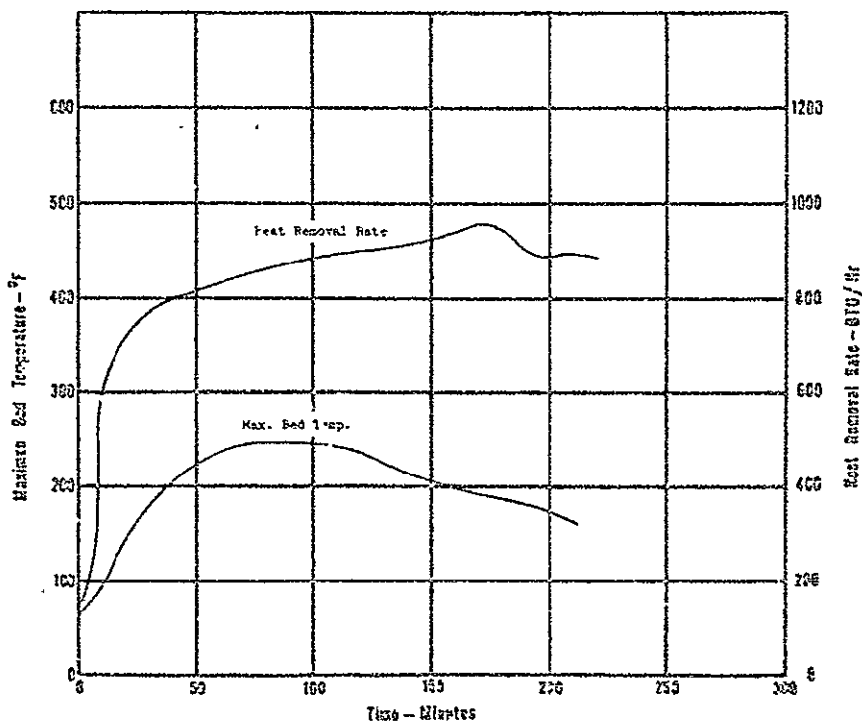
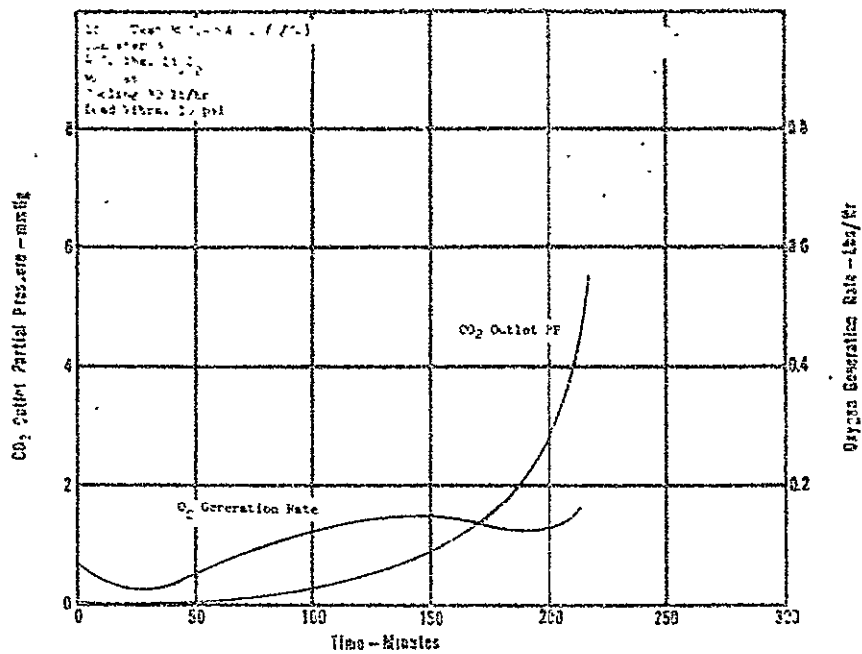
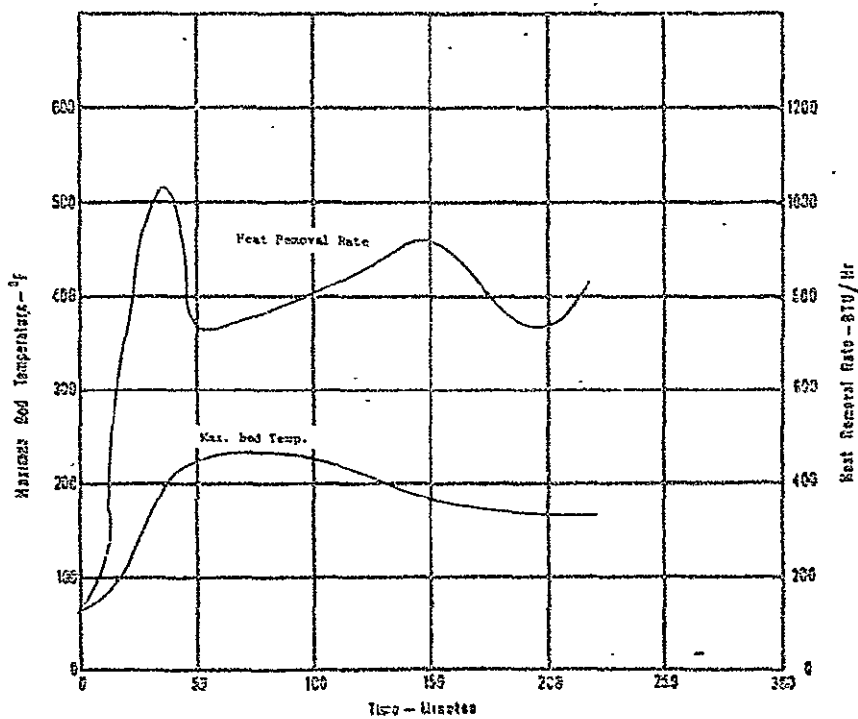
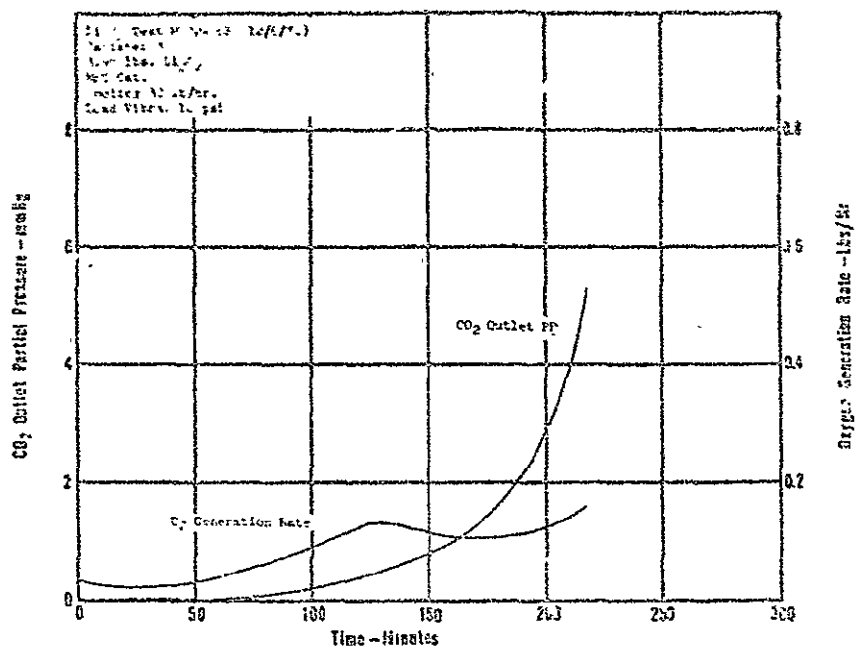
$$v \in \mathcal{P}_n \Rightarrow v \in \mathcal{P}_n$$


Figure 2-41

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Time - Minutes

Figure 4-52

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Sheet 4-63

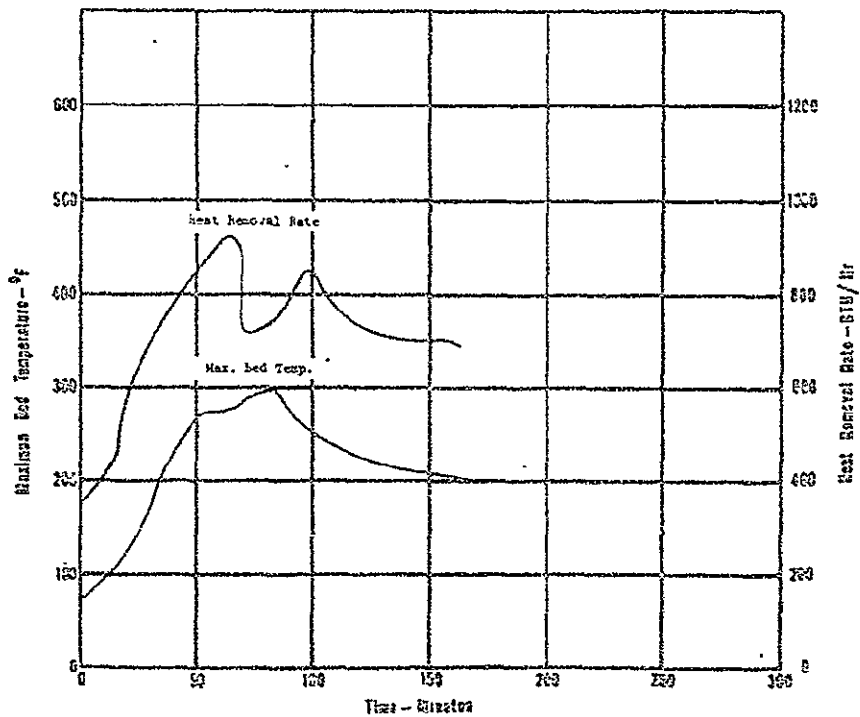
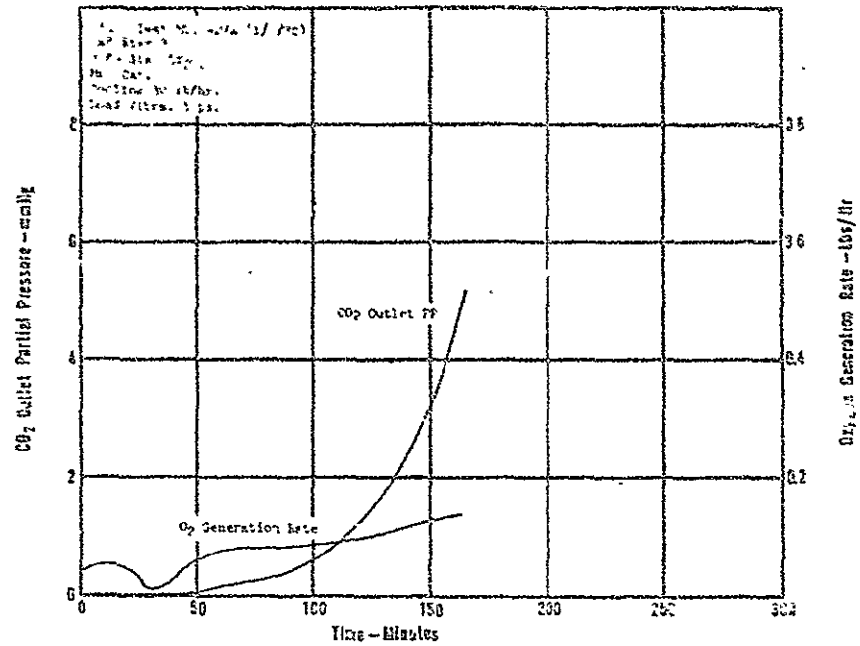
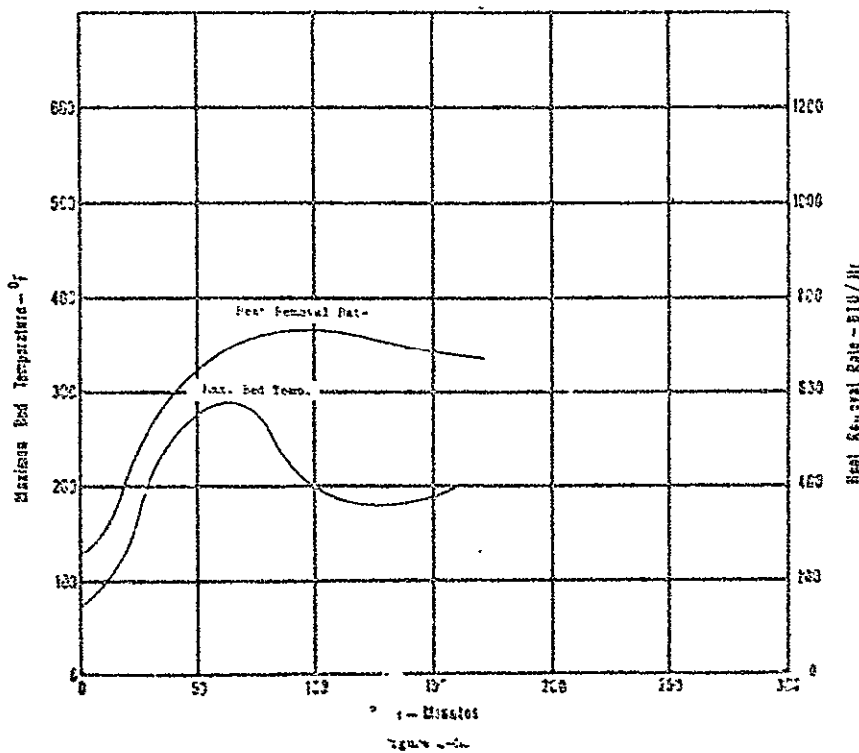
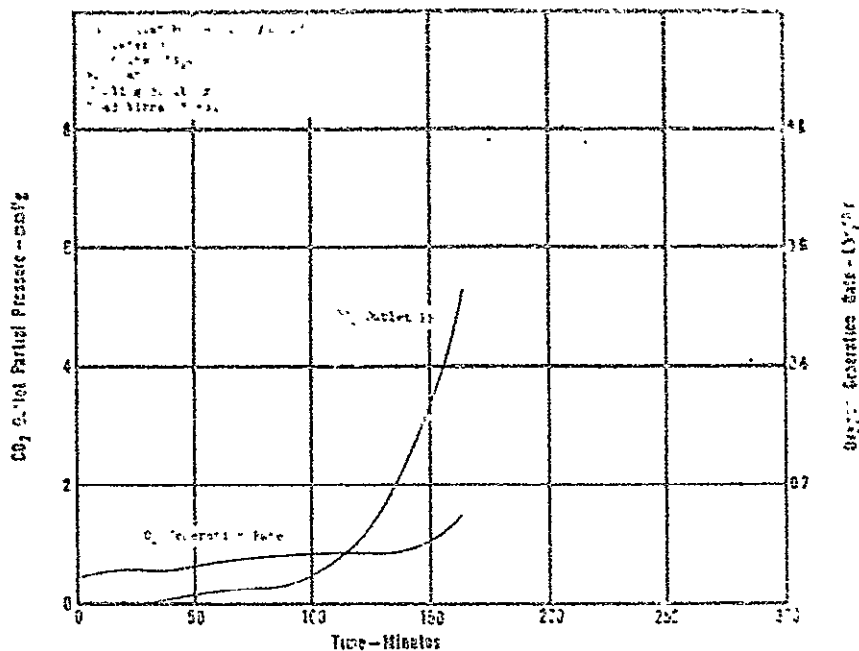


Figure 4-63

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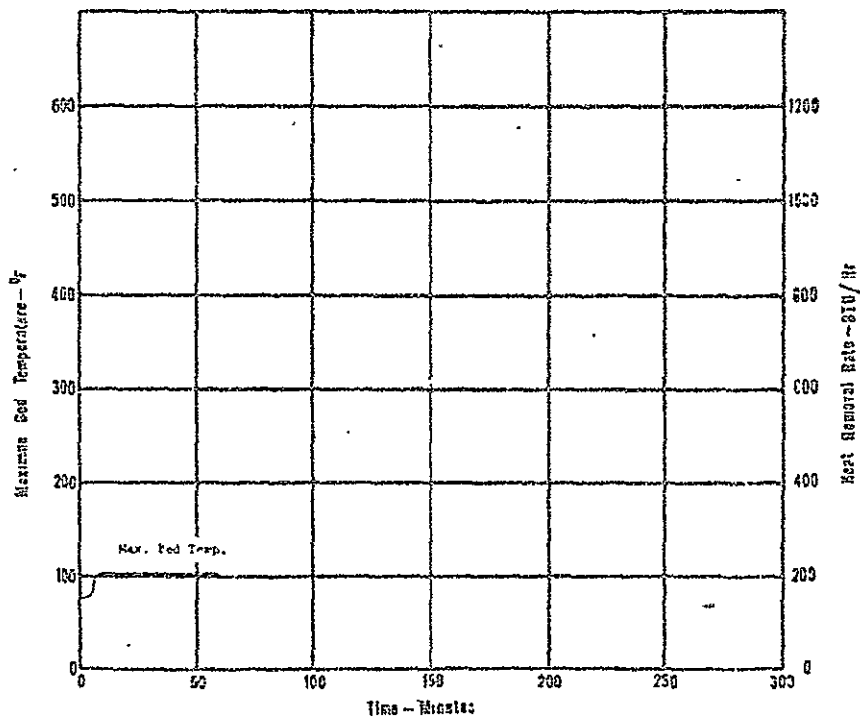
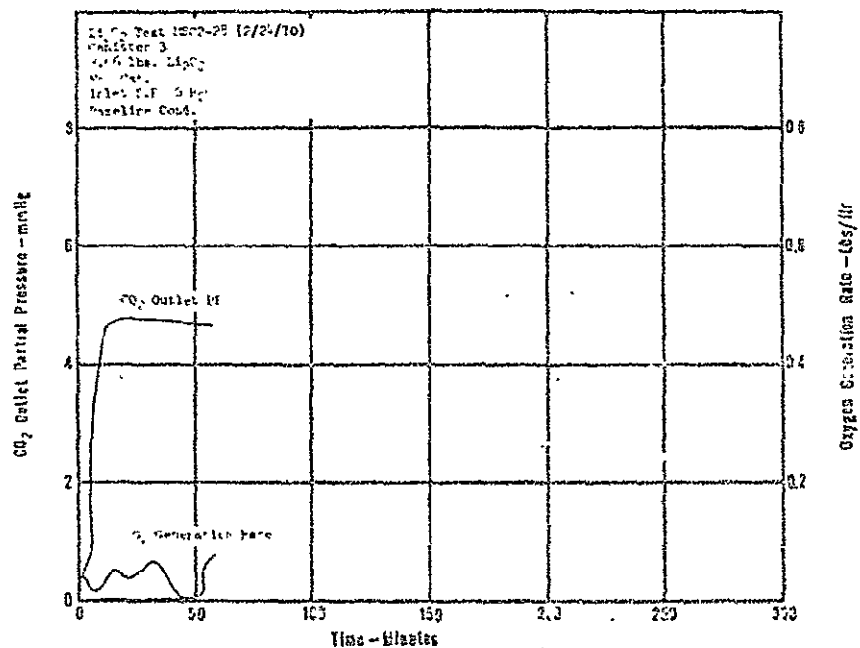


Figure 4-65

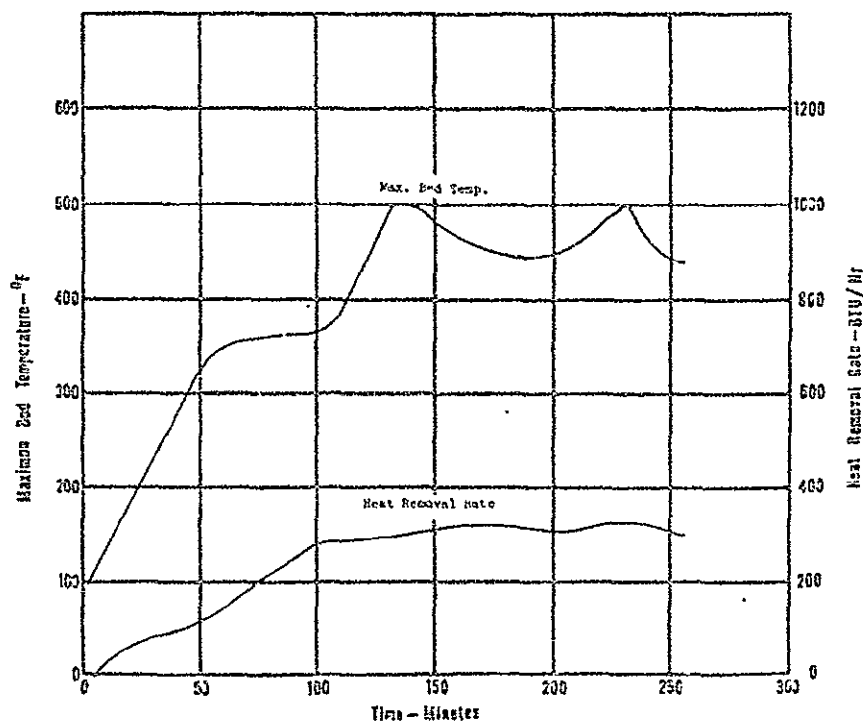
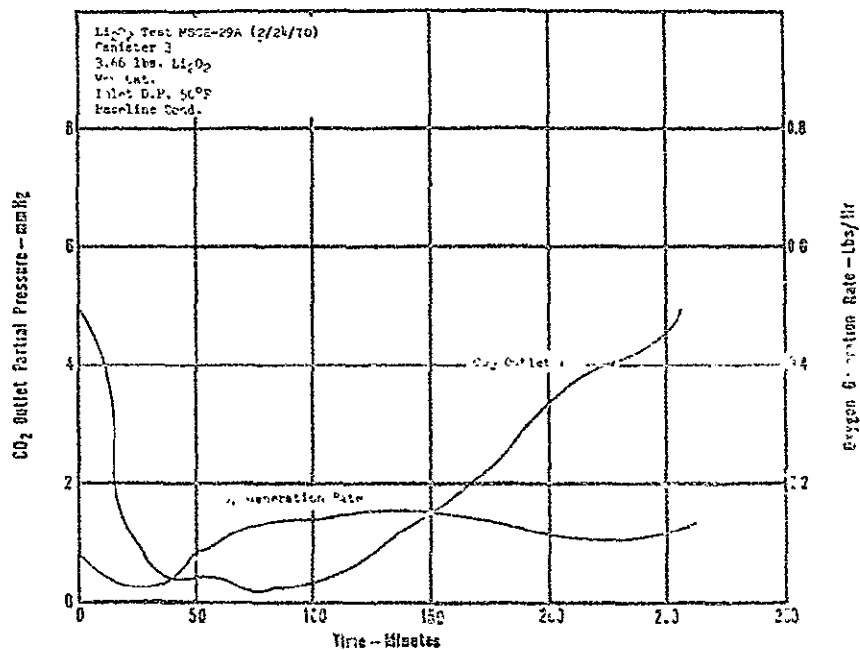


Figure 4-6

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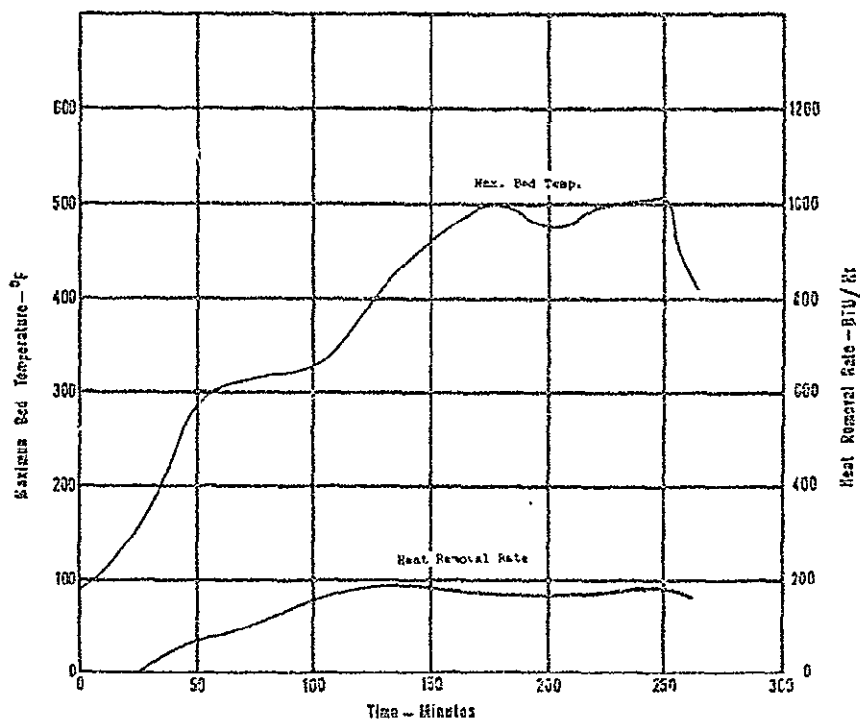
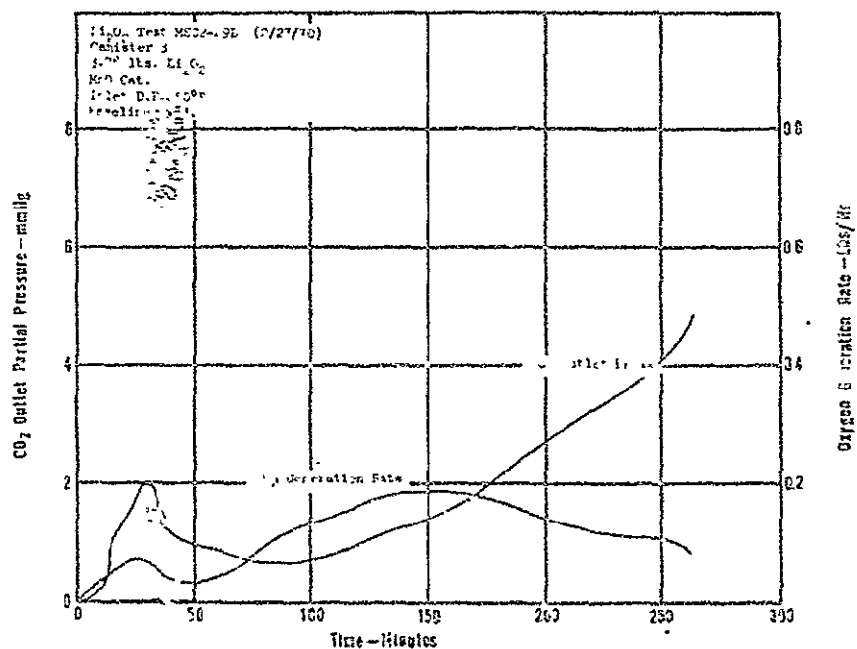


Figure 4-67

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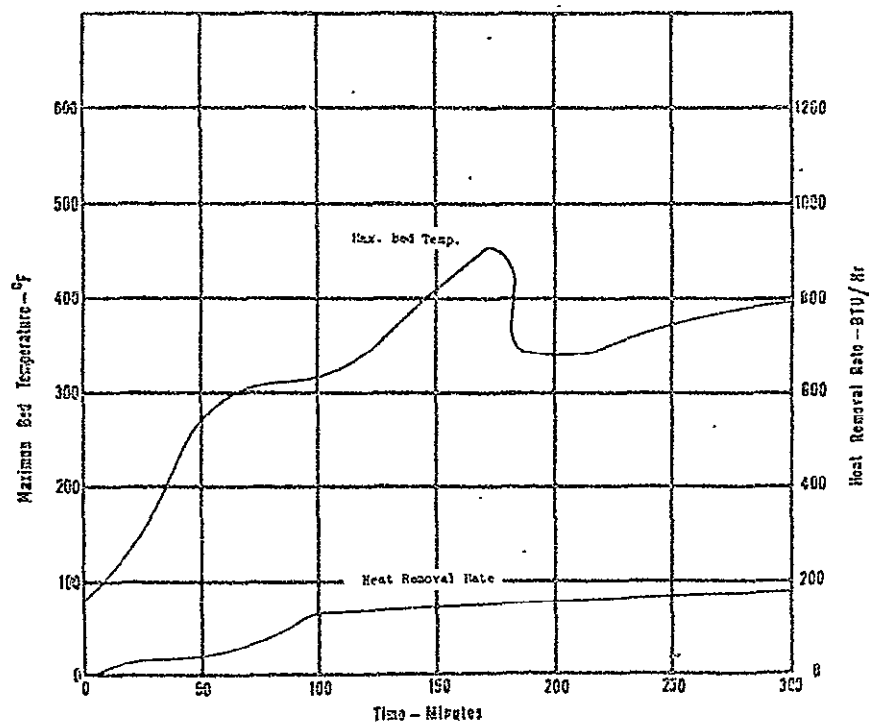
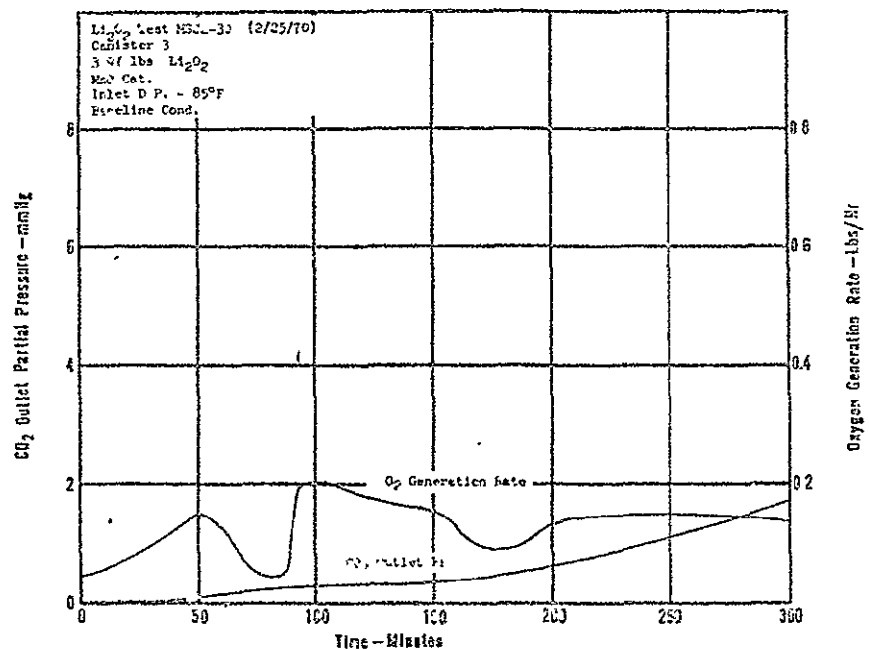


Figure 4-68

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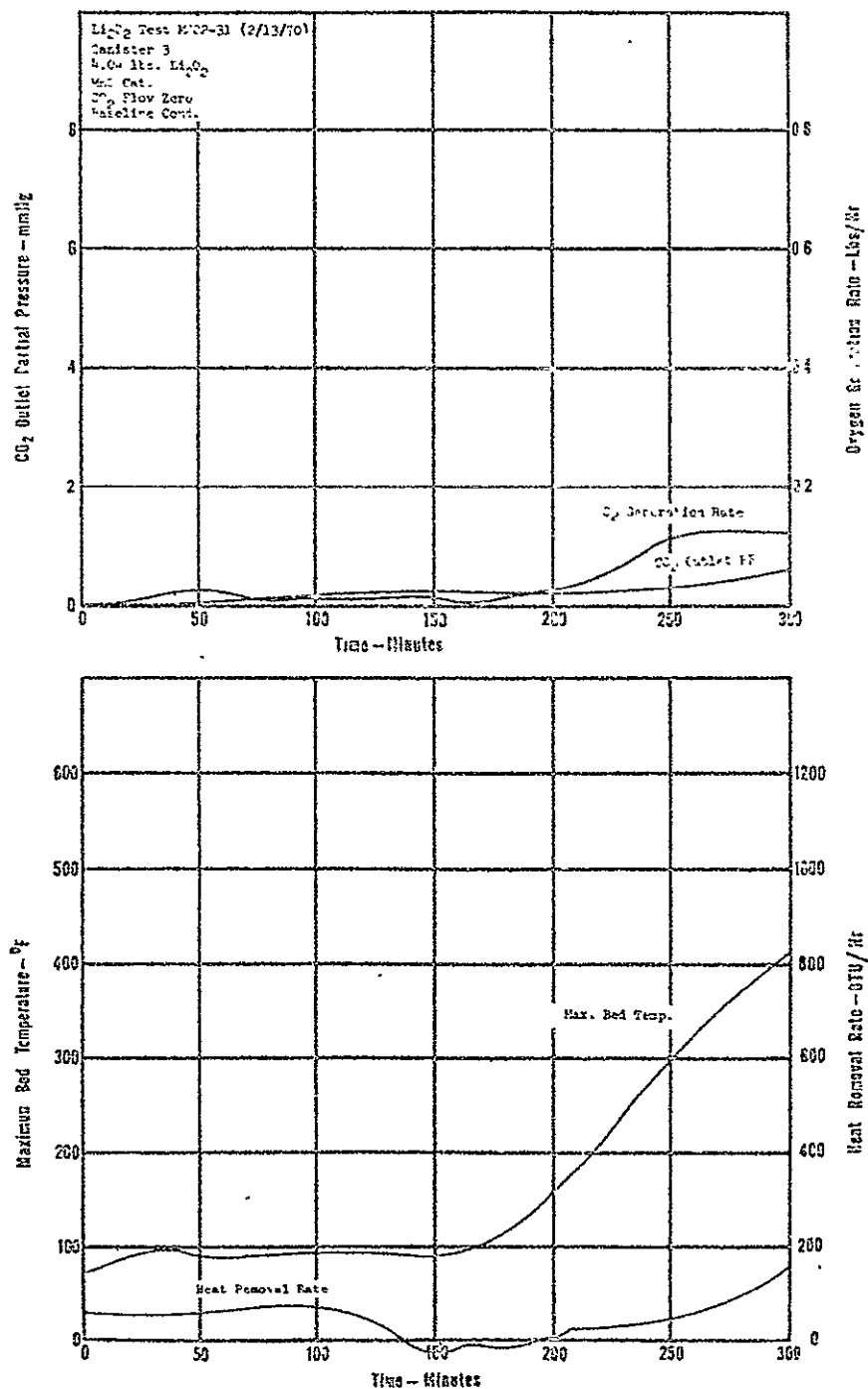


Figure 4-69

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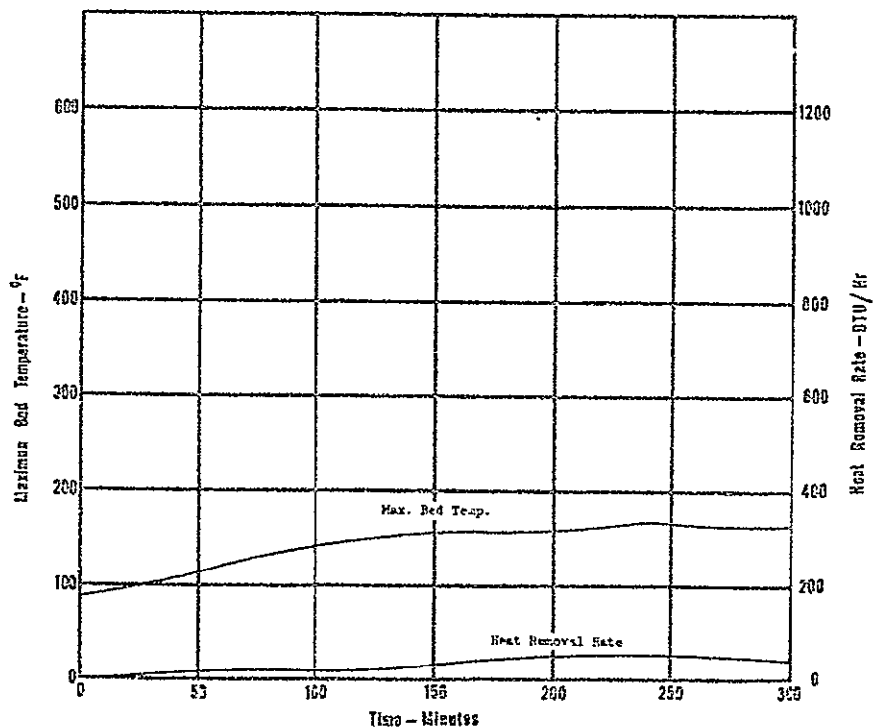
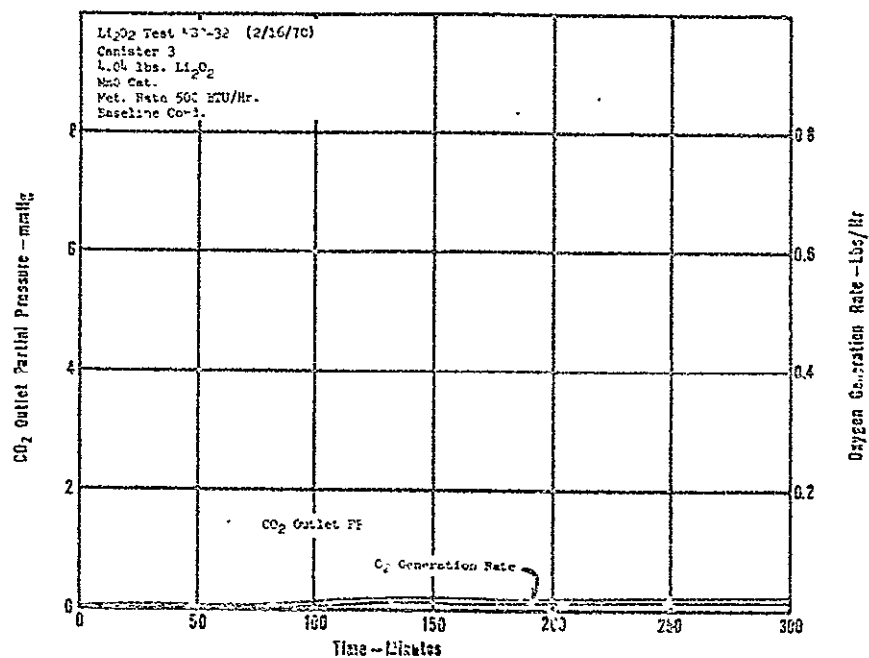


Figure 4-70

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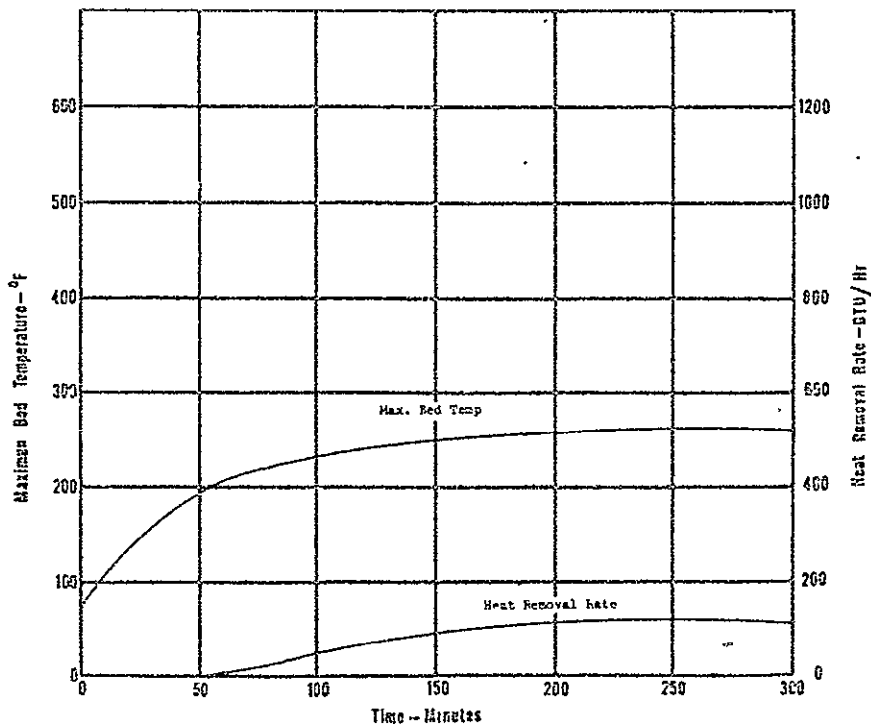
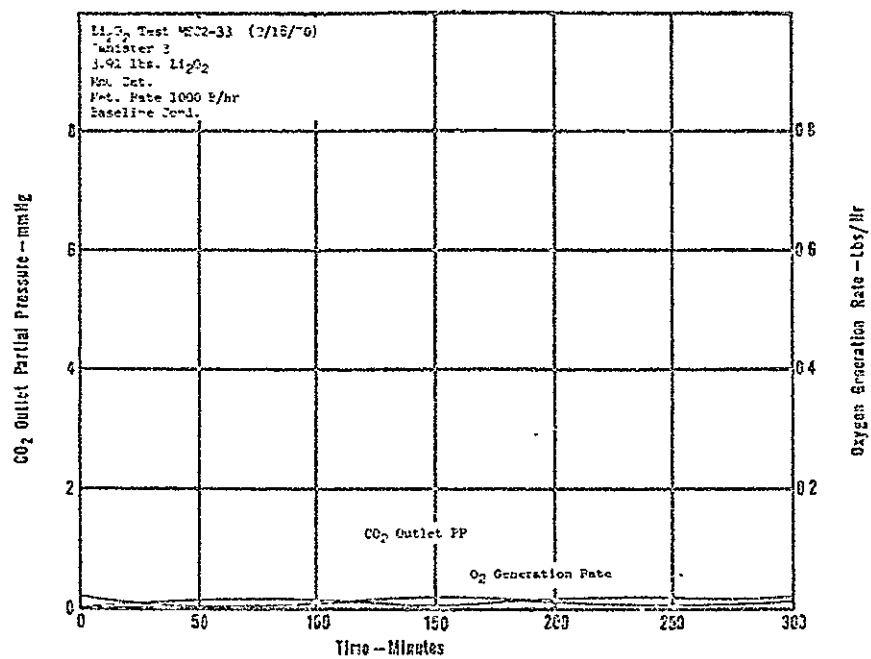


Figure L-71

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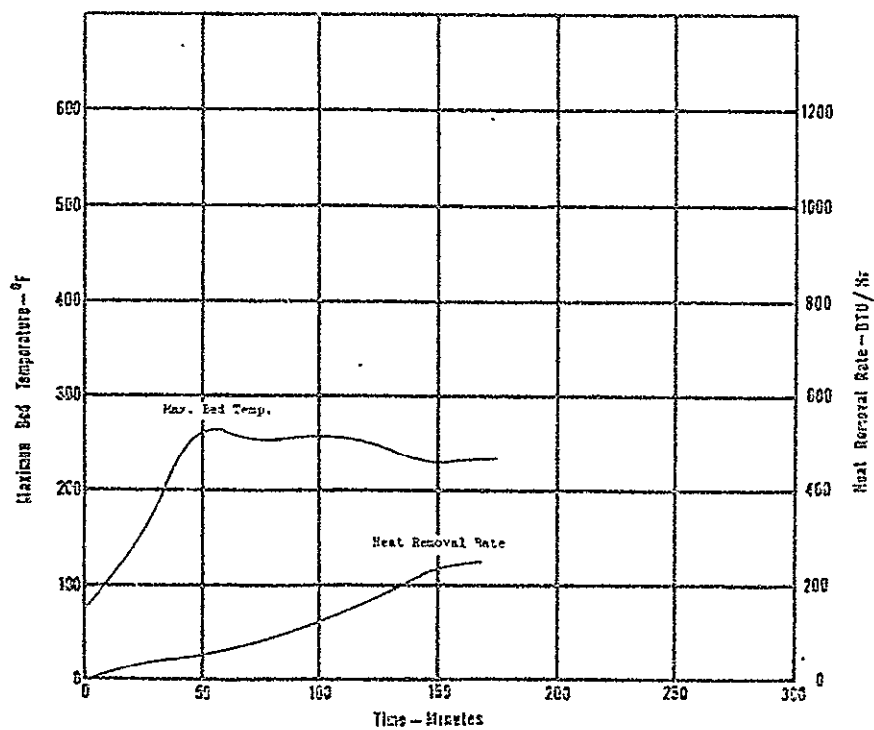
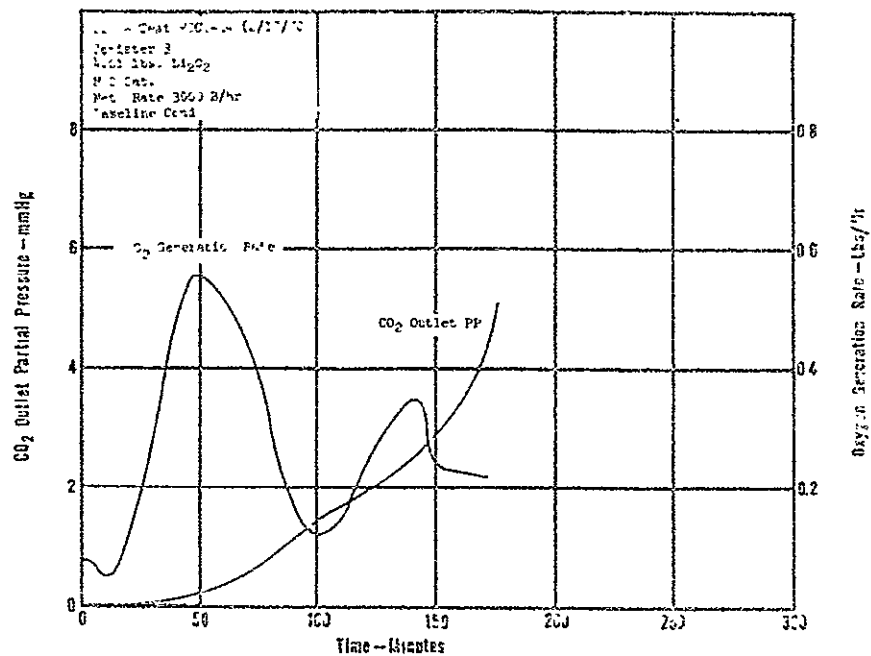


Figure 4-72

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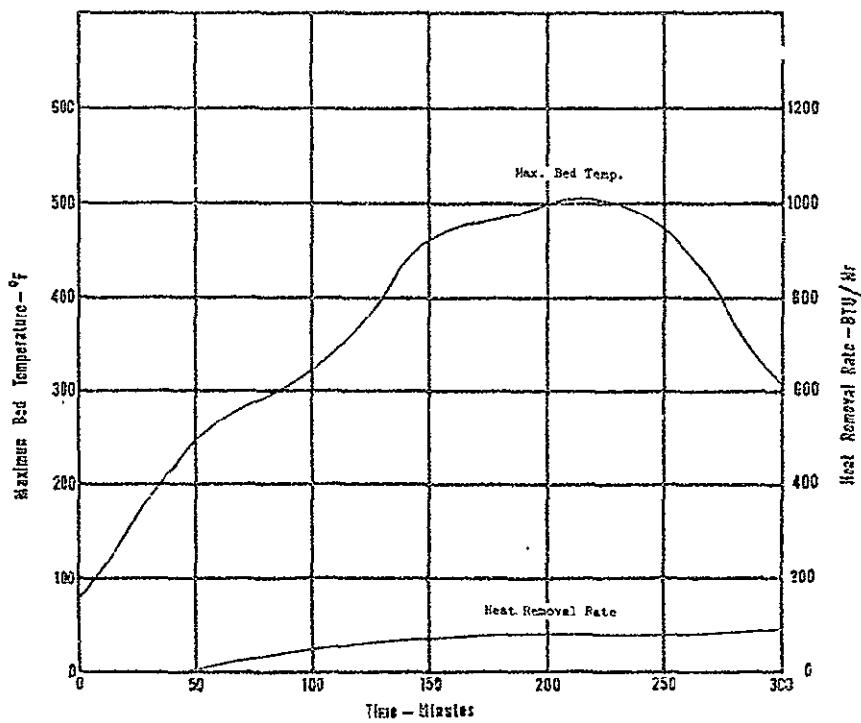
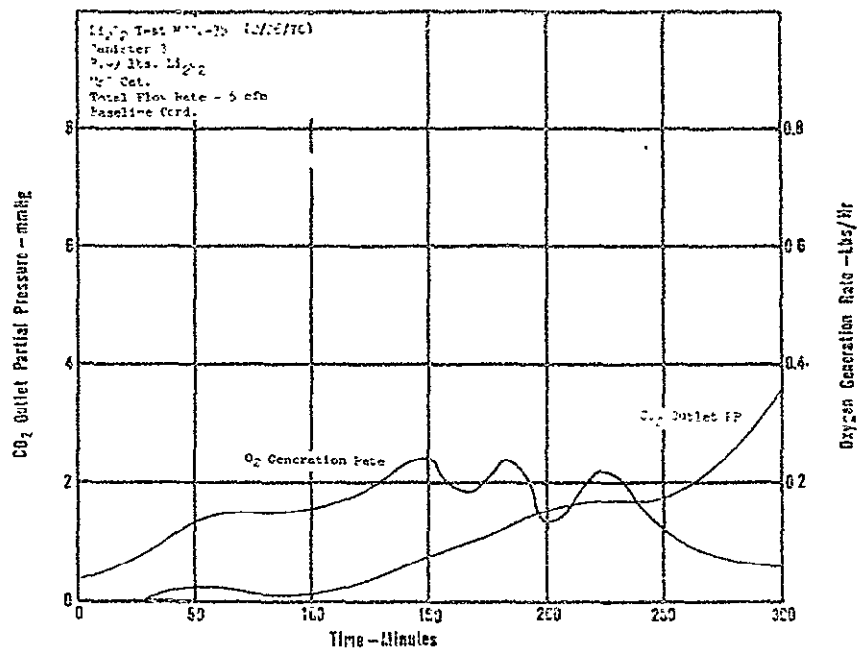


Figure 4-73

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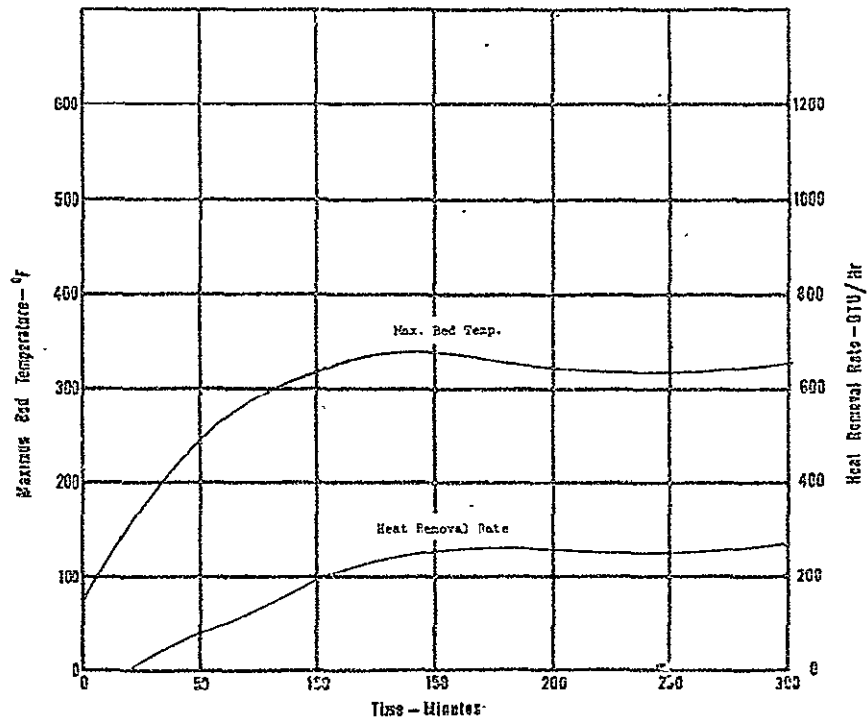
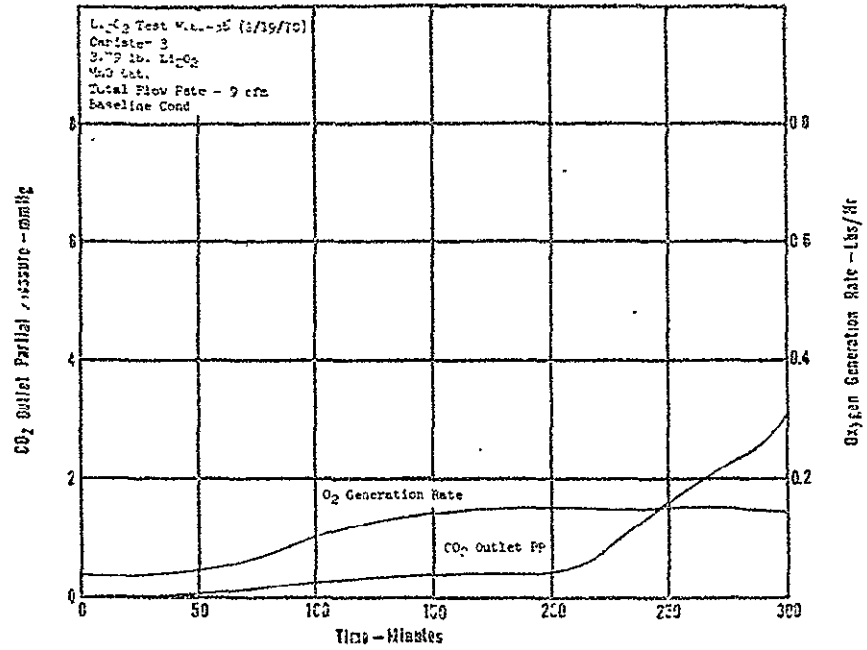


Figure 11-7h

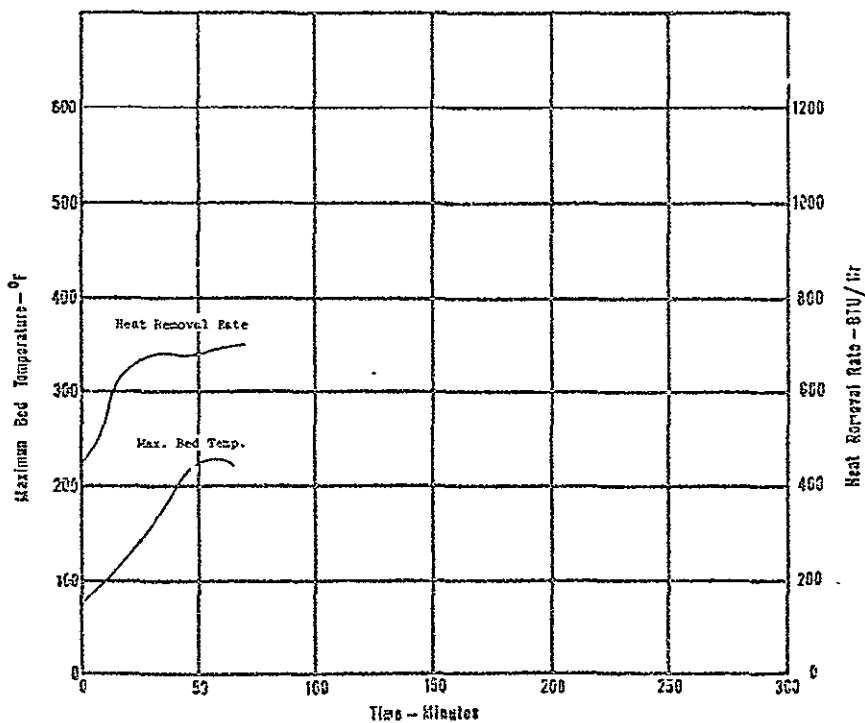
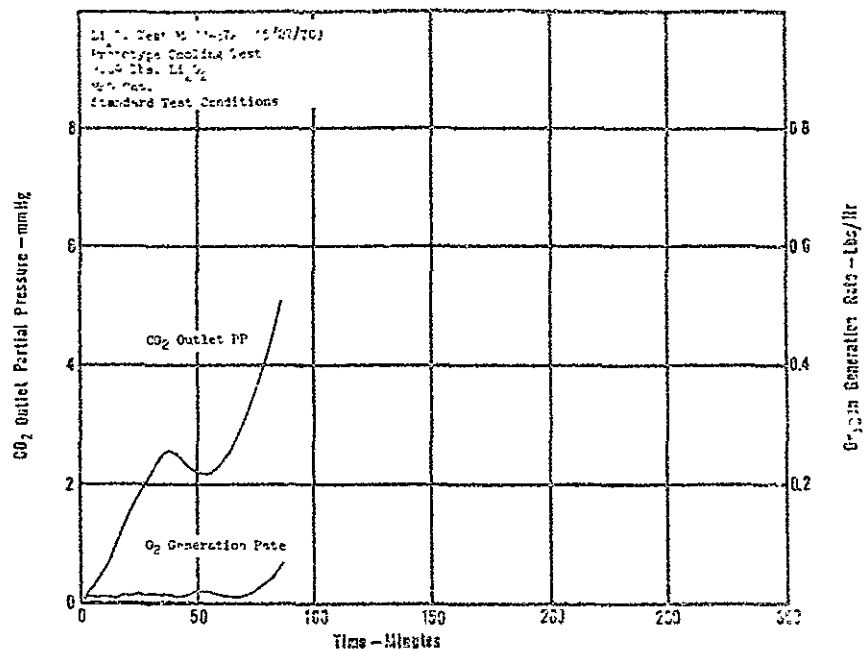


Figure 4-75

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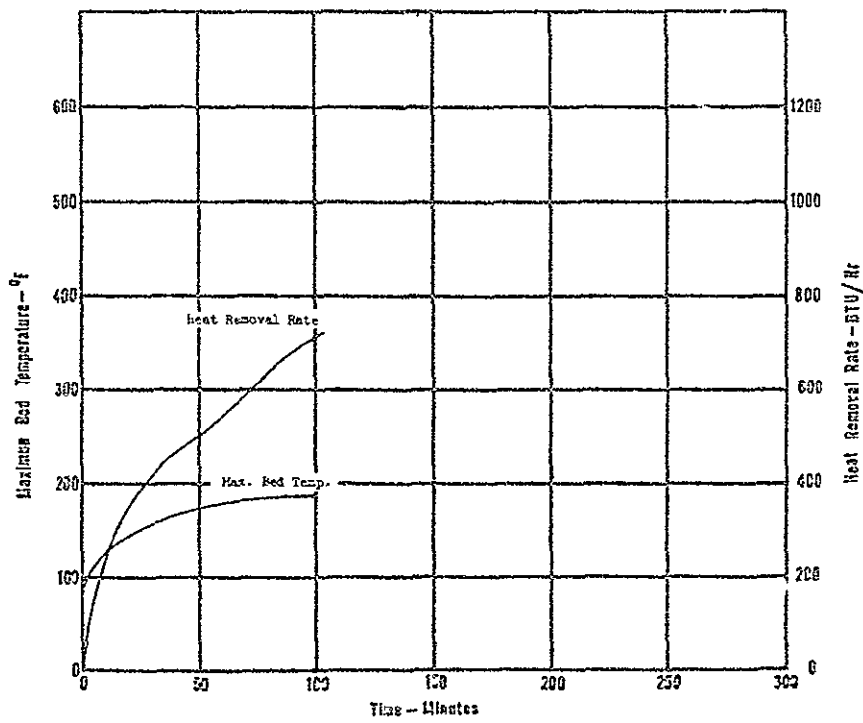
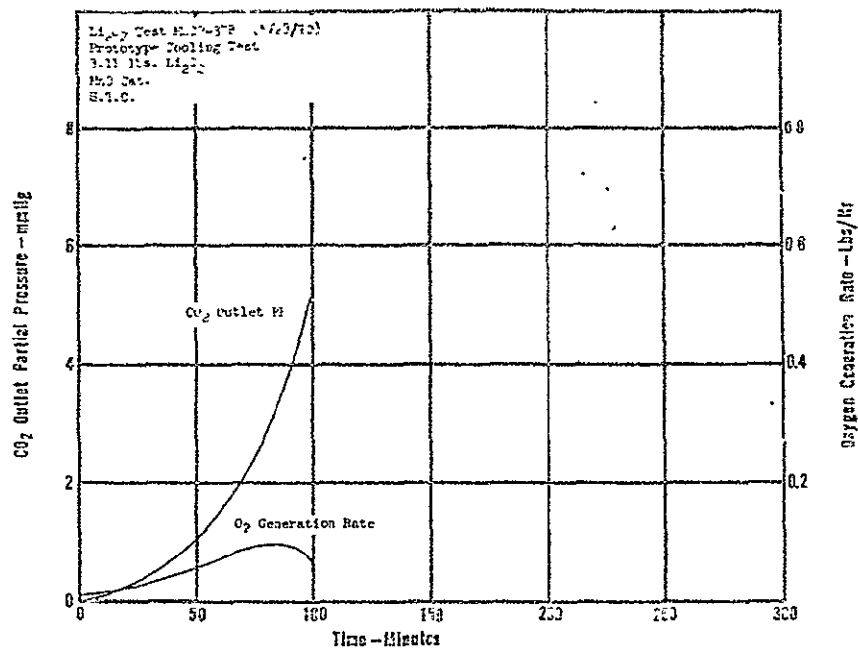


Figure 4-75

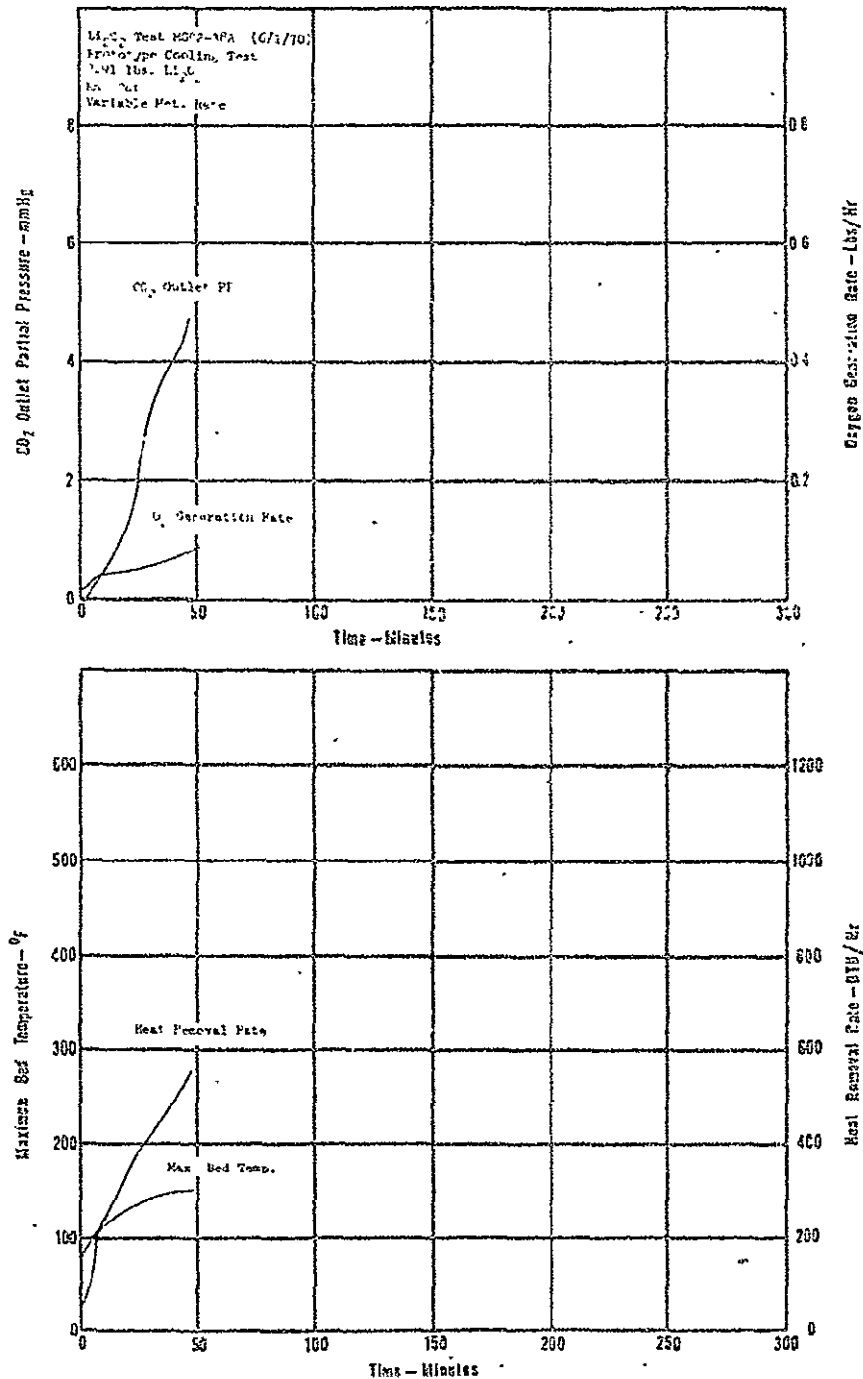


Figure 4-77

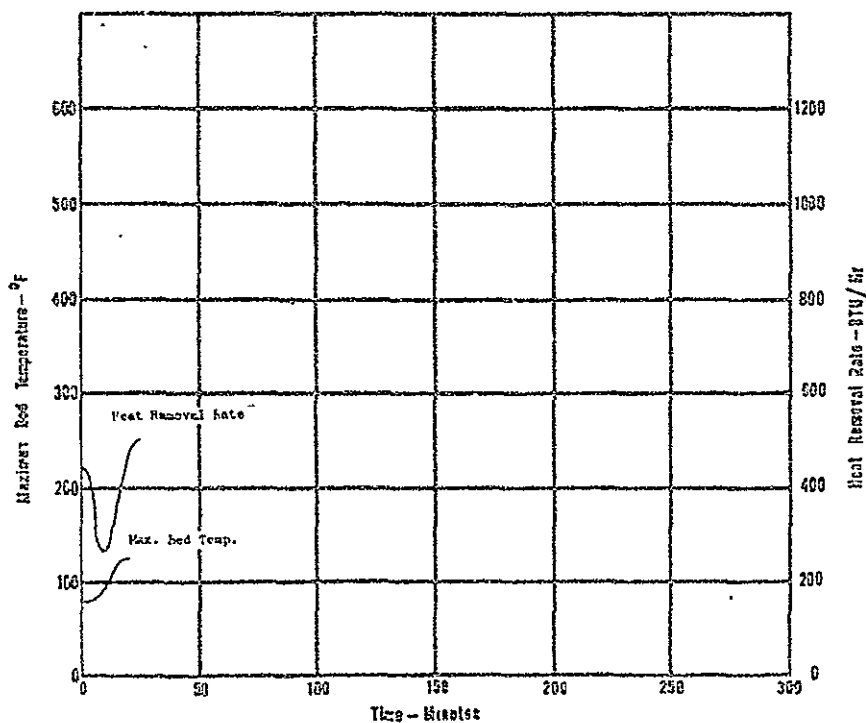
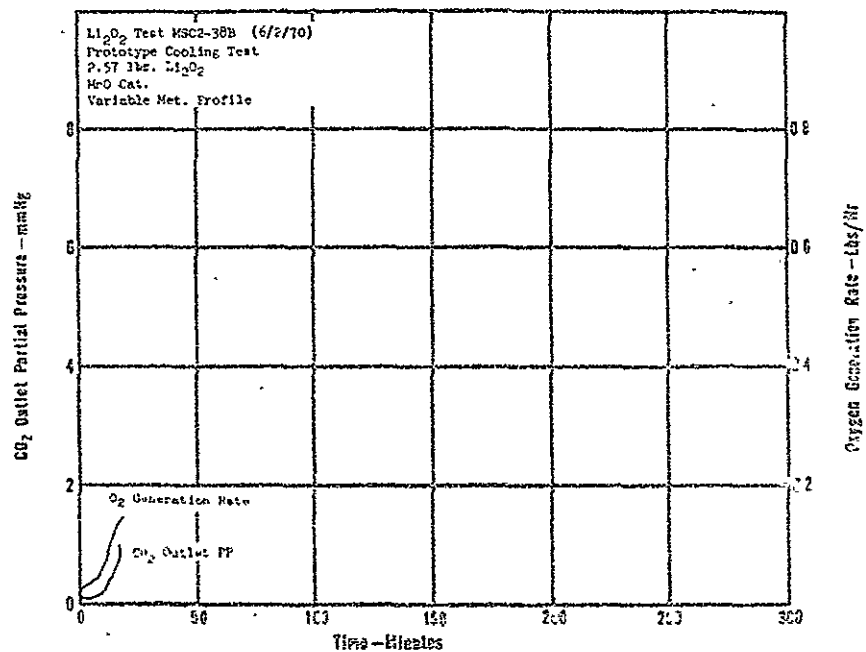


Figure 4-75

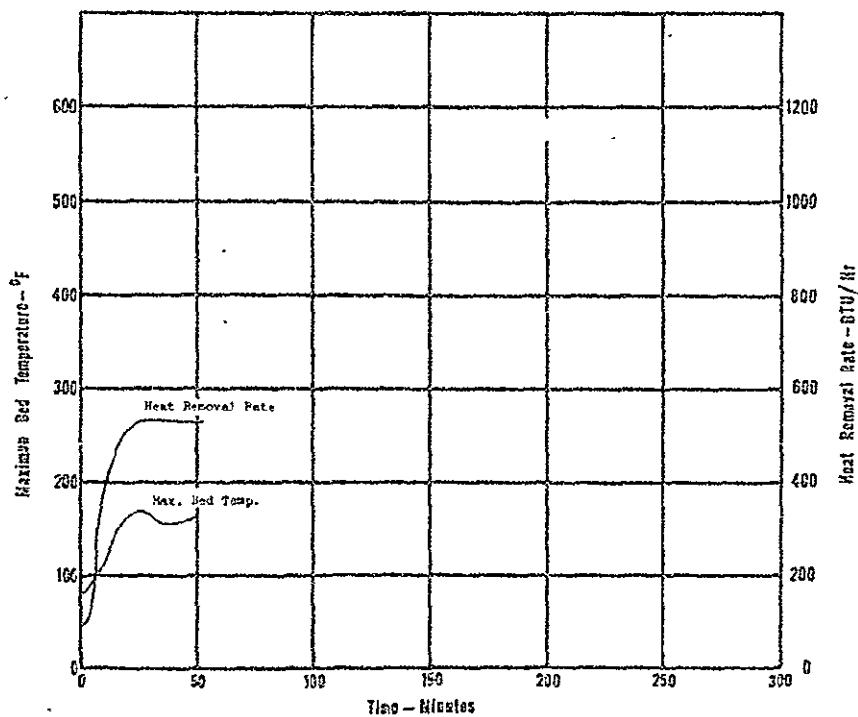
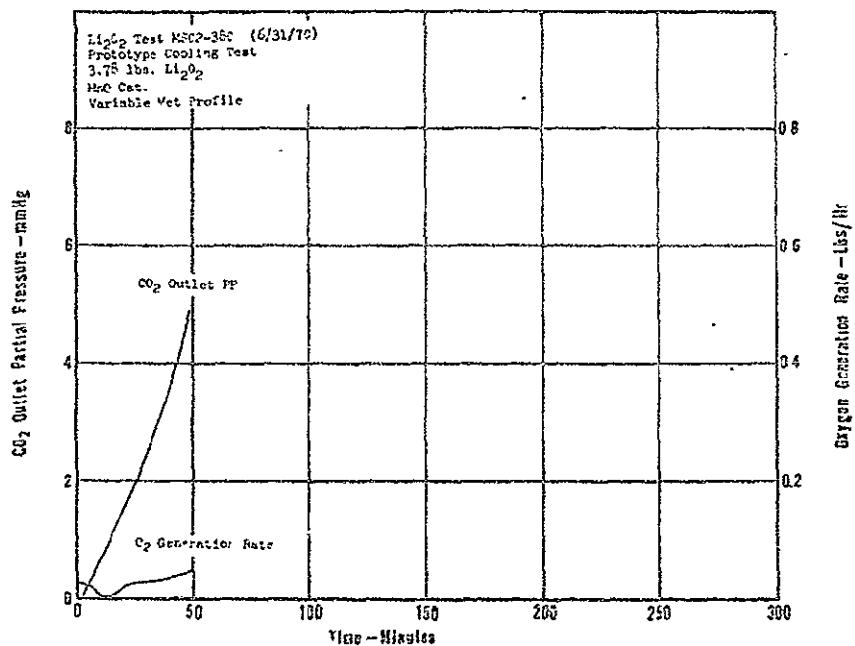


Figure h-79

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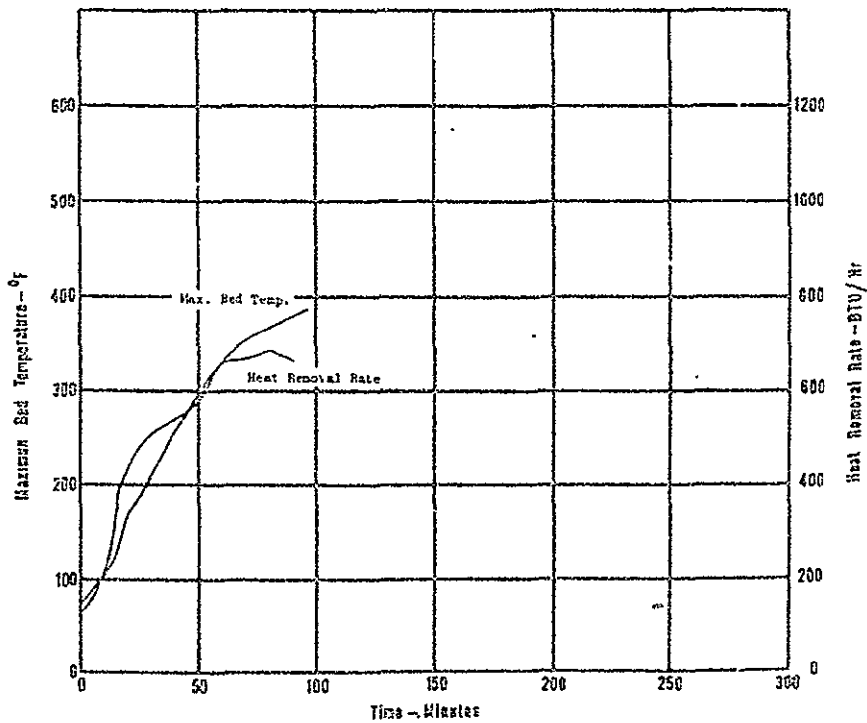
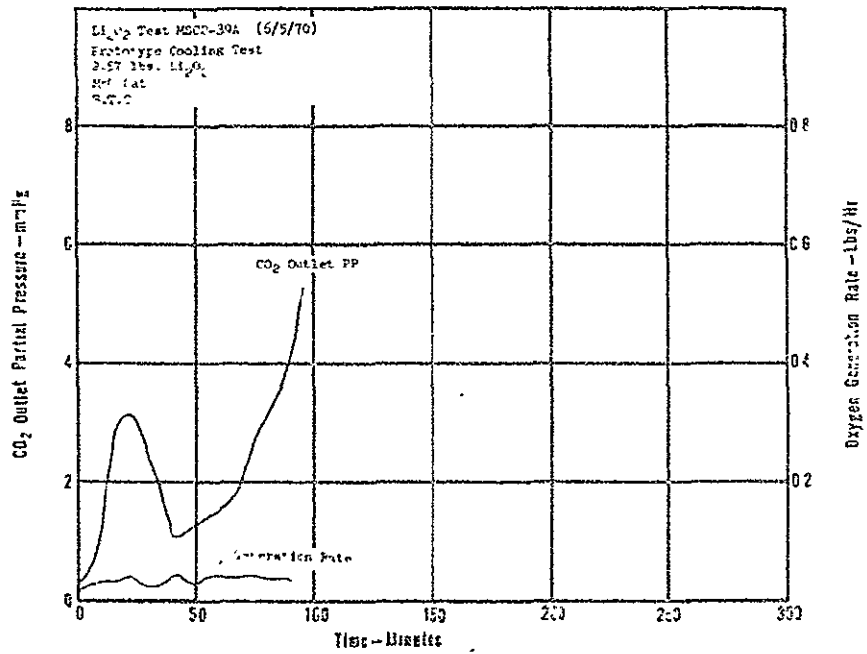


Figure 4-80

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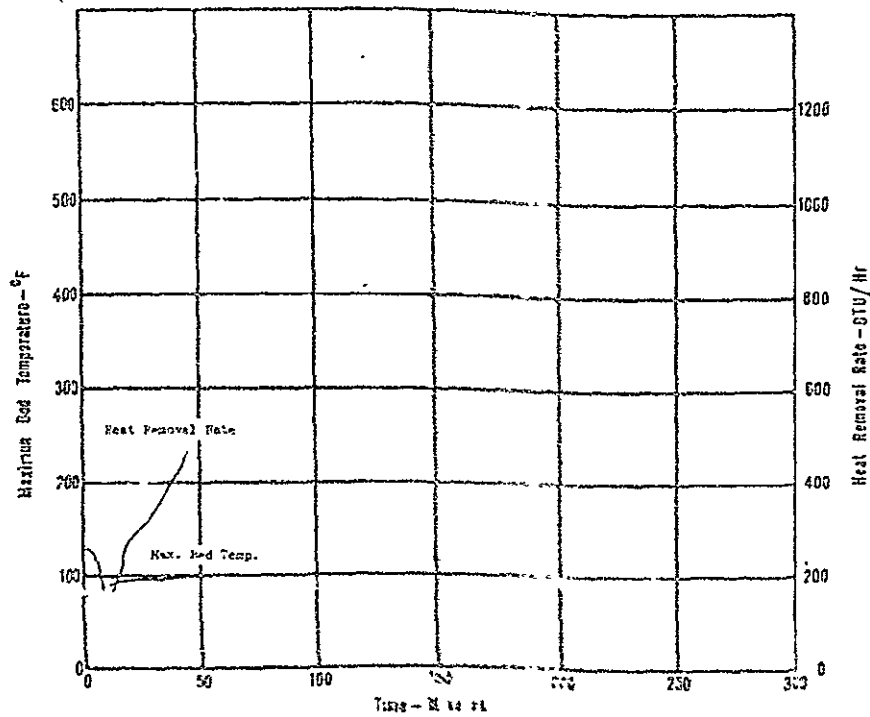
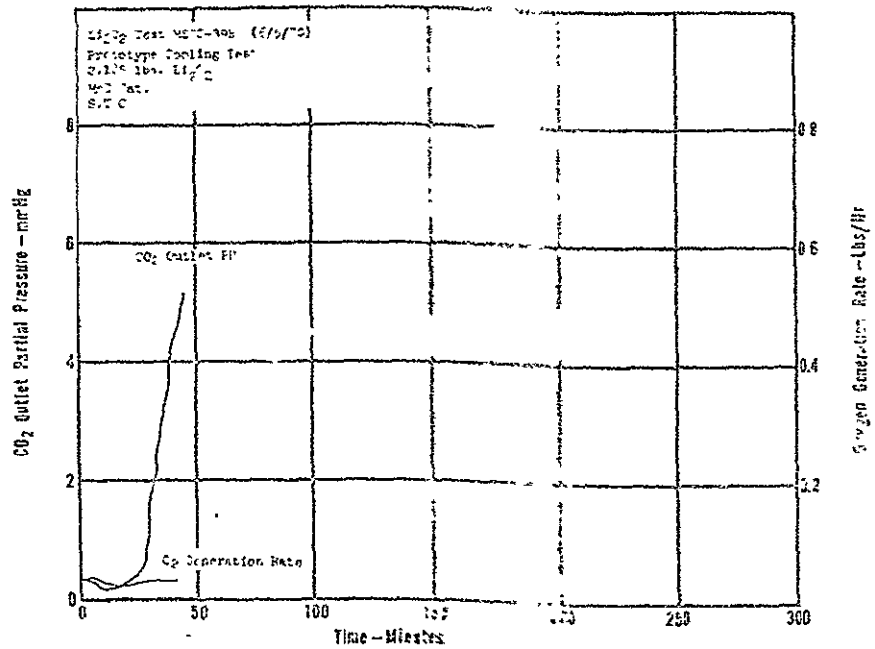


Figure 4-1

SVH32R 5712

TEST DATA SUMMARY

1 O ₂)	Total O ₂ (lb.)	O ₂ Eff. (%)	Total CO ₂ (lb.)	CO ₂ Eff. (%)	Chemical Type	Max. Bed Temp- °F	Evidence Of Thermal Decomposition	Total Test Duration (min.)
	1.971	72.7	1.561	44.28	MnO	647	Yes	276
	1.013	75.0	1.56	42.60	MnO	640	Yes	250
5	0.515	39.0	1.56	43.05	MnO	350	No	230
2	0.916	72.7	1.561	45.10	MnO	630	Yes	245
2	0.402	48.58	1.559	42.63	MnO	185	No	243
1	0.114	30.46	1.539	41.24	MnO	196	No	237
5	0.335	26.25	1.56	44.48	FeSO ₄	260	No	253
4	0.801	63.0	1.30	38.81	FeSO ₄	625	Yes	200
1	0.524	65.66	1.561	45.38	FeSO ₄	270	No	290
2	0.570	72.83	1.561	46.12	FeSO ₄	325	No	280
	0.65	78.30	1.561	52.64	FeSO ₄	230	No	300
	0.545	53.83	1.562	41.41	FeSO ₄	190	No	300
	0.531	62.72	1.561	46.20	MnO ₂	515	No	270
	0.588	65.03	1.559	46.43	MnO ₂	270	No	260
	0.211	26.93	1.106	31.42	MnO ₂	214	No	170
	0.373	42.91	1.559	45.61	MnO ₂	196	No	240
	0.543	67.49	1.559	43.86	TiO ₂	325	No	240
	0.466	34.85	1.561	42.53	MnO + Ag/Cu	190	No	277
	0.356	27.35	1.561	43.72	No Cat.	210	No	244
	0.417	34.71	1.562	47.36	AgNO ₃	200	No	303
	0.544	40.87	1.561	42.75	CaO ³	325	No	316
	0.683	52.39	1.561	43.61	MnO	475	No	330
	0.701	50.19	1.561	40.72	MnO	481	No	330
	0.634	48.66	1.561	43.63	FeSO ₄	478	No	275
2	0.887	69.11	1.561	44.31	FeSO ₄	667	Yes	245
	0.476	37.17	0.588	16.72	MnO	730	Yes	95
	0.516	40.61	1.531	43.89	MnO	355	No	296
	0.587	42.88	1.596	42.44	MnO	364	No	315
	0.814	55.42	1.022	27.67	MnO	762	Yes	158
	0.737	54.67	0.996	27.16	MnO	733	Yes	154
	0.369	26.50	1.559	44.51	MnO	280	No	258
	0.340	24.47	1.561	44.66	MnO	305	No	250
	0.706	45.11	1.556	38.75	MnO	520	No	260
	0.451	28.80	1.559	39.63	MnO	376	No	289
	0.295	20.34	1.561	42.74	MnO	275	No	266
	0.391	24.54	1.561	33.94	MnO	290	No	298

Test Data Summary
TABLE 4-1

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SWKSER 5712

ATA SUMMARY (Continued)

Sample No.	Total O ₂ (Lb.)	O ₂ Eff. (%)	Total CO ₂ (Lb.)	CO ₂ Eff. (%)	Chemical Type	Max. Bed Temp. °F	Evidence Of Thermal Decomposition	Total Test Duration (Min.)
0.591	0.591	41.51	1.559	43.27	MnO	653	No	270
0.621	0.621	47.55	1.556	42.14	MnO	703	Yes	240
0.549	0.549	35.86	1.559	40.47	MnO	760	No	268
0.598	0.598	45.42	1.562	47.15	MnO	170	No	323
0.341	0.341	25.42	1.561	46.32	MnO	163	No	287
0.544	0.633	43.22	1.447	39.27	MnO	615	Yes	223
0.531	0.531	35.38	1.560	41.31	MnO	700	No	306
0.496	0.496	35.78	1.560	44.75	MnO	330	No	254
0.296	0.296	20.05	1.560	42.05	MnO	175	No	255
0.435	0.435	25.18	1.562	35.91	MnO	210	No	374
0.401	0.401	23.76	1.562	36.84	MnO	210	No	380
0.453	0.453	24.31	1.513	45.57	MnO	235	No	230
0.488	0.488	36.94	1.596	48.06	MnO	249	No	260
0.453	0.453	34.35	1.596	48.07	MnO	226	No	285
0.331	0.331	24.45	1.231	36.17	MnO	410	No	287
0.322	0.322	24.15	1.230	36.67	MnO	332	No	250
0.254	0.254	19.40	1.065	32.41	MnO	290	No	208
0.271	0.271	20.53	1.073	32.31	MnO	290	No	210
0.207	0.207	18.25	0.964	33.75	MnO	255	No	188
0.181	0.181	15.95	0.845	29.70	MnO	240	No	165
0.356	0.356	27.06	1.228	37.08	MnO	295	No	250
0.363	0.363	28.93	1.229	38.97	MnO	250	No	280
0.425	0.425	27.99	1.558	40.80	MnO	277	No	250
0.412	0.412	28.35	1.559	42.68	MnO	312	No	240
0.385	0.385	29.06	1.475	44.20	MnO	245	No	227
0.371	0.371	28.41	1.481	45.06	MnO	250	No	228
0.255	0.255	18.72	1.059	35.06	MnO	300	No	163
0.208	0.208	17.70	1.066	36.08	MnO	290	No	164
0.033	0.033	2.73	0.307	10.11	MnO	100	No	60
0.494	0.494	40.91	1.226	40.37	MnO	500	No	257
0.514	0.514	41.22	1.226	39.06	MnO	505	No	270
0.483	0.483	36.83	1.231	37.35	MnO	470	No	415
0.066	0.066	4.91	0.333	9.93	MnO	512	No	360
0.149	0.149	11.20	0.365	11.35	MnO	165	No	740
0.247	0.247	18.17	0.767	23.63	MnO	270	No	680
0.415	0.833	62.92	1.699	51.04	MnO	528	Yes	178
0.624	0.624	47.38	1.229	37.10	MnO	505	No	332
0.464	0.464	37.10	1.230	39.11	MnO	350	No	335
0.059	0.059	5.82	0.520	20.28	MnO	225	No	80
0.119	0.119	11.56	0.518	20.07	MnO	182	No	100
0.028	0.028	2.88	0.290	12.02	MnO	150	No	45
0.028	0.028	3.31	0.129	6.06	MnO	115	No	20
0.034	0.034	2.76	0.322	10.28	MnO	175	No	50
0.144	0.144	16.99	0.579	27.16	MnO	390	No	90
0.025	0.025	3.42	0.258	14.24	MnO	110	No	40

Test Data Summary (Con'td)
TABLE 4-1

Hamilton Standard ^U_A

Test Number	Canister No.	Time To .5-1.0-1.0 (Min.)	Chemical Wt. (Lb.)	Ave. Useable O ₂ (Lb./Hr.)	Time O ₂ < 0.36 lb/hr. (Min.)	Total Usable O ₂ (Lb.)	
MSC2-1A	3	196-211-265	4.05	.2	56	0.52	
MSC2-1B	3	174-190-222	4.10	.23	60	0.52	
MSC2-1C	3	155-180-255	4.00	.128	0	1.515	
MSC2-1D	3	186-200-235	3.82	.192	60	0.709	
MSC2-2A	3	145-182-227	4.12	.167	0	0.402	
MSC2-2B	3	137-160-217	4.12	.105	0	0.414	
MSC2-3A	3	146-169-235	3.87	.0837	0	0.335	
MSC2-3B	3	132-149-189	3.85	.175	41	0.511	
MSC2-3C	3	170-215-277	3.95	.13	0	0.511	
MSC2-3D	3	180-210-265	3.92	.14	0	0.570	
MSC2-4A	3	195-220-284	3.38	.165	0	0.65	
MSC2-4B	3	200-238-295	4.02	.133	0	0.545	
MSC2-5A	3	170-200-265	3.80	.135	0	0.531	
MSC2-5B	3	133-162-245	3.81	.15	0	0.513	
MSC2-6A	3	138-151-166	3.82	.08	0	0.211	
MSC2-6B	3	140-167-232	3.78	.095	0	0.373	
MSC2-7A	3	138-170-250	4.06	.124	0	0.543	
MSC2-7B	3	170-189-265	4.05	.12	0	0.466	
MSC2-7C	3	165-185-235	3.94	.089	0	0.355	
MSC2-7D	3	180-240-295	3.64	.104	0	0.417	
MSC2-7E	3	151-205-298	4.03	.136	0	0.544	
MSC2-8A	3	185-223-314	3.95	.17	0	0.683	
MSC2-8B	3	165-211-320	4.23	.175	0	0.701	
MSC2-9A	3	174-202-263	3.45	.16	0	0.634	
MSC2-9B	3	170-185-227	3.89	.178	69	0.712	
MSC2-10A	3	45- 70- 91	3.88	.192	30	0.303	
MSC2-10B	3	98-181-293	3.85	.125	0	0.516	
MSC2-10C	3	180-182-303	4.15	.146	0	0.587	
MSC2-11A	1	53- 59-135	4.45	.192	5.7	0.519	
MSC2-11B	1	58- 68-138	4.42	.159	52	0.407	
MSC2-12A	2	135-165-235	4.22	.092	0	0.369	
MSC2-12B	2	180-205-235	4.21	.085	0	0.540	
MSC2-13A	1	140-160-240	4.64	.177	0	0.706	
MSC2-13B	1	135-160-255	4.74	.113	0	0.451	
MSC2-14A	2	145-185-245	4.40	.074	0	0.295	
MSC2-14B	2	175-205-275	4.83	.098	0	0.391	

4.1 Continued

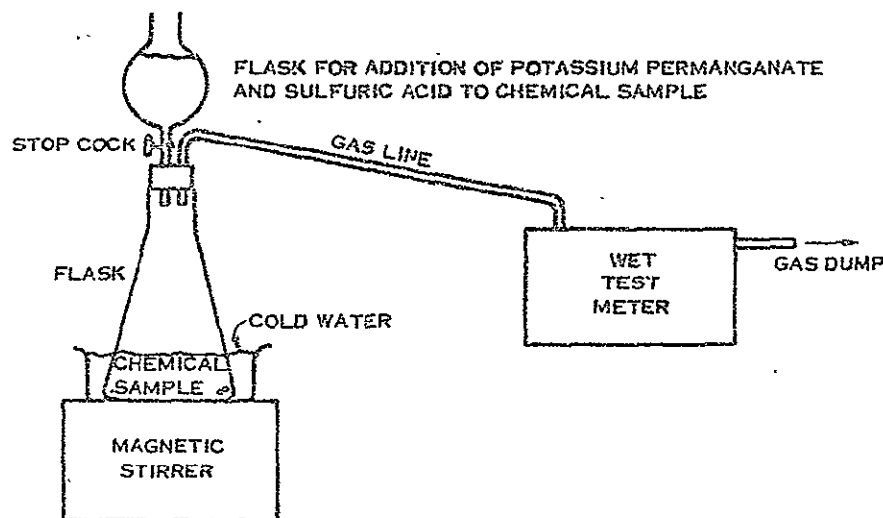
(14) Evidence of the occurrence of thermal decomposition.

(15) Total test duration (minutes).

4.2 Chemical Analysis Data

In order to confirm the validity of the measured test results, chemical analyses were made of post-mortem samples of the test b-15. These samples were analyzed to determine the amount of carbon dioxide removed and the quantity of oxygen evolved. The results of the chemical analysis and a comparison of these with the measured test results is shown in Table 4-2. These results show excellent agreement for carbon dioxide removal and good agreement with the oxygen evolution test results. It is believed that the discrepancy in the oxygen evolution results was due to post test oxygen evolution during the period that the canister was cooling prior to being unloaded.

The chemical analysis data was measured using the apparatus shown in Figure 4-82.



Chemical Test Apparatus

Figure 4-82

Hamilton $\frac{U}{A}$
Standard

Test Number	Canister No.	Time To .5-1.0-4.0 (Min.)	Chemical Wt. (Lb.)	Ave. Useable O ₂ (Lb./Hr.)	Time O ₂ < 0.36 lb/hr. (Min.)	Total Useable O ₂ (Lb.)	Total O ₂ (Lb.)
MSC2-15A	1	110-140-250	4.34	0.148	0	0.591	0.591
MSC2-15B	1	85- 95-210	4.45	0.156	26	0.604	0.604
MSC2-15C	1	90-120-245	4.64	0.137	0	0.549	0.549
MSC2-16A	3	200-240-315	3.99	0.149	0	0.598	0.598
MSC2-16B	3	150-195-275	4.06	0.085	0	0.341	0.341
MSC2-17A	1	80-100-205	4.44	0.146	30	0.544	0.639
MSC2-17B	1	125-150-285	4.55	0.133	0	0.531	0.533
MSC2-17C	1	145-180-265	4.20	0.124	0	0.496	0.496
MSC2-18A	1	155-170-233	4.47	0.075	0	0.296	0.296
MSC2-18B	1	225-260-350	5.24	0.109	0	0.435	0.435
MSC2-19	1	239-281-359	5.11	0.10	0	0.401	0.401
MSC2-20A	1	115-160-215	4.00	0.113	0	0.453	0.453
MSC2-20B	1	165-195-225	4.00	0.122	0	0.488	0.488
MSC2-20C	1	147-181-265	4.00	0.113	0	0.453	0.453
MSC2-21A	3	164-204-271	4.10	0.083	0	0.331	0.331
MSC2-21B	3	124-170-244	4.04	0.081	0	0.322	0.322
MSC2-22A	3	100-140-192	3.95	0.073	0	0.284	0.284
MSC2-22B	3	110-145-207	4.00	0.077	0	0.271	0.271
MSC2-23A	3	102-161-169	3.44	0.067	0	0.207	0.207
MSC2-23B	3	84- 97-158	3.43	0.066	0	0.181	0.181
MSC2-24A	3	98-129-227	3.99	0.089	0	0.356	0.356
MSC2-24B	3	116-159-264	3.80	0.091	0	0.363	0.363
MSC2-25A	1	94-135-228	4.60	0.106	0	0.425	0.425
MSC2-25B	3	125-158-232	4.40	0.103	0	0.412	0.412
MSC2-26A	3	127-155-214	4.02	0.102	0	0.385	0.385
MSC2-26B	3	121-156-213	3.96	0.098	0	0.371	0.371
MSC2-27A	3	90-108-153	3.64	0.094	0	0.255	0.255
MSC2-27B	3	103-116-154	3.56	0.076	0	0.208	0.208
MSC2-28	3	1- 2- 8	3.66	0.033	0	0.033	0.033
MSC2-29A	3	100-134-242	3.66	0.123	0	0.494	0.494
MSC2-29B	3	0-127-248	3.78	0.128	0	0.514	0.514
MSC2-30	3	178-229-394	3.97	0.121	0	0.483	0.483
MSC2-31	3	335-----	4.04	0.0165	0	0.066	0.066
MSC2-32	3	-----	4.04	0.037	0	0.149	0.149
MSC2-33	3	-----	3.91	0.062	0	0.247	0.247
MSC2-34	3	63- 78-167	4.01	0.140	50	0.415	0.633
MSC2-35	3	134-182-314	3.99	0.156	0	0.624	0.624
MSC2-36	3	181-223-318	3.79	0.116	0	0.464	0.464
MSC2-37A		8- 13- 73	3.09	0.044	0	0.059	0.059
MSC2-37B		30- 47- 89	3.11	0.071	0	0.119	0.119
MSC2-38A		12- 18- 42	2.91	0.0375	0	0.028	0.028
MSC2-38B		18-----	2.57	0.085	0	0.028	0.028
MSC2-38C		7- 12- 40	3.78	0.041	0	0.034	0.034
MSC2-39A		3- 8- 82	2.57	0.096	0	0.144	0.144
MSC2-39B		21- 23- 37	2.185	0.037	0	0.025	0.025

Chemical Analysis of Post Mortem Test Bed Samples

Test No. MSC2-	Initial O ₂ Con. cc/g	Final O ₂ Con. cc/g	Final CO ₂ Con. cc/g	Total CO ₂ Removed			Total O ₂ Generated			Final Bed Weight		
				Chem. Anal.	Test Data	Wca/ Wtd	Chem. Anal.	Test Data	Wca/ Wtd	Measured	Calculated	Wm/Wc
1A	242	42	197	1.8	1.8	1.0	1.01	1.0	1.01	5.09	4.61	1.1
1B	242	33	185	1.67	1.62	1.03	1.08	1.02	1.06	5.00	4.52	1.1
1C	224	99	192	1.77	1.81	.99	.50	.61	.82	5.13	5.70	.90
2A	242	127	165	1.56	1.57	.99	.433	.405	1.07	5.26	5.60	.94
2B	242	114	162	1.51	1.54	.98	.53	.42	1.26	5.17	4.75	1.09
3A	198	74	206	1.73	1.64	1.05	.55	.37	1.48	4.66	4.73	.98
3B	198	29	173	1.38	1.28	1.08	.83	.78	1.06	4.44	4.40	1.01
3C	198	62	205	1.79	1.88	.95	.63	.63	1.0	4.85	4.60	1.06
3D	198	57	210	1.78	1.80	.99	.67	.65	1.03	4.71	4.60	1.02
4A	221	48	226	1.94	1.95	.99	.68	.80	.85	4.80	4.42	1.09
4B	198	56	209	1.94	1.94	1.0	.68	.66	1.03	5.16	5.87	.88
5A	228	106	193	1.77	1.75	1.01	.43	.595	.725	5.08	5.36	.95
5B	228	107	185	1.67	1.69	.99	.44	.64	.69	5.02	5.06	.99
6A	228	135	131	1.07	1.1	.97	.34	.21	1.63	4.52	4.54	.99
6B	228	109	173	1.57	1.57	1.0	.41	.37	1.11	5.06	4.77	1.06
7A	233	134	164	1.43	1.54	.93	.39	.535	.73	4.84	5.70	.85
7B	230	92	185	1.75	1.80	.97	.585	.55	1.06	5.27	4.63	1.13
8A	224	52	221	2.12	2.14	.99	.80	.81	.99	5.32	4.85	1.10
8B	224	65	213	2.08	2.14	.97	.78	.82	.95	5.42	5.57	.97
9A	210	54	212	1.84	1.78	1.03	.75	.70	1.07	4.83	4.63	1.04
9B	210	28	192	1.61	1.60	1.00	.70	.89	1.01	4.65	4.84	.96

Table 4-2

Hamilton U
Standard A₂

STP-1

4.2 Continued

A fifty gram, homogeneous chemical sample is placed in the flask. Any oxygen in the chemical is released when a saturated potassium permanganate solution is added and is measured by the wet test meter. The carbon dioxide in the chemical is released by adding a 50% sulfuric acid solution in 100 milliliter increments. Gas evolution is complete when the addition of a 100 milliliter increment of sulfuric acid increases the meter reading by only that volume.

5.0 PERFORMANCE ANALYSIS

This section presents the performance analysis results for the test data of Section 4.0.

5.1 Catalyst Evaluation

Various catalyzing agents were evaluated during this phase of the program in an effort to determine the most favorable activator for promoting oxygen evolution from lithium peroxide. Although the primary purpose of catalyst addition is the enhancement of oxygen evolution, CO₂ absorption performance was also considered in the catalyst selection.

Those catalysts which were subjected to the most extensive testing included ferrous sulfate (FeSO₄), manganese oxide (MnO), and manganese dioxide (MnO₂). Other catalysts evaluated on a limited testing basis included titanium dioxide (TiO₂), manganese oxide with silver coated copper wire, silver nitrate (AgNO₃), and calcium oxide (CaO). Nickel sulfate (NiSO₄) was considered on the basis of testing conducted during Phase I.

The test item utilized for this phase was canister No. 3, shown in Figure 3-7. The canister face measures 6.6 in. x 14.0 in., giving a flow area of 92.5 in². The bed length for all tests was 2.87 in. A continuous 370 inch length of 1/8 inch diameter copper tubing was used for bed cooling. The cooling coil provides flow in a plane perpendicular to the direction of flow. Cooling water enters the coil on the downstream face of the bed and exits after passing through the row of coils along the upstream face.

Catalysts were evaluated at various cooling rates to determine the bed temperature range providing optimum performance. The maximum rather than the average bed temperature was used as a basis for establishing the optimum performance temperature range since it is this temperature which is indicative of the reaction taking place. Monitoring of thermocouples located throughout the bed shows the maximum temperature proceeding from the inlet to the outlet side of the bed coincident with the location of the reaction as the test proceeds to completion.

Table 5-1-1 presents a summary of the results for those tests conducted during this phase. The columns of tabulation are defined as follows:

- (1) Test Number
- (2) Catalyst (Chemical Formula Shown)

Performance Analysis Summary

Test Number	Catalyst	Chemical Wt. (Lbs.)	Time To .5-1.0-4.0 (Min.)	Ave Useable O ₂ (Lb./Hr.)	Max. T °F	Cooling Water Flow Rate (Lb./Hr.)
MSC2-1A	MnO	4.05	196-214-265	0.2	647	None
MSC2-1B	MnO	4.10	174-190-222	0.23	640	None
MSC2-1C	MnO	4.00	155-180-255	0.128	350	3
MSC2-1D	MnO	3.82	186-200-235	0.192	630	None
MSC2-2A	MnO	4.12	145-182-227	0.167	185	30
MSC2-2B	MnO	4.12	137-160-217	0.105	196	30
MSC2-3A	FeSO ₄	3.87	146-169-235	0.0837	260	5
MSC2-3B	FeSO ₄	3.85	132-149-189	0.175	625	None
MSC2-3C	FeSO ₄	3.95	170-215-277	0.13	270	3
MSC2-3O	FeSO ₄	3.92	180-210-265	0.14	325	2
MSC2-4A	FeSO ₄	3.38	195-220-284	0.165	230	30
MSC2-4B	FeSO ₄	4.02	200-238-295	0.138	190	30
MSC2-5A	MnO ₂	3.80	170-200-265	0.135	515	0, 10 & 90
MSC2-5B	MnO ₂	3.81	133-162-245	0.15	270	5
MSC2-6A	MnO ₂	3.82	138-151-166	0.08	214	30
MSC2-6B	MnO ₂	3.78	140-167-232	0.095	196	30
MSC2-7A	TiO ₂	4.06	138-170-230	0.134	325	3, 5 & 80
MSC2-7B	MnO + Ag/Cu	4.05	170-189-265	0.12	190	30
MSC2-7C	No Cat	3.94	165-185-235	0.089	210	30
MSC2-7D	Ag NO ₃	3.64	180-240-295	0.101	200	30
MSC2-7E	CaO	4.03	151-205-298	0.136	325	2
MSC2-8A	MnO	3.95	185-223-314	0.17	475	T _B 350 - 450
MSC2-8B	MnO	4.23	165-211-320	0.175	481	T _B 425 - 475
MSC2-9A	FeSO ₄	3.45	174-202-263	0.16	473	T _B 400 - 500
MSC2-9B	FeSO ₄	3.89	170-185-227	0.178	667	1/2
MSC2-10A	MnO	3.88	45- 70- 91	0.192	730	None
MSC2-10B	MnO	3.85	98-181-213	0.125	355	2
MSC2-10C	MnO	4.15	180-182-303	0.146	364	2
MSC -14	NiSO ₄	3.38	0- 8-202	0.11	309	30
MSC -15	NiSO ₄	3.18	0- 90-210	0.14	542	30
MSC -16	NiSO ₄	3.26	25-110-240	0.14	560	30
MSC -16b	NiSO ₄	3.13	20-100-205	0.15	552	30
MSC -16c	NiSO ₄	3.30	67- 86-140	0.25	592	30
MSC -16d	NiSO ₄	3.70	145-185-280	0.12	360	30
MSC -20	NiSO ₄	3.23	70- 95-195	0.13	563	30

TABLE 5-1-1

5.1 Continued

- (3) Chemical Weight (lbs)
- (4) The elapsed time at which the canister outlet partial pressure of carbon dioxide is 0.5, 1.0, and 4.0 mm Hg (minutes).
- (5) Average usable oxygen generation rate (lb/hr) to an elapsed time of 240 minutes. This excludes all oxygen which would be vented overboard during those periods where generation is in excess of 0.36 lb/hr.
- (6) Maximum bed temperature (°F)
- (7) Cooling water flow rate (lb/hr)

Data from Phase I testing of nickel sulfate catalyst is also presented for selected tests where the chemical weight and canister geometry are the same as those of the Phase II tests.

From the results tabulated in Table 5-1-1 and a study of the performance curves shown in Figures 5-1-1 through 5-1-7, lithium peroxide catalyzed with manganese oxide is shown to provide the best CO₂ absorption and O₂ generation performance. The following discussion provides a justification for the observed performance of those catalysts tested. This explanation of results has been made in terms of their agreement with previously published literature concerning the nature of Li₂O₂ reactions.

General Observations

Before proceeding to evaluate the catalyst performance, the following general observations are presented which constitute the basis on which explanation of the test results can be made.

Based on tests conducted during this phase and on previous test experience, it is apparent that optimum CO₂ absorption and O₂ generation performance must necessarily occur at a bed temperature range below that of rapid lithium peroxide thermal decomposition. Rapid decomposition results in an undesirably short period of high O₂ evolution and subsequent degraded CO₂ performance resulting from poor utilization of the lithium oxide (Li₂O) product.

As shown by Boryta in Reference 1, good CO₂ absorption by lithium peroxide, requires the existence of the intermediate product lithium hydroxide monohydrate. This can be explained in terms of molar volume ratios where it is desired that the reaction product be of smaller volume than the substrate. This condition decreases

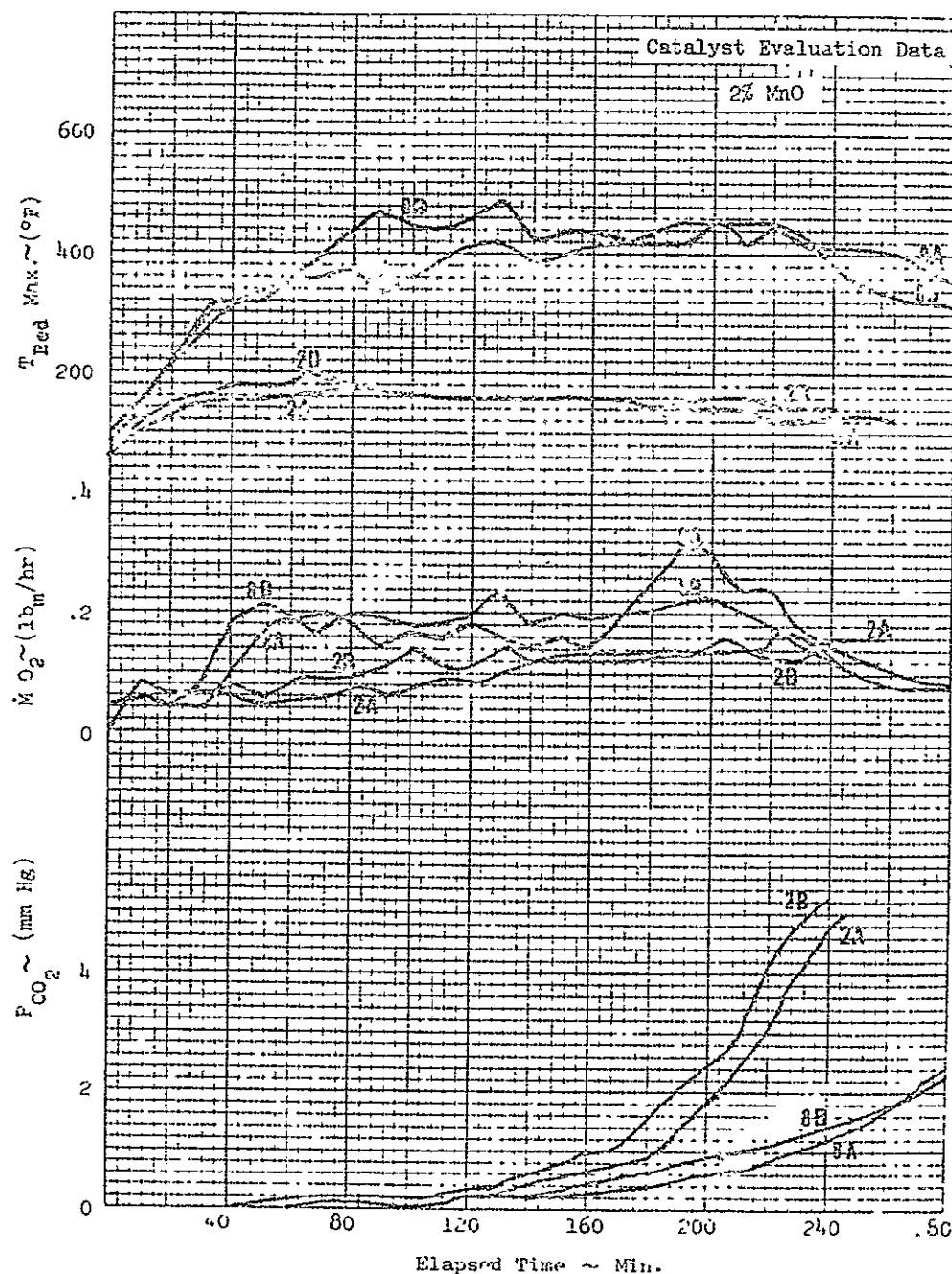
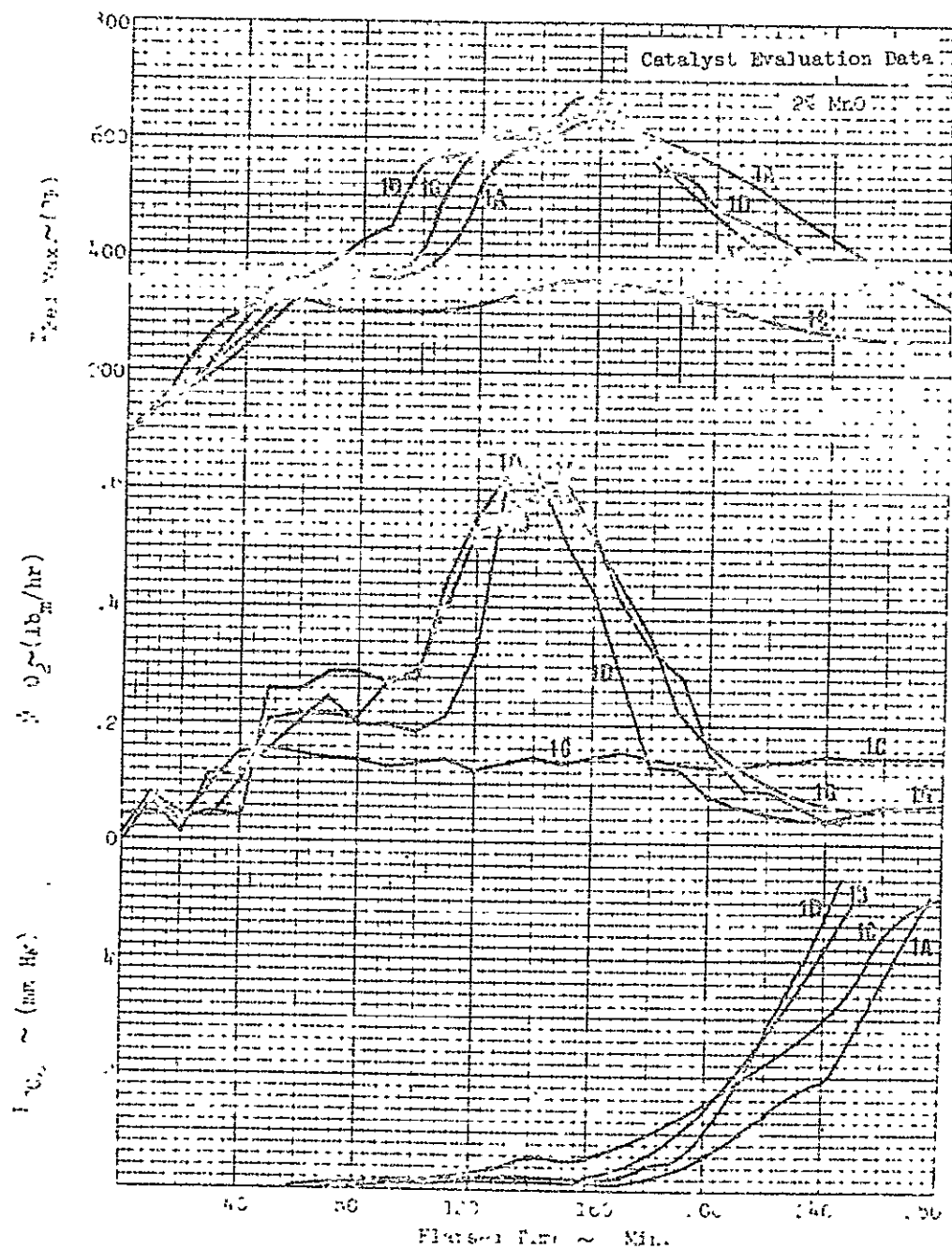


Figure 5-1-1



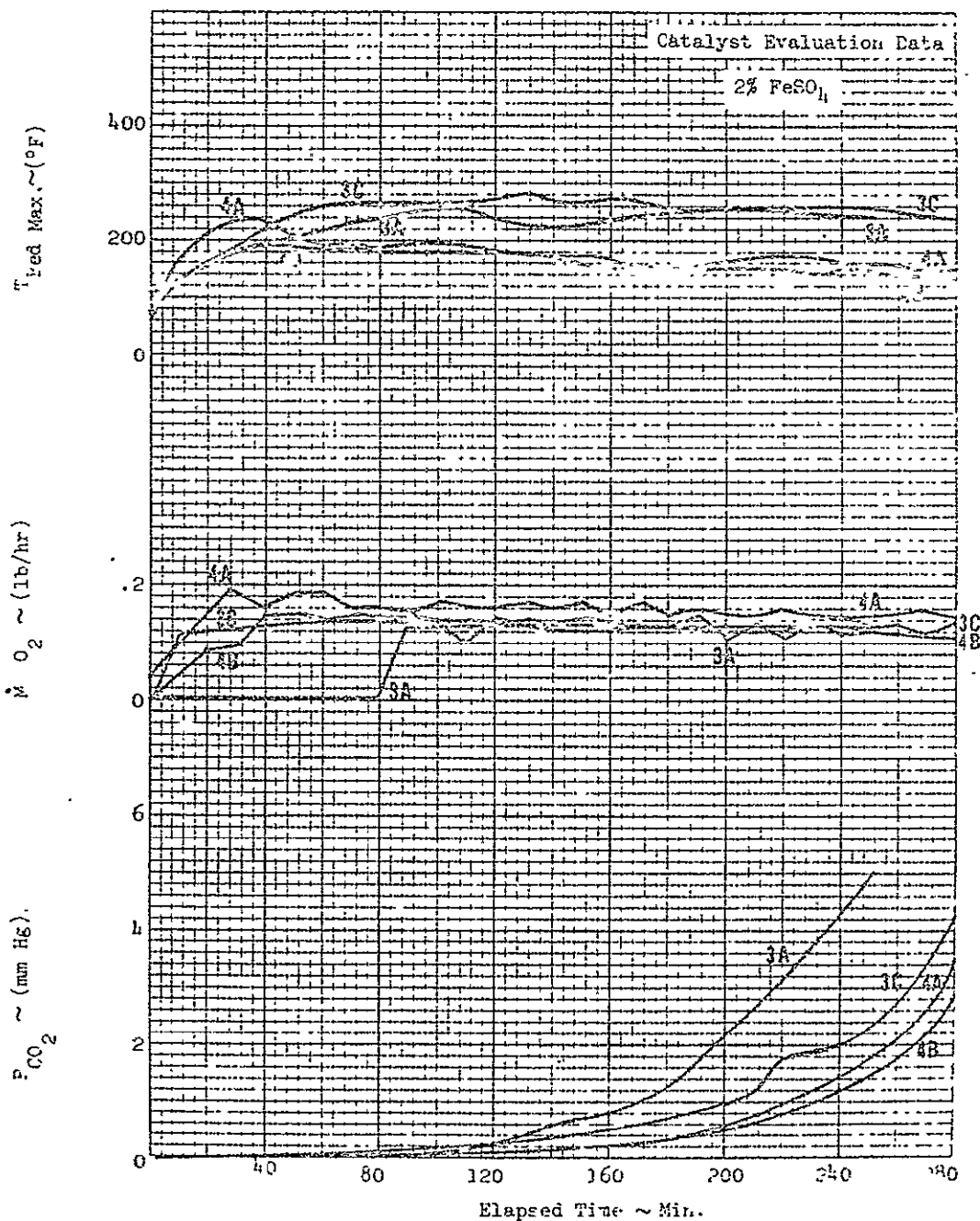


Figure 1-1-3

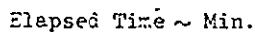


Figure 5-1-4

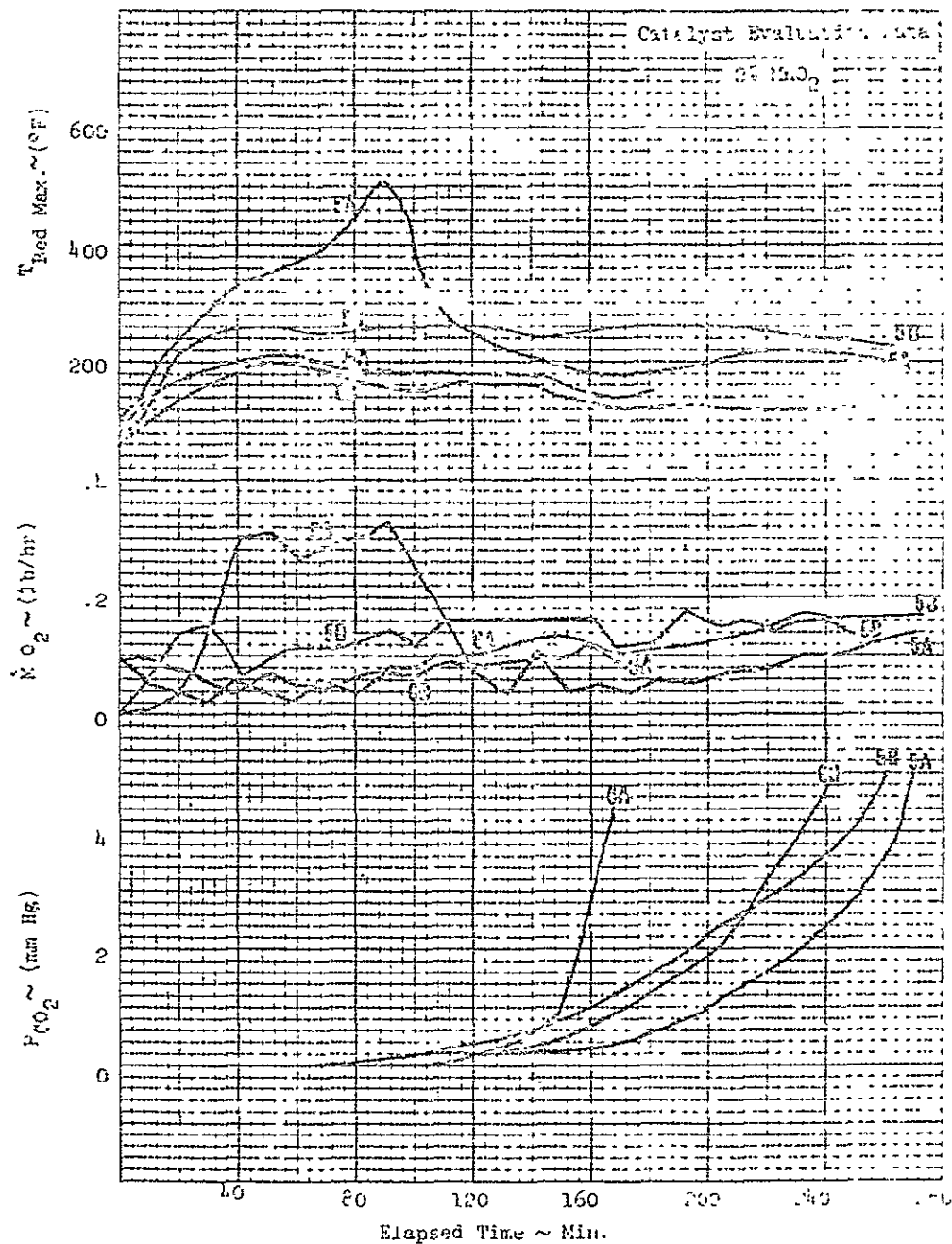


Figure 5-1-5

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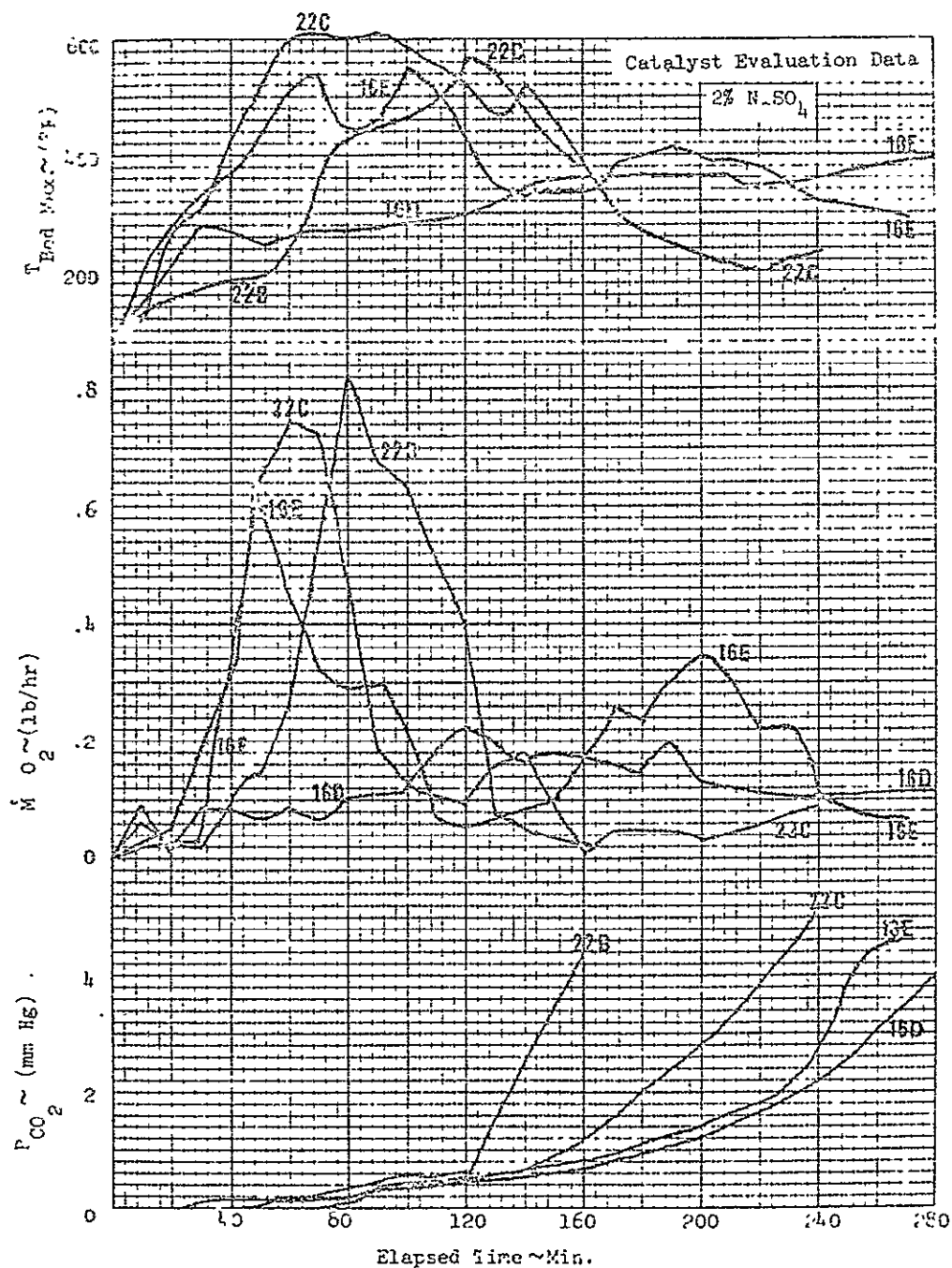


Figure 5-1-6

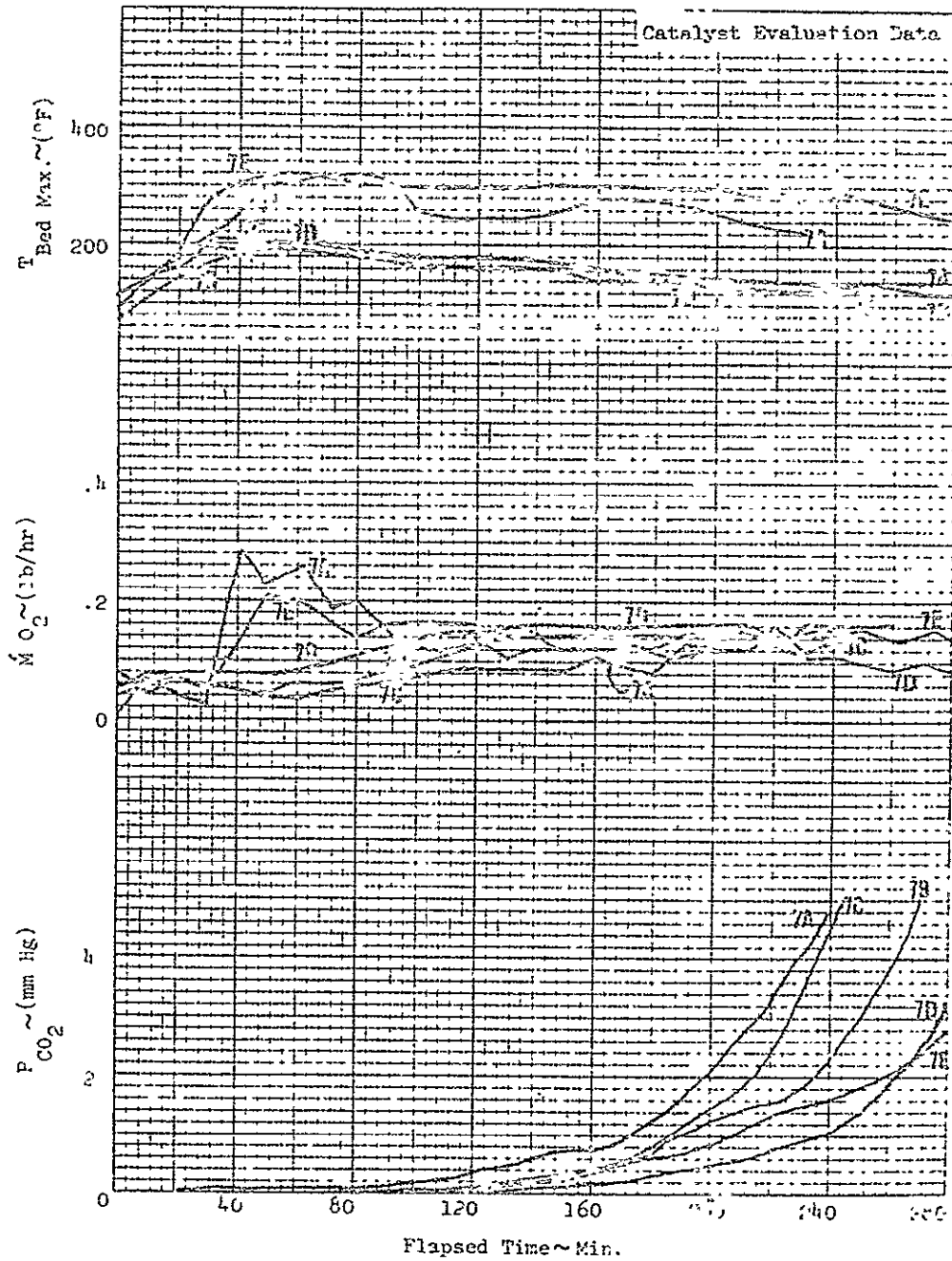


Figure 5-1-7

5.1 Continued

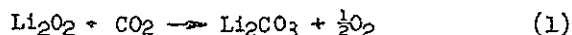
any diffusion resistance to CO₂, which may be created by a reaction product coating over the unreacted substrate.

The poor CO₂ utilization, typical of those tests where rapid lithium peroxide decomposition occurs, is then the result of the absence of monohydrate as an intermediate product. The lithium oxide reacting with CO₂ becomes coated with a lithium carbonate (Li₂CO₃) product of higher molar volume. Performance decreases as a result of the diffusion resistance created by the particle coating. The following tabulation shows the CO₂ utilization efficiency to be noticeably higher for those tests where no significant decomposition has occurred.

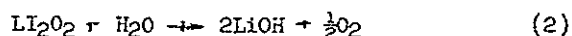
Test No.	Catalyst	Weight Pounds	Max. T OF	CO ₂ Utilization	Decomposition
				Efficiency (%) at 4.0 mmHg	
3B	FeSO ₄	3.85	630	35	Yes
9B	FeSO ₄	3.89	567	42	Yes
3D	FeSO ₄	3.92	325	40	No
9A	FeSO ₄	3.95	478	48	No
1A	MnO	4.05	630	47	Yes
1B	MnO	4.10	680	30	Yes
8A	MnO	3.95	475	57	No
8B	MnO	4.23	481	54	No

It is desirable to operate at some temperature below that point where rapid decomposition occurs. Favorable CO₂ absorption and O₂ generation performance have been observed at two temperature ranges; one range existing at low temperature, up to 225° and the other at high temperature, 400 to 500°F.

At the low bed temperature range good CO₂ performance is possible through the existence of lithium hydroxide monohydrate. The monohydrate, as explained previously with relation to lithium oxide, serves as an intermediate which provides a good molar volume ratio. From tests conducted by Selezneva, Reference 2, the percent of theoretical CO₂ absorbed directly by lithium peroxide at low temperature is very small.

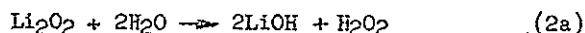


With water vapor only, however, a reaction resulting in O₂ generation begins at much lower temperatures.



Continued

The net reaction above can also be represented by the following equations which are described by Markowitz in Reference 3 and Bach in Reference 4.



As shown in Figure 5-4-1, CO_2 absorption is greater in the presence of water vapor. The presence of water vapor shows no direct relationship to improved CO_2 performance if the mechanism of absorption involves equation 1. No lithium hydroxide is formed by this reaction and there is no explanation as to why low temperature should result in improved CO_2 performance. Equation 2, referred to as the hydrolysis of lithium peroxide, is the more likely reaction. The presence of CO_2 would improve performance since CO_2 reacting with lithium hydroxide would create additional water to increase the hydrolysis of the peroxide.

The favorable performance shown at the low temperature range can then be explained in terms of CO_2 absorption by lithium hydroxide and lithium hydroxide monohydrate and the resultant effect of the monohydrate on this performance. Tests conducted at Hamilton Standard with water cooled lithium hydroxide have substantiated the desirability of operation at low bed temperatures.

It has been specified that this low temperature range should be below 225°F . The temperature referred to is the maximum bed temperature or temperature of the reaction zone where equation 2 is occurring. Most of the chemical bed, however, is below 150°F where lithium hydroxide monohydrate can exist and the absorption of CO_2 can take place readily.

As the bed temperature rises above this range an attendant deterioration in CO_2 performance results due to increasing instability of the monohydrate. This trend continues until vigorous CO_2 absorption can take place by direct reaction with lithium peroxide; equation 1. This is shown to occur at approximately 390°F . Good CO_2 performance exists between this temperature and that point where rapid lithium peroxide decomposition begins, approximately 500°F . Above the point of rapid decomposition, CO_2 performance deteriorates due to poor utilization of the lithium oxide product as described previously.

With respect to O_2 production, the general observation made from phase I testing showed that increased bed temperature resulted in

5.1 Continued

higher O₂ evolution. This is substantiated by the phase II test results shown in Figure 5-1-8. Oxygen evolution at low temperatures is provided through thermal decomposition of the hydrogen peroxide created by hydrolysis of lithium peroxide. As the temperature increases hydrolysis increases, as shown in Figure 5-1-9, and hydrogen peroxide decomposition is more complete. The result is increased O₂ generation. As the temperature approaches 390°F, more of the O₂ evolved is due to direct reaction of CO₂ with the lithium peroxide. This continues until the temperature is reached where rapid thermal decomposition of lithium peroxide accounts for virtually all of the O₂ produced.

Catalyst Performance

Having established the general characteristics described in the previous section, application of these observations can be made in explaining the results of the catalyst evaluation tests.

Those catalysts subjected to a sufficient number of tests to establish performance trends with respect to bed temperature appear to fall into the following two categories:

- (1) Catalysts which show good CO₂ and O₂ performance at the low bed temperature range, below 225°F.
- (2) Catalysts which show good CO₂ and O₂ performance at the high bed temperature range, 400 to 500°F.

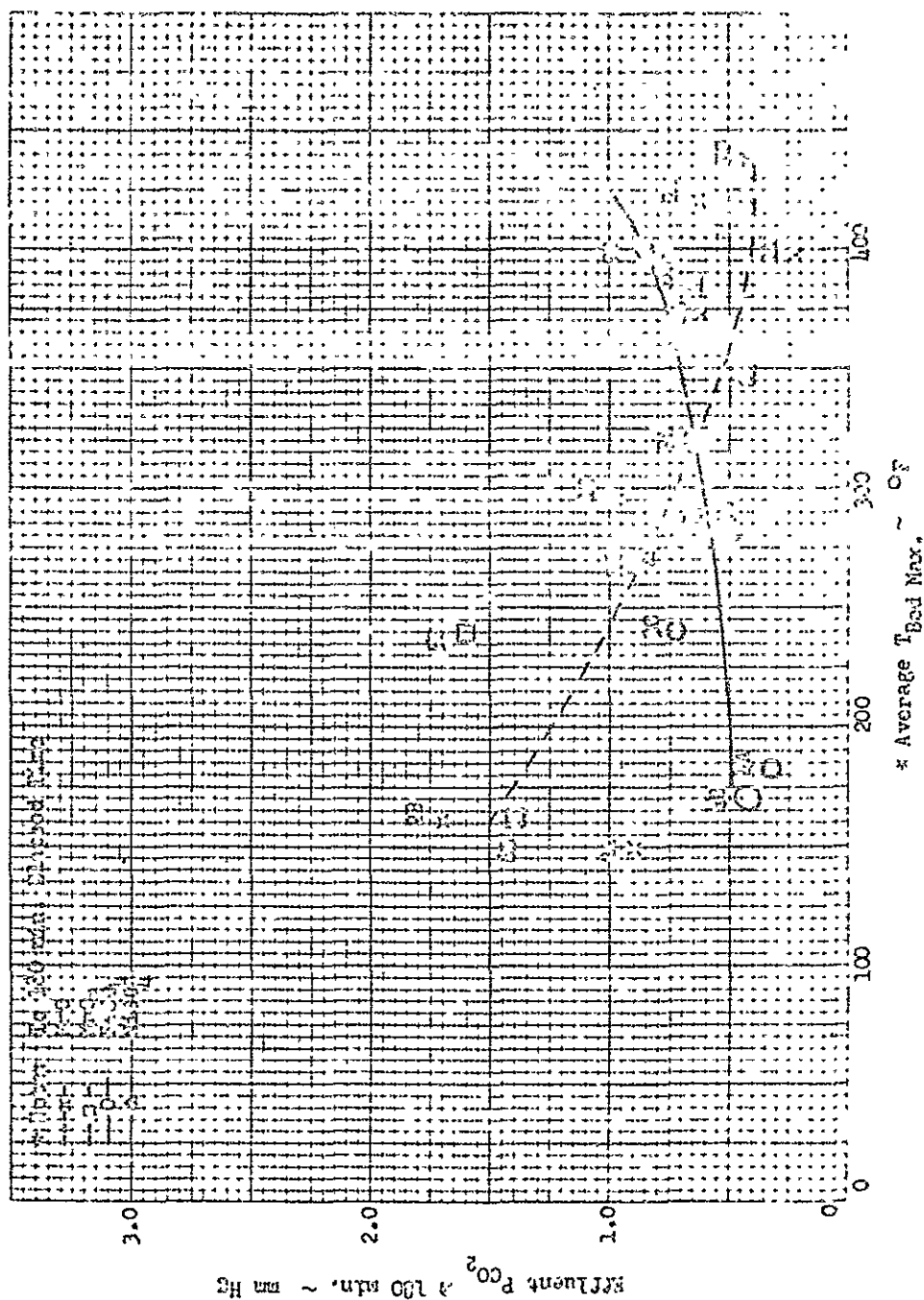
The high and low temperature ranges referred to here are those previously discussed as showing good CO₂ performance. The more active catalysts, as indicated by the heat generated during the manufacturing process when the catalyst and lithium peroxide powder are mixed, fall into the first category. They would include ferrous sulfate and nickel sulfate. Their performance trends are shown in Figures 5-1-3, 5-1-4, and 5-1-6. The less active catalysts, manganese oxide and manganese dioxide, are included in the second category; their general performance trends being shown in Figures 5-1-1, 5-1-2, 5-1-5.

Other catalysts were tested on a limited basis, but their evaluation is restricted to the observed performance at only one temperature level. In no case was exceptional performance indicated. The evaluation is therefore limited to those four catalysts mentioned which have undergone sufficient testing to demonstrate their performance capabilities.

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CO₂ Partial Pressure vs. Bed Temperature

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5.1 Continued

The better overall performance shown by ferrous and nickel sulfate at lower temperatures can be seen in Figures 5-1-8 and 5-1-9. These compounds, being active peroxide catalysts, are effective at low temperatures in decomposing hydrogen peroxide, creating the lithium peroxide hydrolysis. Good O_2 evolution at low temperatures is a result of the effectiveness in decomposing the hydrogen peroxide into its water vapor and oxygen products. The water vapor available by this reaction serves to promote hydrolysis of the lithium peroxide creating additional lithium hydroxide and monohydrate. The result is increased CO_2 absorption. At the higher temperature range, however, CO_2 absorption decreases. It is the apparent result of lithium peroxide decomposition occurring at or below the $300^\circ F$ point shown by Selezneva as that temperature necessary for active CO_2 absorption. In other words, the temperature for active CO_2 absorption directly by lithium peroxide cannot be attained by the active catalysts without incurring rapid decomposition. It should be noted that the catalysts were selected on the basis of being activators for decomposing hydrogen peroxide. Their effect, however, is also to lower the decomposition temperature of lithium peroxide. In decomposition tests carried out at Hamilton Standard, it was observed that the addition of 2 percent nickel sulfate lowered the beginning of the decomposition range from $531^\circ F$ to $467^\circ F$. Ferrous sulfate, being more active, would tend to lower this temperature even further; thus narrowing the range of desirable high temperature operation which must exist above $390^\circ F$.

In the case of the weaker catalysts, manganese oxide and dioxide, low temperature operation is poor. The catalyst is not active enough to effectively decompose the hydrogen peroxide. A low O_2 generation rate results and CO_2 performance is poor since the water needed to promote lithium peroxide hydrolysis is made unavailable as undecomposed hydrogen peroxide. At higher temperature, however, a sufficient range exists which allows active CO_2 absorption directly by lithium peroxide without incurring rapid decomposition.

At low temperature, nickel sulfate and ferrous sulfate show good overall performance. Ferrous sulfate, however, being the more active of the two catalysts provides the best performance as shown in Figures 5-1-8 and 5-1-9 by tests MCC2-4A and 4B. The higher activity of ferrous sulfate provides the most effective low temperature decomposition of hydrogen peroxide of any catalyst tested.

At high temperatures, 400 to $500^\circ F$, all the catalysts show good O_2 evolution, but only the less active catalysts, manganese oxide and dioxide, are accompanied by good CO_2 absorption. Manganese oxide

5.1

Continued

is the only one of these two catalysts for which sufficient data exists at higher temperatures to firmly establish the desirability of its performance in this range. The performance shown by the weaker catalysts at high temperature leaves some question as to whether any catalyst is required at all for operation at this range. The reactions involved in the CO_2 absorption - O_2 generation process at high temperature, as described previously, involve direct absorption of CO_2 by the lithium peroxide with O_2 as the resultant reaction product; no catalyst being actively involved. The test data available for uncatalyzed peroxide at this temperature range, however, is insufficient to establish any definite conclusions regarding its performance as being comparable to manganese oxide catalyzed peroxide.

The best performance of ferrous sulfate and manganese oxide are shown by tests MSC2-4A and 4B and tests MSC2-1A, 1B and 1C, respectively. For these tests, Figures 5-1-8 and 5-1-9 show comparable CO_2 performance for the two catalysts but at 150°F the higher usable O_2 production for the manganese oxide; the higher temperature of the manganese oxide tests being more effective in releasing O_2 than the catalyzing effect of ferrous sulfate at low temperature. On an overall performance basis, therefore, manganese oxide provides the best results of those catalysts evaluated, but it does not achieve the desired result of promoting low temperature oxygen evolution.

Small-Scale Catalyst Testing

Twenty four additional tests, using 6 gram samples, were run to evaluate various catalysts to promote oxygen generation for Li_2O_2 via the water vapor reaction.



The objective was to identify a catalyst which would promote this reaction at low temperatures ($<150^\circ\text{F}$) so that maximum benefit could be obtained from the LiOH for CO_2 control.

The results of the program showed that the catalysts fell into two categories:

- (1) They reacted so rapidly with the Li_2O_2 that it was impossible to manufacture granules, or reacted violently at elevated temperatures.

5.1 Continued

- (2) They showed little or no increased O_2 evolution over uncatalyzed Li_2O_2 below about $300^\circ F$. Above this temperature, some of the precious metals showed evidence of increased generation rates. The O_2 evolution as a function of temperature for these materials is shown of Figures 5-1-10 through 5-1-13.

Table 5-1-2 lists the catalysts investigated and lists pertinent comments for those which could not be mixed with Li_2O_2 .

A test rig, shown schematically in Figure 5-1-11, was constructed in the RSD Product Research Laboratory. Small lithium peroxide samples (approximately 6 grams) were placed in the glass U-tube through which was passed a constant flow of humidified nitrogen at a $65^\circ F$ dew point, and 1 atmosphere total pressure. The U-tube was placed in an electrically heated sand bed and temperature measured by a thermocouple placed in contact with the external surface of the U-tube at the lower portion of the tube. The effluent gas from the U-tube was passed through a gas chromatograph to detect oxygen. The dew point of the effluent gas was held constant at $65^\circ F$. The test conditions were scaled to be representative of the conditions existing for a 4 lb. Li_2O_2 bed at APLES conditions. These conditions were:

Carrier gas - N_2
Inlet flow rate - 800 cc/min (0.026 sc/min)
Pressure - 14.7 psia
Inlet dew point - $65^\circ F$
Bed temperature rise - $40^\circ F/min$

The first two tests were run over a temperature range of 100 to $250^\circ F$. Oxygen liberation at these temperatures proved to be insignificant, and in order to expedite testing, it was decided to screen the samples over a higher temperature range ($250-400^\circ F$) and retest, starting at a lower temperature, with those candidates which showed promise at elevated temperatures. Unfortunately, none of the catalysts tested provided high O_2 yields below about $350^\circ F$. The data is presented in Figures 5-1-10 through 5-1-13 and shows the silver nitrate catalyst provided the highest O_2 yield (85% of the available O_2) but not until the bed temperature reached $400^\circ F$.

The tabulation below shows the percent of the available O_2 evolved as a function of temperature for the various catalysts.

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Continued

Catalyst	Operating Conditions			
	Temp.	Pressure	Flow	Conversion
Benzotripyridine	4	1	10	10
Vanadium pentoxide	2	1	10	10
Vanadium pentoxide	1	11	10	10
Pd on asbestos	3	3	10	10
Pt on asbestos	0	3	14	10
Ru on carbon	0	5	15	10
Ru on CaCO_3	4	6	14	10
Ferrous Sulfate	0	0	10	10
Vanadium pentoxide	1	3	10	10
Pd on aluminum	0	0	21	10
Ru on aluminum	0	6	21	10
Ru on CaCO_3	0	5	21	10

It can be seen that little or no benefit is derived from the catalysts until the bed temperature reached in the order of 100°C. Further, the manganese oxide (MnO), which has been reported to give good performance in full scale (Advanced Portable Life Support System) tests, provided inferior performance.

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Catalysts Investigated

Listed below are the catalysts which have been investigated. The information regarding the unstable catalysts. All were of 1/4 inch diameter, unless otherwise noted.

1. Palladium on asbestos
2. Rhodium on carbon
3. Manganese oxide
4. Ferrous sulfate
5. 1% Rhodium on calcium carbonate
6. 5% Ruthenium on carbon
7. Palladium on carbon
8. Platinum on asbestos
9. Ruthenium on calcium carbonate
10. Ruthenium on aluminum powder
11. Palladium on aluminum powder
12. Silver nitrate
13. Silver needles
14. Nickel sulfate
15. Copper oxide - Decomposed at 200-260°F with a flame and a violent reaction
16. Sodium Formaldehyde sulfoxylate - Decomposed at 200-300°F with a violent reaction
17. Sodium hydrosulphite - Decomposed at 200°F with a violent reaction
18. Zinc formaldehyde sulfoxylate - Decomposed at 400°F with a violent reaction
19. Zinc oxide - Decomposed at 400-500°F with a violent reaction
20. Cobalt Napthanate - Reacted during mixing, granules could not be made
21. Magnesium - Violent reaction during mixing
22. Titanium dioxide
23. Calcium oxide
24. Alumina powder

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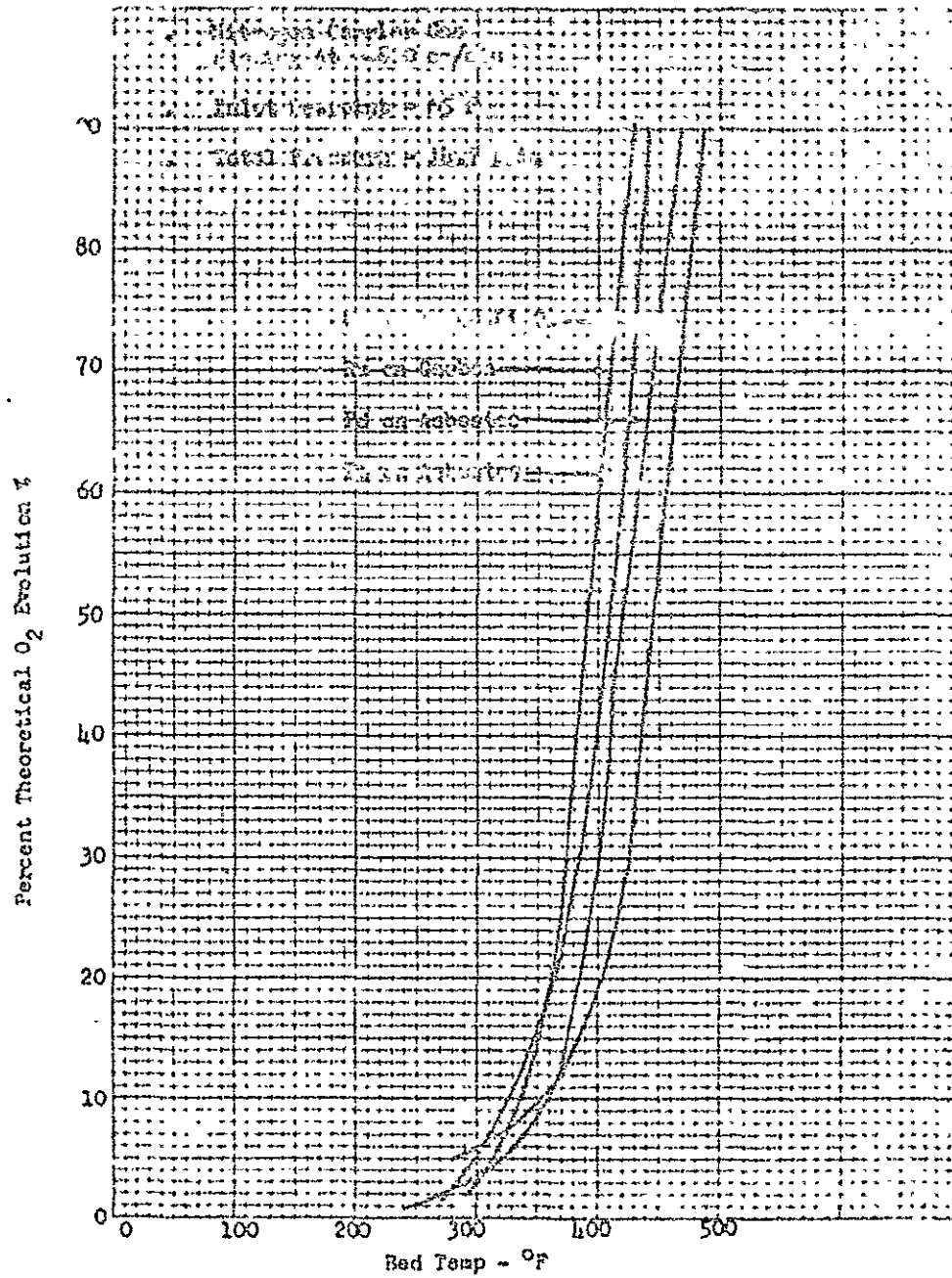


Figure 5-1-10

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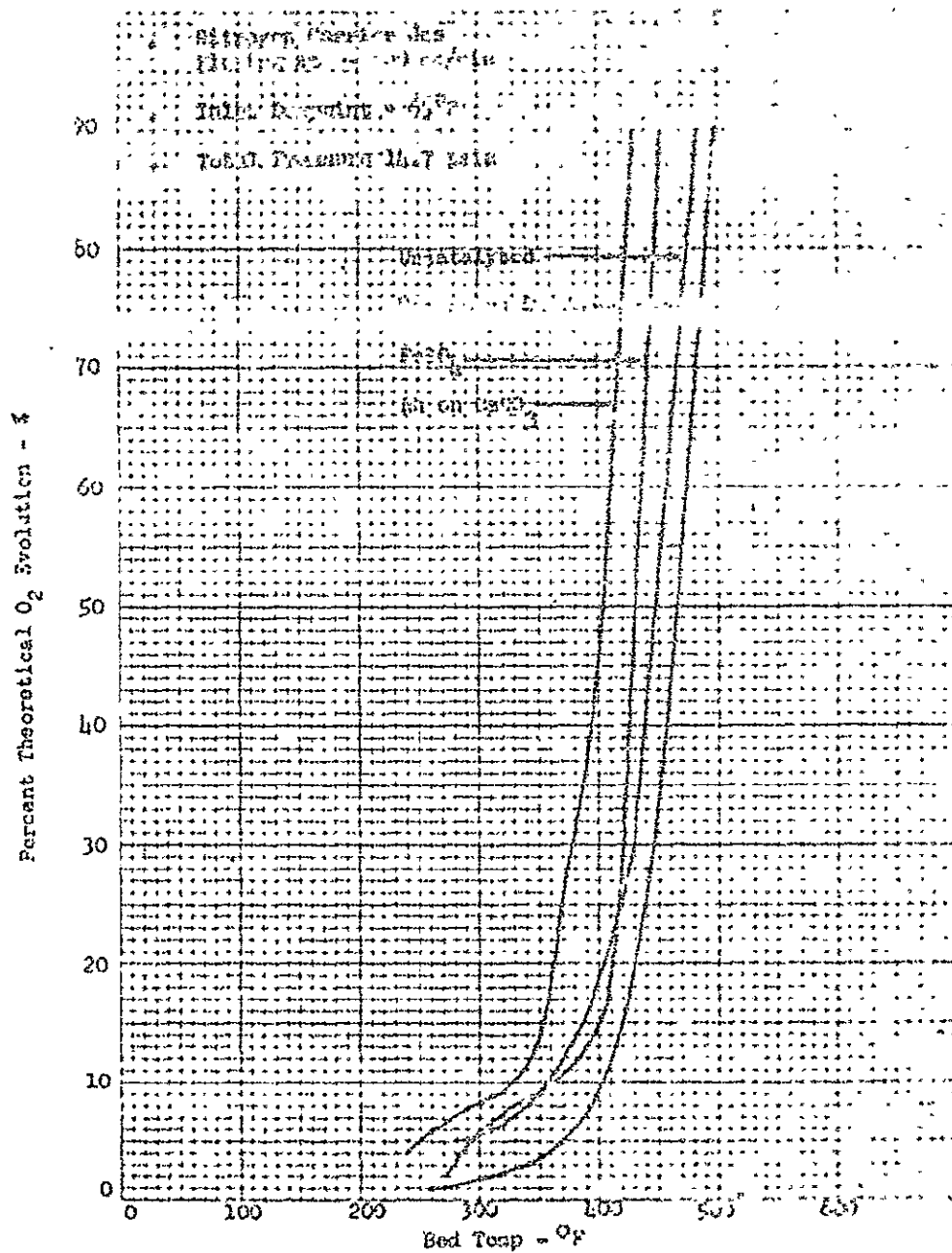


Figure 5-1-11

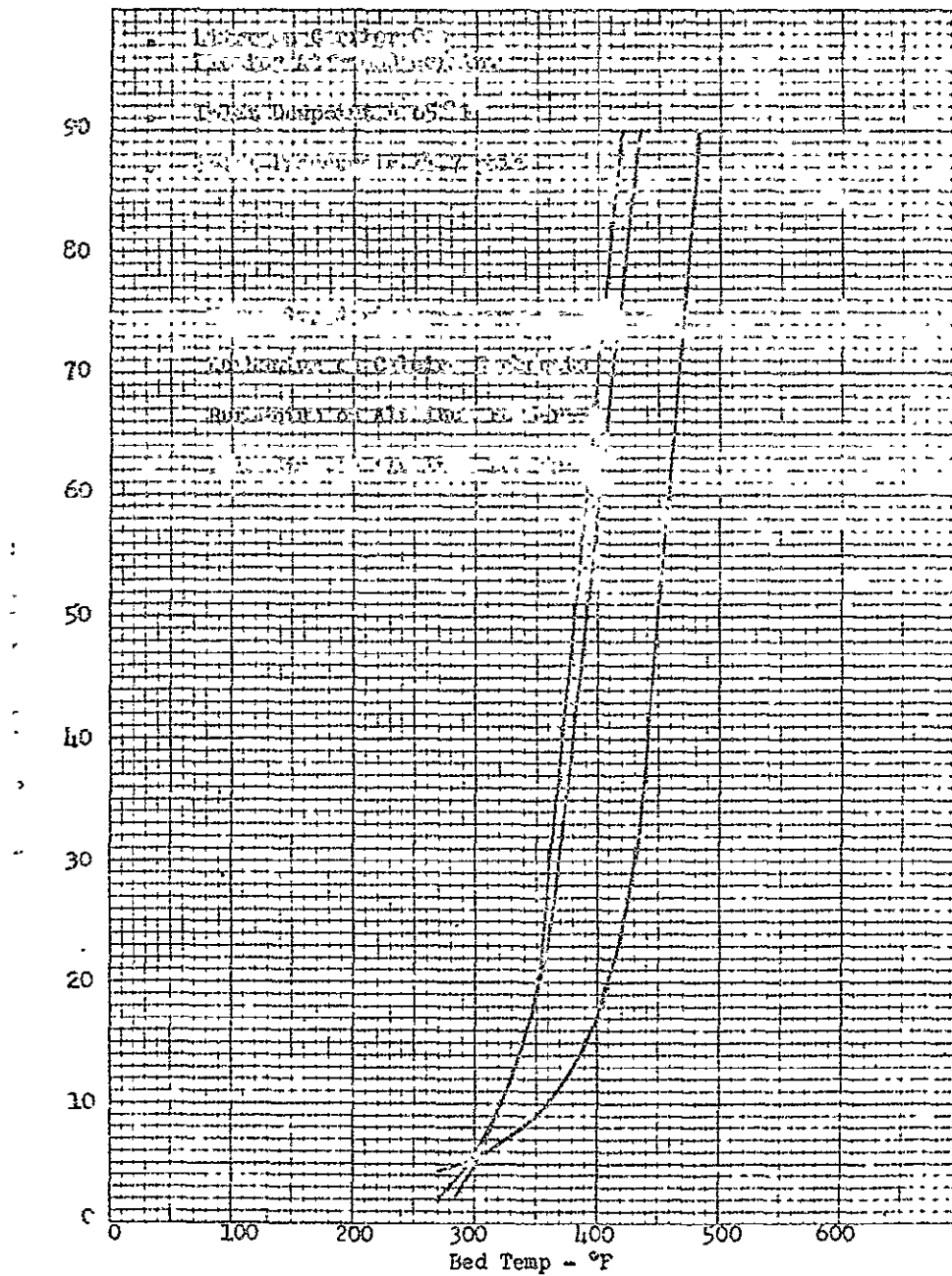


Figure 5-1-12

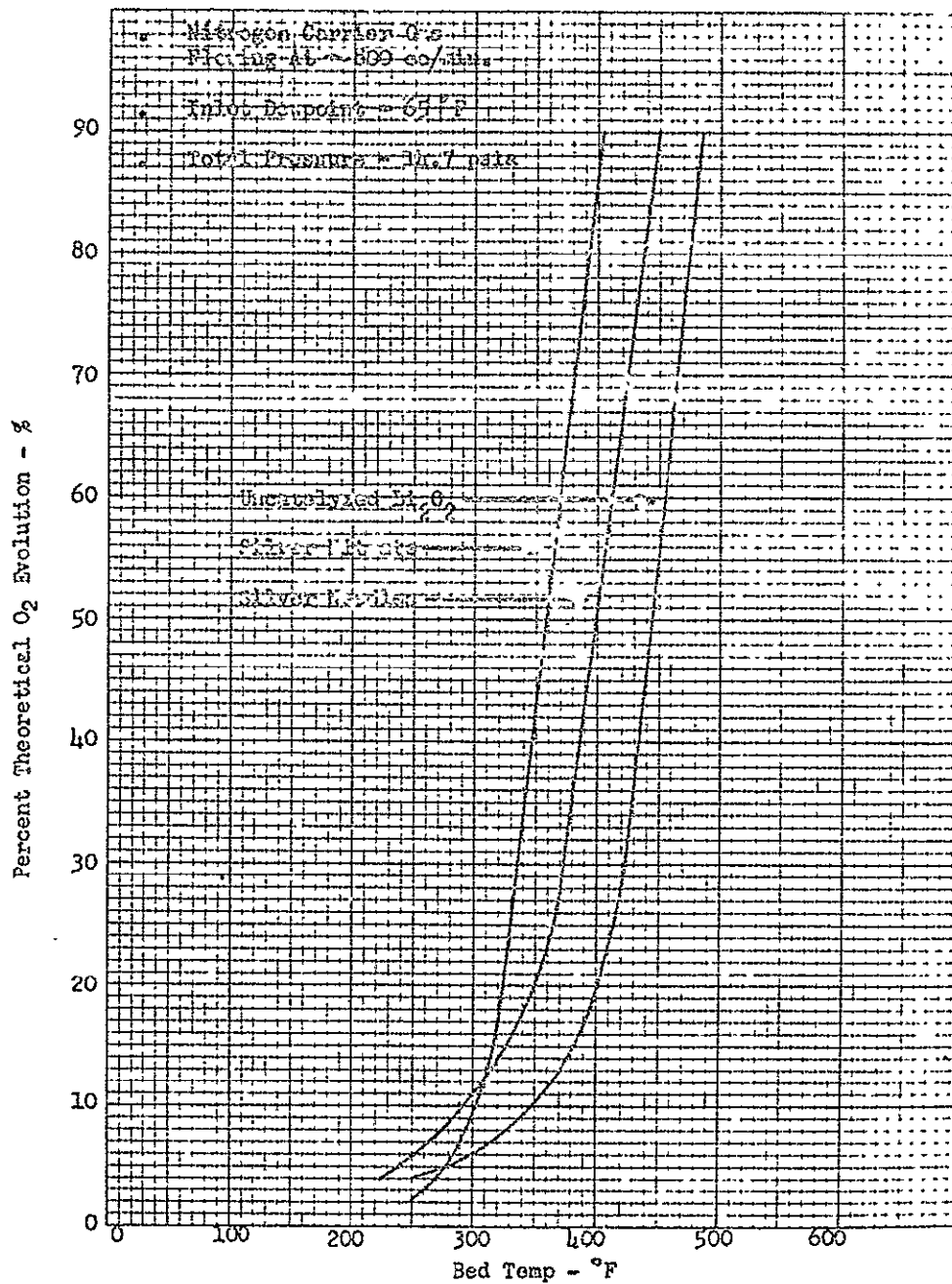
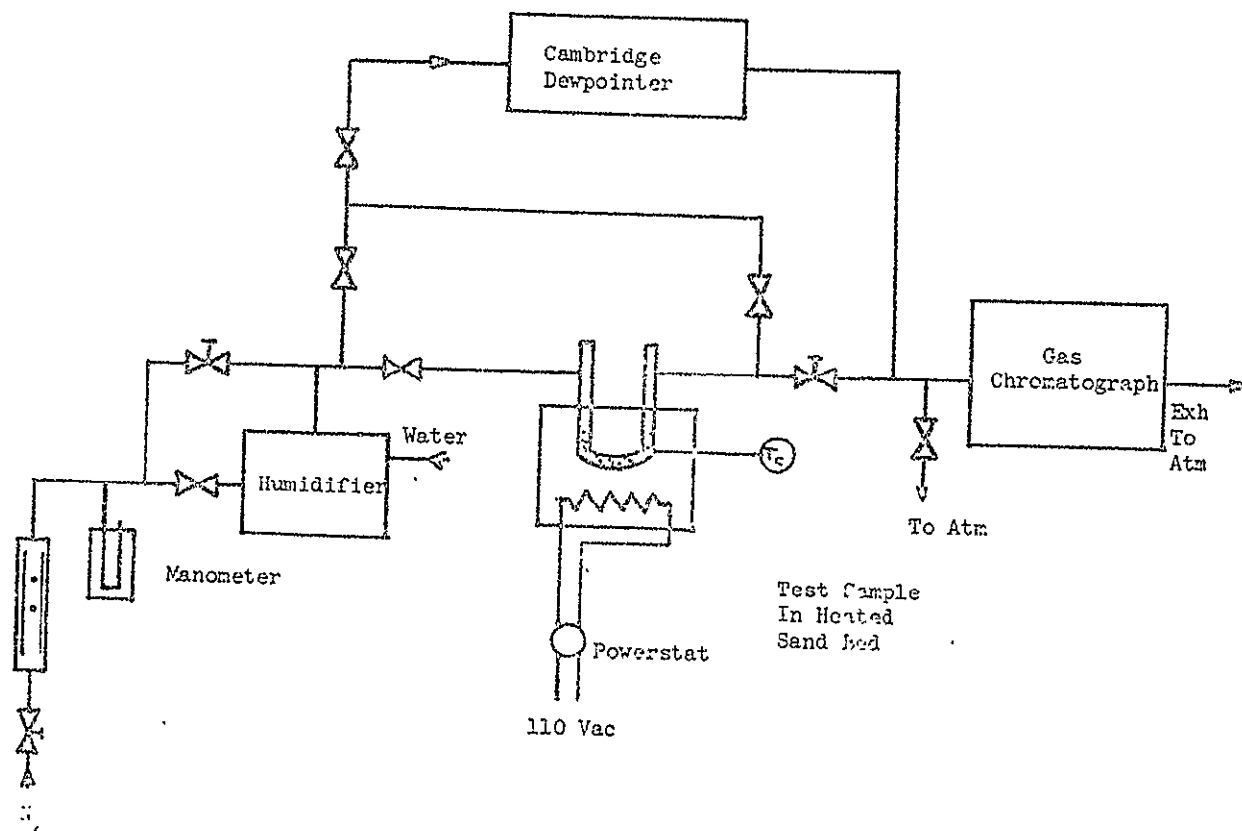


Figure 5-1-13



Lithium Peroxide Experimental Catalyst Test Rig

Figure 5-1-14

5.2

Bed Cooling EvaluationTest Canister Description

The initial phase of the Li_2O_2 cooling study consisted of the fabrication of two test canisters. Both canisters were similar in construction, both being rectangular in cross section and having water jackets on four sides with gas inlet and outlet on opposite ends. Canister 2 had internal conducting rods to act as heat transfer paths through the bed while canister 1 was designed to allow for installation of internal cooling coils. Sketches of the Li_2O_2 test canisters are given in Figures 5-2-1 and 5-2-2.

Passive Cooling

Passive cooling tests were run to simulate sublimator cooling by flowing 40°F water through the lower cooling face at a rate of approximately 270 lbs/hr. Test results for this cooling technique showed that low bed temperatures in the 300°F range could be maintained by passive cooling if the canister had internal conducting rods. In the canister without internal conducting rods bed temperatures reached 755°F. Heat removal rates for passive cooling with internal conducting rods were approximately 1500 BTU/hr. while the rate for passive cooling without internal rods was approximately 1300 BTU/hr. A summary of test data for passive cooling is given in Table 5-2-1.

Passive/Dynamic Cooling

Passive/dynamic cooling tests consisted of flowing water through all four sides of the test canister. The water temperature for these tests was 50°F and the flow rates were 20 lb/hr. through each side and 40 lb/hr. through both the top and bottom face. Passive/dynamic cooling with internal heat conduction rods maintained the bed temperature below 300°F with a maximum heat removal rate of 1100 BTU/hr. Passive/dynamic cooling without internal rods allowed bed temperatures to reach 520°F with a heat removal rate of 1160 BTU/hr. A summary of passive/dynamic cooling test data is presented in Table 5-2-2.

Dynamic Cooling

Dynamic cooling tests consisted of cooling the chemical bed by circulating water through copper coils in the bed. Various configuration coils, water flow rates, and water inlet temperatures were evaluated. Initial testing of coil configurations running parallel and perpendicular to the gas flow showed no difference in performance between parallel and perpendicular coils.



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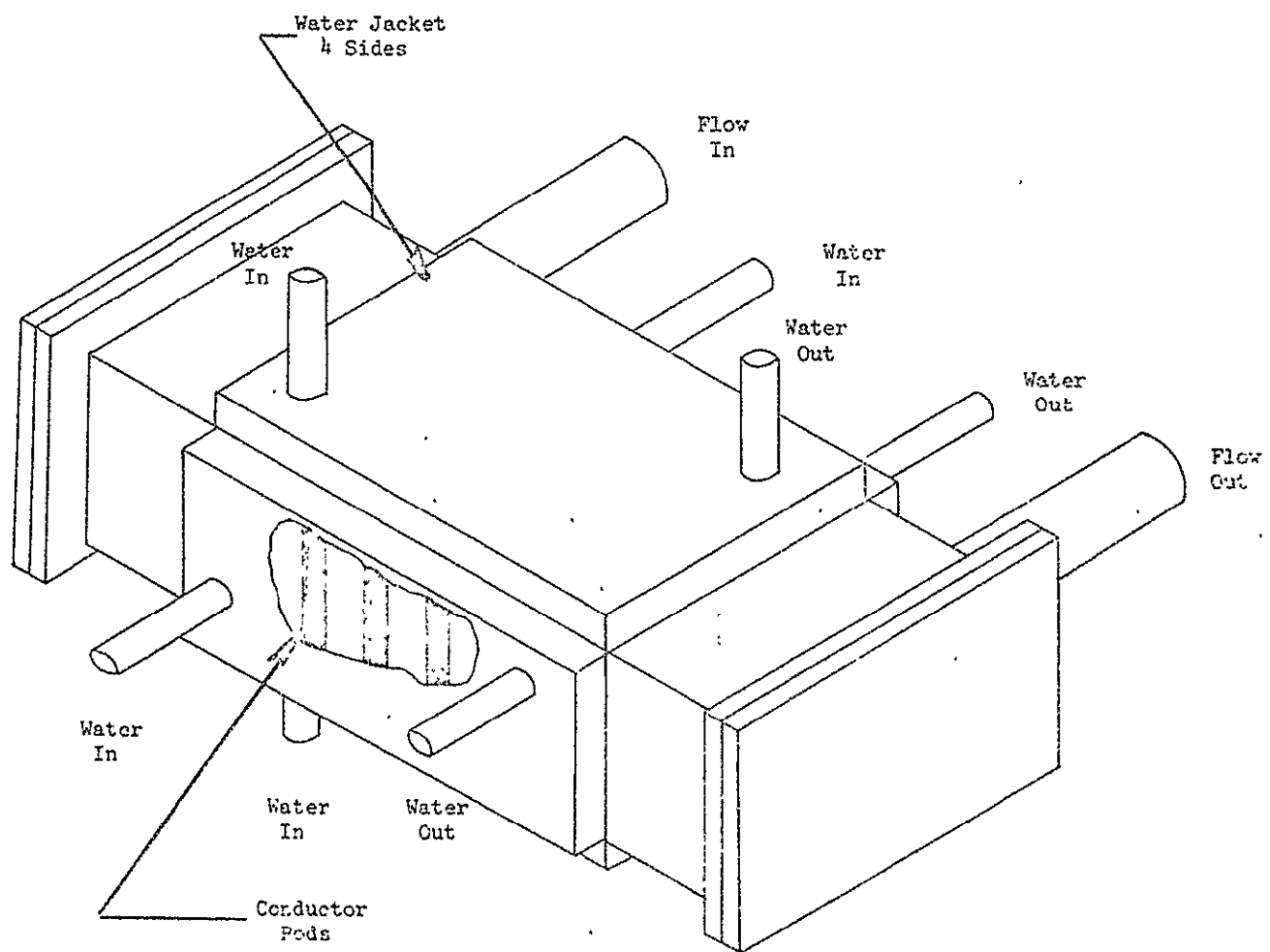
Tests With Internal Coils

PASSIVE ~ high flow rate into top water jacket only (water in (2)), 35-4 water
 PASSIVE/DYNAMIC ~ Water flow into all four water jackets (water in (1) & (2))

LYNAXIC ~ Water through internal coil

(water in (3))

Canister #1



Tests With Conductive Xerbers:
 1. High flow rate into top water jacket only, 2 °C water
 2. High flow rate into top water jacket only, 2 °C water

Cellator #.

Figure 5-2-2

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Passive/Dynamic Cooling Data Summary

<u>Test No.</u>	<u>Wt. Lbs.</u>	<u>Time (Min) To .5 - 1.0 - 4.0 mm Hg</u>	<u>Ave O₂ Lb./Hr.</u>	<u>Max. T °F</u>	<u>Heat Removal Rate BTU/Hr.</u>	<u>Internal Rods</u>
13B	4.64	140 - 160 - 240	.177	520	1160	No
13B	4.74	135 - 160 - 255	.113	376	957	No
14A	4.40	145 - 185 - 245	.074	275	1105	Yes
14B	4.83	175 - 205 - 275	.098	290	1068	Yes

TABLE 5-1-2

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5.1 Dynamic Cooling (Continued)

Testing was also conducted to evaluate variables in performance between coils having equal surface area but with a different number of passes through the chemical bed. This was done by testing a short 1/8" G.I. coil and a long 1/8" G.I. coil. Results showed bed temperatures in the 700°F range and heat removal rates of 1000 BTU/hr. for the 1/4" coil and temperatures in the 100°F range with heat removal rates of approximately 1 BTU/hr. for the 1/8" coil. These results supported the conclusion that the most important consideration in bed cooling is to distribute heat removal paths through the chemical bed. A summary of dynamic cooling test data is presented in Table 5-2-2.

Test Data Evaluation

The final conclusions reached in the thermal control study were based on the desirability to maintain low bed temperatures with a minimum system penalty, that is, a low heat removal rate. A penalty factor equal to the maximum bed temperature times the maximum heat removal rate divided by 10^5 was evaluated for each test. A low penalty factor represents a superior cycling technique. Penalty factors for each test are given in Table 5-2-4. Average values of maximum bed temperature, maximum heat removal rate, and penalty factor versus cooling technique are given in Figures 5-2-3, 5-2-4 and 5-2-5 respectively.

Test data from the Li_2O_2 cooling evaluation indicates that for any cooling technique employed, a means of heat transfer through the bed must be provided. Of the cooling techniques evaluated, dynamic cooling showed the best performance by maintaining a low bed temperature with the lowest heat removal rate penalty.

Dynamic Cooling Data Summary

<u>Test No.</u>	<u>Wt. Lbs.</u>	<u>Time (Min) To .5 - 1.0 - 4.0 mm Hg</u>	<u>Ave O₂ Lb./Hr.</u>	<u>Max. T °F</u>	<u>Heat Removal Rate BTU/HR.</u>	<u>Description</u>
15A	4.34	110 - 140 - 250	0.148	653	997	Baseline Conditions - Cools perpendicular to gas flow - low temperature coolant - 1/4" coils
15B	4.45	85 - 95 - 210	0.156	703	1079	
15C	4.64	90 - 120 - 245	0.137	760	1050	
16A	3.99	200 - 240 - 315	0.149	170	1200	Same as 15 except high temperature coolant 1/8" coils
16B	4.06	150 - 195 - 275	0.085	163	687	
17A	4.44	80 - 100 - 205	0.146	615	1010	Same as 15 except cools parallel to gas flow 1/4" coils
17B	4.55	105 - 150 - 285	0.133	700	767	
17C	4.20	145 - 180 - 265	0.124	330	685	
18A	4.47	155 - 170 - 238	0.075	175	871	Same as 17 except high temperature coolant and 1/8" coils
18B	5.24	225 - 260 - 350	0.109	210	773	
20A	4.0	115 - 160 - 215	0.113	235	978	Variable metabolic profile - 1/8" coils with high temperature coolant - 33 1/4" coils rate
20B	4.0	165 - 195 - 225	0.122	249	598	
20	4.0	147 - 191 - 205	0.113	226	1016	

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Cooling Technique Data Comparison

	TEST NUMBER	MAXIMUM TEE = A TEMPERATURE	MAXIMUM HEAT = P PERCENTAGE RATE	COOLING PENALTY FACTOR = $\frac{A \times P}{10^5}$	COMMENTS
Passive Cooling No Internal Rods	11A 11P	755 733	1339 1381	10.1 10.0	
Passive Cooling With Internal Rods	12A 12B	280 305	1056 901	2.96 2.75	
Passive/Dynamic Cooling No Internal Rods	13A 13B	520 376	1160 933	6.05 3.52	Thermocouple Error
Passive/Dynamic Cooling With Internal Rods	14A 14B	275 290	1105 1068	3.04 3.1	
Dynamic Cooling 1/4" Coil	15A 15B 15C 17A 17B 17C	653 703 760 615 700 330	997 1079 1050 1010 767 685	6.5 7.55 8.0 6.2 5.4 2.26	Dewpoint Variation ↓
Dynamic Cooling 1/8" Coil	16A 16B 18A 18B 19 20A 20B 20C	170 163 175 210 210 235 249 226	1200 687 871 773 738 978 898 1016	2.04 1.12 1.52 1.62 1.55 2.29 2.24 2.29	Dewpoint Error Variable Metabolic Rate ↓

TABLE 5-2-4

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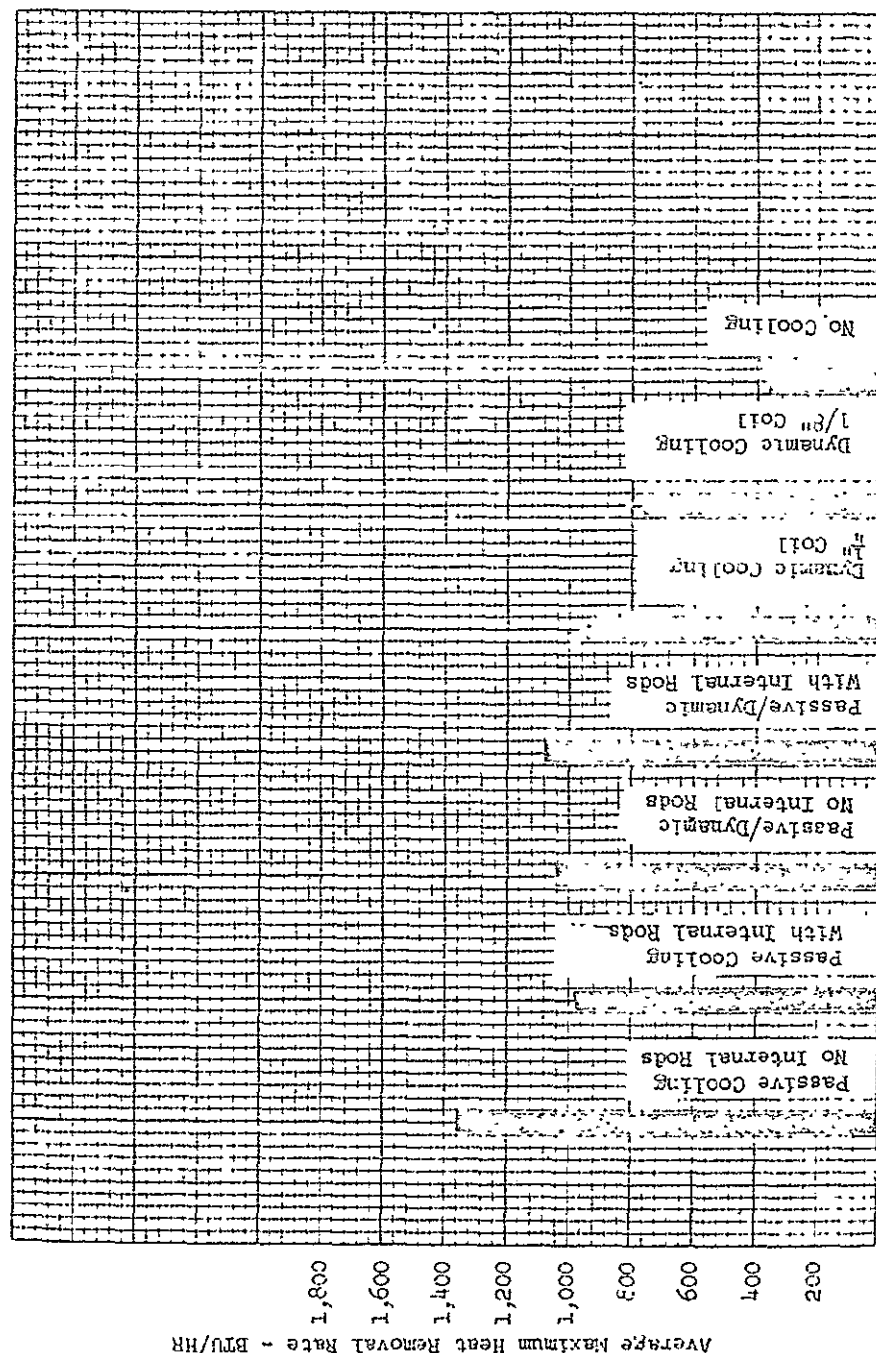
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Average Values of Maximum Red Temperature



100
90
80
70
60
50
40
30
20
10
0

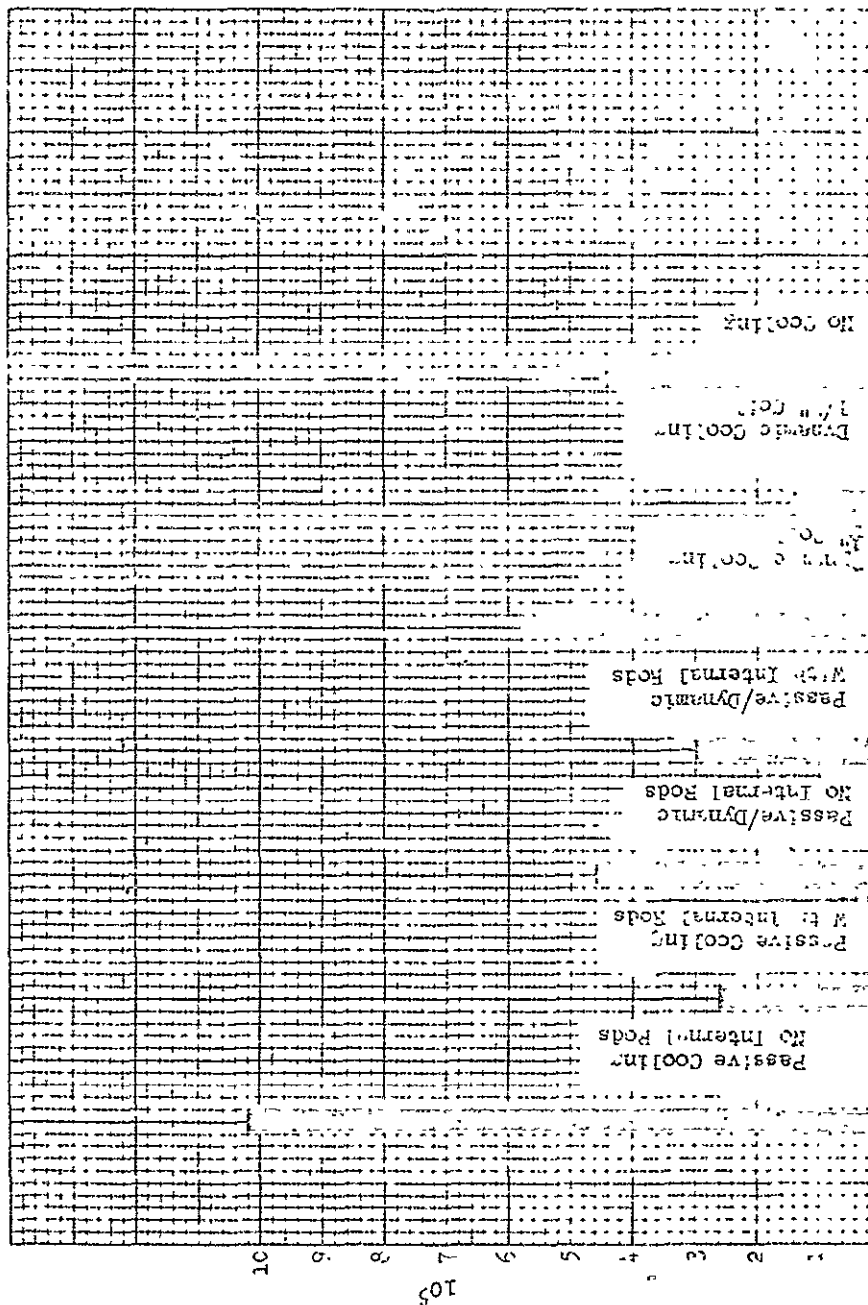
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Average Maximum Heat Removal Rate - BTU/HR

Figure 5-2-4
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Average Values of Penalty Factor versus Cooling Technique



5.3 Procedural TestsCanister Loading Process

A series of six tests was conducted to establish the optimum technique for loading a lithium peroxide canister. These tests also provided the information necessary for the development of the canister loading specification which is presented in Section 7.0.

Two tests were conducted on beds loaded at each of three distinct vibration levels. Vibration was applied in a transverse mode using a pneumatic ball vibration which was attached to the canister flange. The vibration levels were controlled by adjusting the supply pressure of the dry nitrogen used to operate the vibrator. Although the vibrator supply pressure was monitored and used as a relative indicator of vibration frequency, it was found that the desired packing could be best obtained by direct observation of the relative particle motions.

The catalyst used in all tests was manganese oxide. The selected cooling flow rate resulted in bed temperatures below the optimum range of operation for manganese oxide. Although the performance is not representative of the best available using manganese oxide, the same cooling rate was maintained for all loading tests. This was done in the interest of preserving constant test conditions so a relative comparison of loading techniques could be made. Tests MSC2-25A and 25B used a canister bed length of 2 7/8". The bed length for the remaining loading tests was reduced to 2 1/2 inches to provide a chemical bed weight below four pounds.

Test results show that insufficient or excessive vibration decreases the packing density of the bed with subsequent degraded CO₂ performance. The best performance was obtained for densely packed beds.

It was observed that desirable packing is attained when the lithium peroxide granules tumble uniformly and slide slowly to occupy voids in the bed. If the vibration frequency is too high a boiling effect of the particles is observed where voids are maintained due to the rapid particle motion. Insufficient vibration results in a failure of the particles to move adequately to occupy existing voids.

The evaluation of loading techniques can be made by observing the differences in CO₂ performance, better performance being the result of higher packing density where the particle diffusion characteristics are improved and the chemical weight per unit volume is increased. The other performance parameter, oxygen generation, is essentially the same for each of the six tests.

5.3 Canister Loading Process (Continued)

Test performance results for this test series is summarized in Table 5-3-1.

Tests MSC2-27A, 27B and 26A, 26B represent chemical beds of the same volume. The noticeably lower bed weights of tests 27A and 27B indicate a poorer packing density resulting from a vibration level which is too low. Figure 5-3-1 shows the degraded CO_2 performance which results. Tests 25A and 25B were loaded at high vibration frequency. The bed weights are higher and the CO_2 performance is better than the other canister loading tests. The chemical bed volume, however, was also higher and on an equivalent volume basis, the performance is less desirable than that shown for tests 26A and 26B.

The following conclusions were drawn from the test series conducted to evaluate canister loading techniques. The optimum loading vibration frequency is that frequency which corresponds to a state, observed visually, where the lithium peroxide granules tumble uniformly and slide slowly to occupy voids in the bed. Excessive vibration, observed as a boiling effect of the particles, or insufficient vibration, indicated by failure of the particles to move to occupy voids, were found to result in lower packing densities and degraded performance. Although the optimum loading vibration frequency may change with the canister and cooling configuration employed, a good packing density may be obtained for any configuration by observation of the granules and adjustment of the vibration frequency to produce the desired particle motion.

Manufacturing Process

A series of eight tests was conducted using lithium peroxide manufactured in accordance with the process specification presented in Section 7.0. These tests provided a performance evaluation of lithium peroxide made in two distinct granule sizes and with two different inert binders. Two tests were conducted for each combination of granule size and binder material.

The maintenance of constant cooling conditions for these tests as explained in relation to the canister loading series, results in a similarity of oxygen generation performance. Carbon dioxide removal performance is therefore used as the parameter for evaluating the effects of granule size and binder. The results of the test series are given in Table 5-3-2. Figure 5-3-2 presents test data for tests 21A, 21B, 23A, 23B, 26A and 26B. Tests 26A and 26B were conducted under baseline conditions using the original binder material and are presented for comparison.

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Comparison of Catalyst Loading vs. Test Performance

<u>Test No.</u>	<u>Wt. Lbs.</u>	<u>Time (Min.) To .5 - 1.0 - 4.0 mm Hg</u>	<u>Avg O₂ Lb./Hr.</u>	<u>Max. T °F</u>	<u>Vibrator Supply Press. psi</u>
25A	4.60	96 - 135 - 229	.108	277	20
25B	4.40	125 - 156 - 231	.103	312	20
26A	4.02	126 - 156 - 214	.102	245	10
26B	3.96	121 - 156 - 213	.098	250	10
27A	3.64	90 - 109 - 153	.083	300	5
27B	3.56	102 - 116 - 156	.076	290	5

TABLE 5-3-1.

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Comparison of Catalyst Granular Size vs. Test Performance

<u>Test No.</u>	<u>Wt. Lbs.</u>	<u>Time (Min.) To .5 - 1.0 - 4.0 mm Hg</u>	<u>Ave O₂ Lb./Hr.</u>	<u>Max. T °F</u>	<u>Granule Size & Binder Material</u>
21A	4.10	164 - 204 - 271	0.083	410	Baseline conditions Granule Size A Binder X
21B	4.04	124 - 170 - 244	0.081	332	Baseline conditions Granule Size A Binder X
23A	3.44	102 - 161 - 169	0.067	255	Repeat 21 except Granule B
23B	3.43	84 - 97 - 158	0.066	240	Repeat 21 except Granule B
26A	4.02	127 - 155 - 214	0.102	245	Baseline Conditions except original binder
26B	3.96	121 - 156 - 213	0.098	250	Baseline Conditions except original binder

TABLE 5-3-2

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Figure 5-3-1

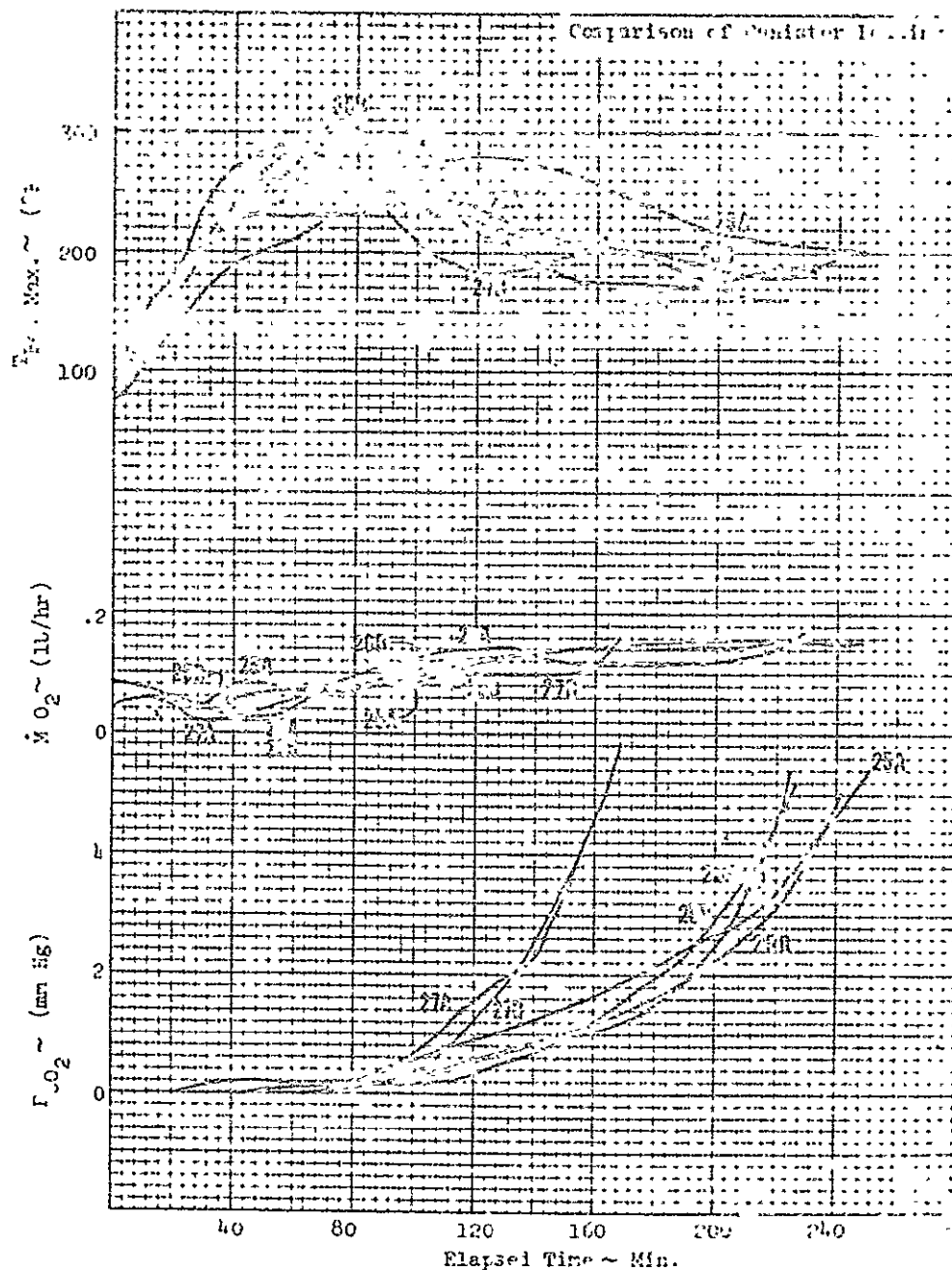


Figure 5-3-1

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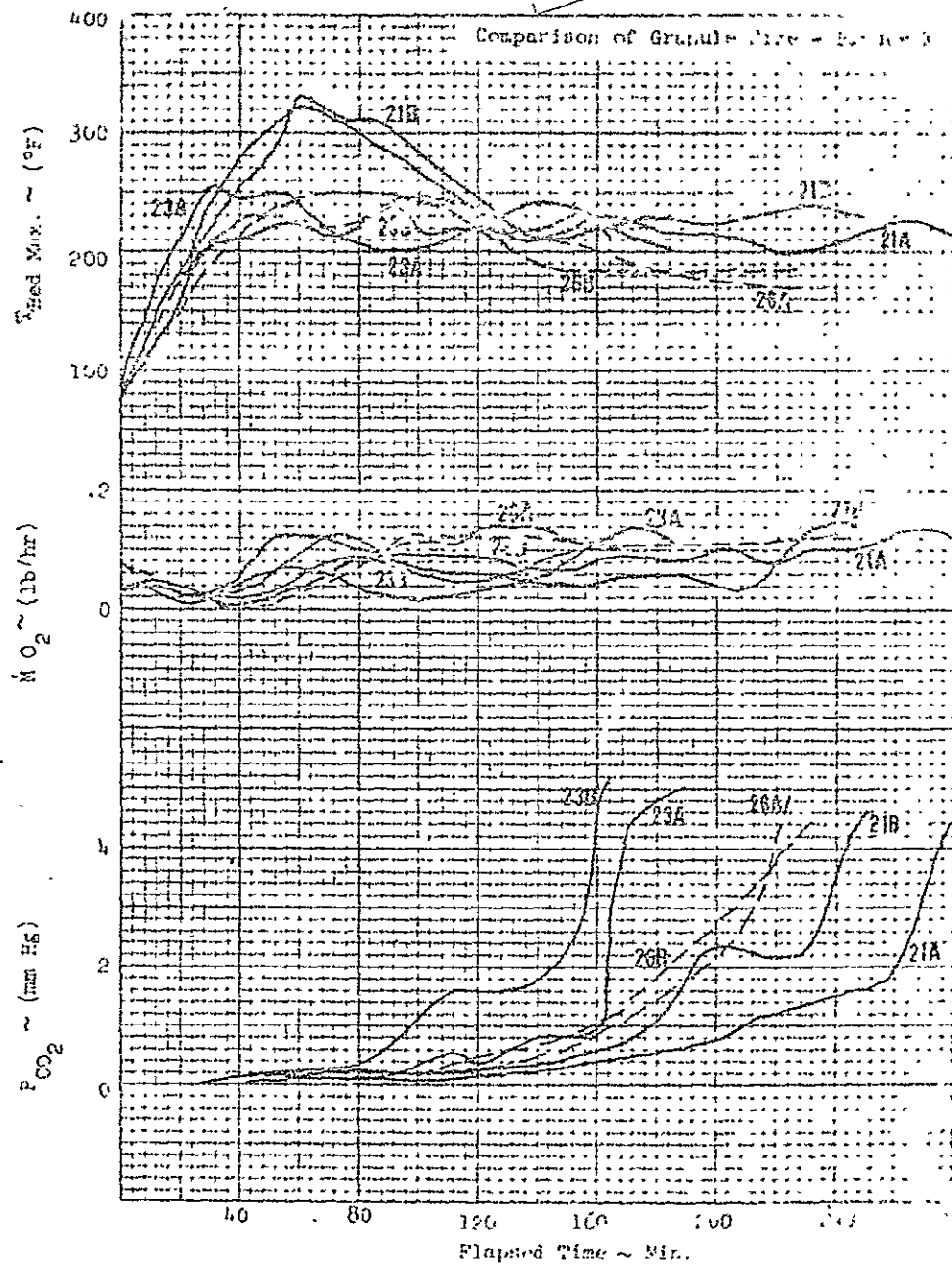


Figure 1-1

4.3 Manufacturing Process (Continued)

The binder material used for test series 21 and 23 provided the best performance of the two new materials evaluated. On a relative basis with respect to metabolic rate, however, the original binder material exhibits better CO₂ adsorption characteristics with improved performance repeatability. In addition to better performance, granules made from the original binder material were harder and had less tendency to pulverize during handling and while being subjected to loading vibration.

The granule sizes evaluated were 16 and 8 mesh; the latter being the mesh size of the granules used for all previous testing. Figure 5-3-2 gives a comparison of the two granule sizes made with the first binder material and Figure 5-3-3 gives the same comparison for granules made with the second binder material. The apparent bulk density for both granule sizes, based on the loaded bed weight, is essentially the same (25 lb/ft³). It should be noted that the lower bed weight of tests 23A and 23B is the result of the shorter bed length.

From these tests it is apparent that the smaller granule size provides the best CO₂ adsorption capabilities. The improved performance of the smaller granules can be explained in terms of the larger active surface area exposed. The effect is somewhat analogous to the results obtained in the Phase I testing where low bulk density, higher surface area granules were found to result in increased CO₂ adsorption. The advantages of high surface area have been explained previously with relation to the catalyst evaluation tests. The use of smaller granules can therefore be considered a desirable step toward increasing the performance characteristics of lithium peroxide.

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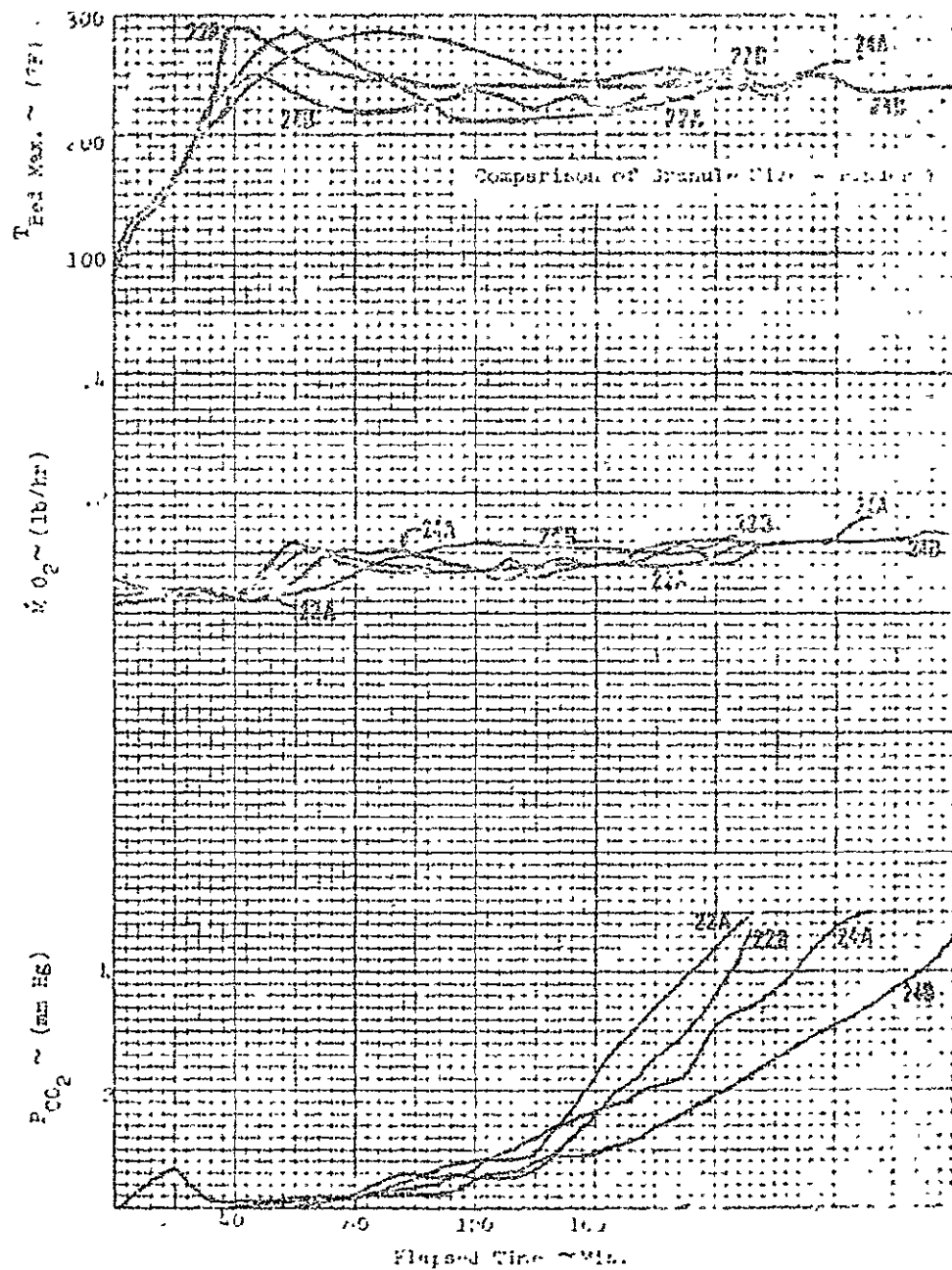


Figure 4-3

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Off Design Evaluation

The following series of tests was conducted to evaluate the effect of water vapor, carbon dioxide, and total flow rate variation on the performance of lithium peroxide. Additionally, these tests provided a better understanding of the chemical and physical processes occurring during bed operation.

Inlet Water Vapor Variation

Tests were conducted under three water inlet conditions. These included inlet conditions where the gas stream water content was zero, 50°F dew point, and 85°F dew point. The results are shown graphically in Figure 5-4-1. Test 28 which had zero inlet water was run for one hour during which time the effluent CO₂ partial pressure immediately exceeded the maximum scale of the analyzer; bed temperature remained constant with little O₂ production. A 50°F dew point test, 29A, was run using the same chemical. The drying effect on the chemical bed from the zero water test caused the effluent CO₂ partial pressure to immediately exceed the maximum scale. After a short period of operation a sufficient water concentration was reached within the bed to allow CO₂ adsorption to take place. A second 50°F dew point test, 29B, was run to provide performance results for a "fresh" chemical bed. Similar results were observed indicating that at low inlet water conditions a period of time is required for sufficient water to enter the bed to allow CO₂ adsorption to occur. The performance advantages and the importance of water in promoting those reactions which result in improved performance are apparent from the results of the 85°F dewpoint test. The higher water concentration increases the direct reaction of lithium peroxide with water vapor resulting in increased production of lithium hydroxide.



The more favorable molar volume ratio created by the hydroxide intermediate results in better CO₂ adsorption. Since the maximum bed temperature profiles are similar, there is no significant difference in the oxygen generation for the 50 and 85°F dewpoints. It is important to note that although decreasing the dewpoint degrades CO₂ performance, it does not impair the ability of the bed to reach temperatures high enough to provide favorable oxygen generation. It is preferable from an overall performance standpoint, however, to operate at high dewpoint conditions.

Inlet CO₂ Variation

Four tests, the results of which are shown in Figure 5-4-2, were conducted to evaluate the effects of inlet CO₂ variation on lithium peroxide performance. Test 31 was run with zero CO₂ until an elapsed time of 180 minutes. At this point the CO₂ flow rate was set at a rate comparable to 1600 BTU/hr. With the inlet gas containing water vapor at a 70°F dewpoint and

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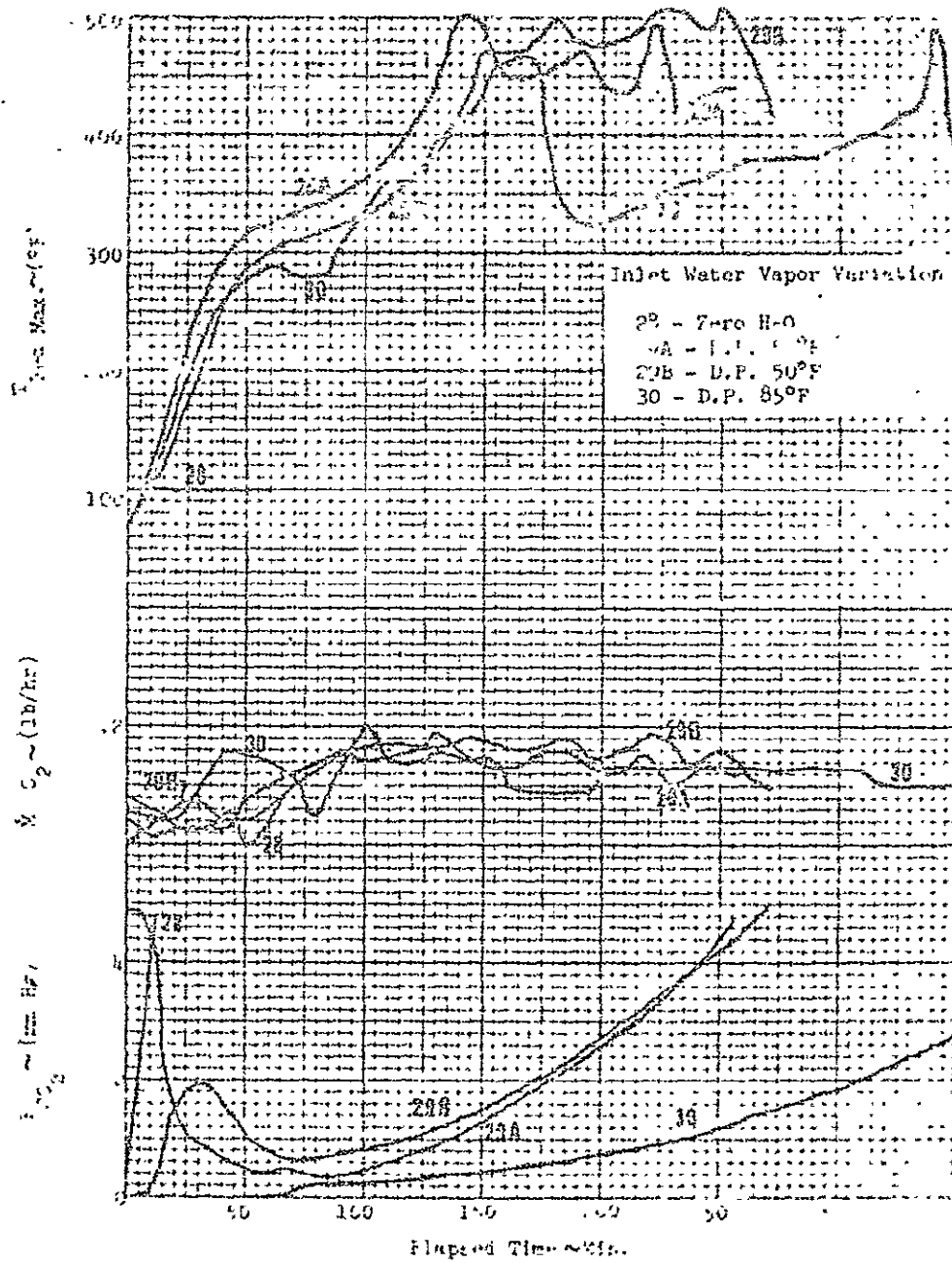


Fig. 1-4-1

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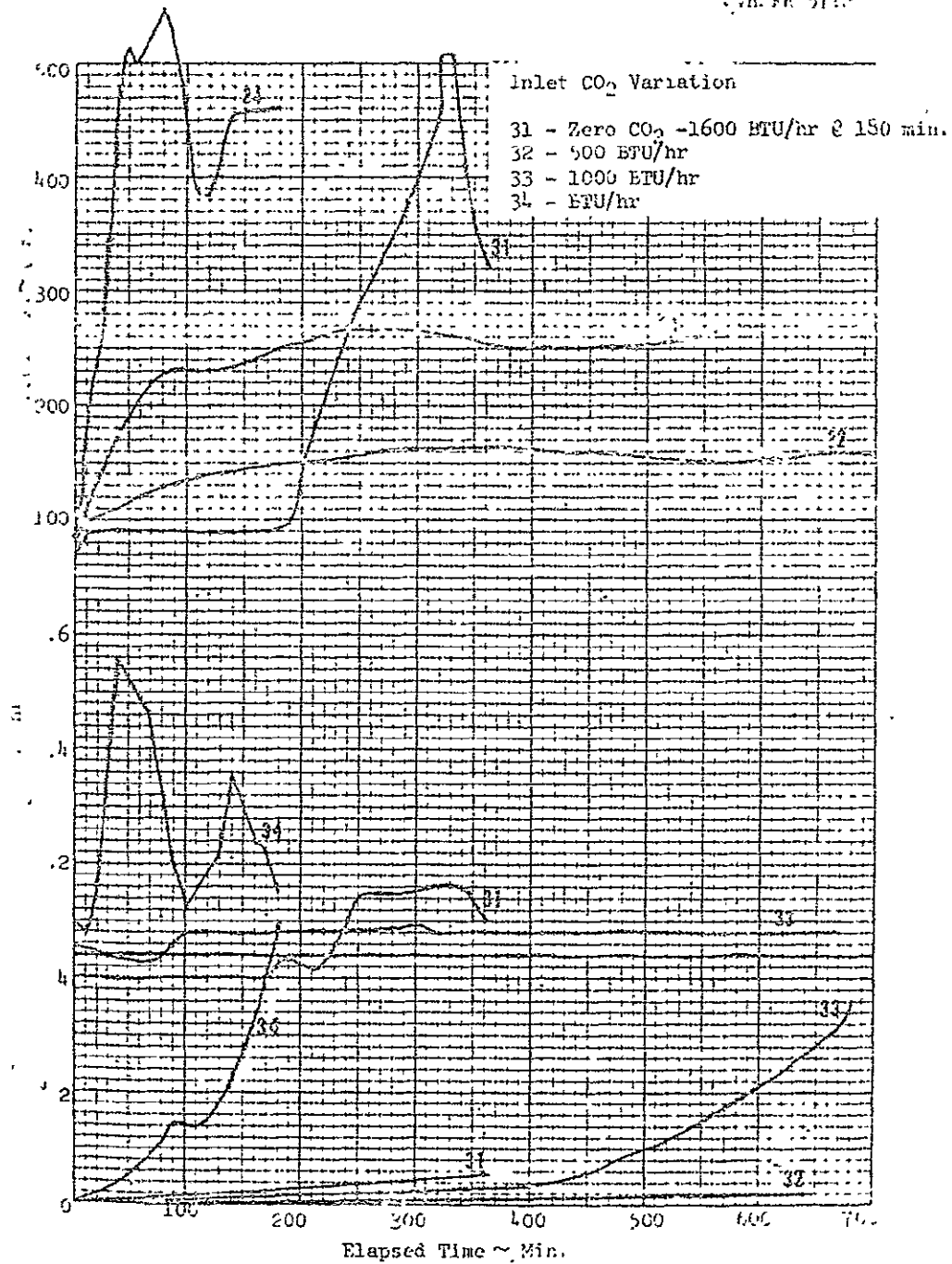


Figure 5-4-2

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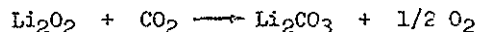
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5.4 Off Design Evaluation (Continued)

CO₂, no reaction was observed between the water vapor and lithium peroxide. The bed temperature remained constant at about 400°F and no oxygen generation occurred.

For this series of tests the results clearly show the expected decrease in CO₂ performance with increasing metabolic rate. The important performance results of the CO₂ variations, however, are shown by the oxygen generation and maximum bed temperature curves; these curves being directly related to the amount of influent CO₂. The effect of increasing the CO₂ flow rate is to increase the direct reaction of CO₂ with lithium peroxide.



Higher oxygen generation and bed temperatures result.

The high sensitivity of bed temperature and oxygen generation to the inlet CO₂ concentration or metabolic rate is a characteristic which demands prime consideration in effecting the system configuration design. For lithium peroxide catalyzed with manganese oxide it has been shown that operation at bed temperatures above 400°F is essential for good overall performance. The CO₂ inlet variation series, however, indicates that the crewman's metabolic rate must be above 12 or 1300 BTU/hr. in order to provide a CO₂ flow rate sufficient to attain the temperature required. The sensitivity of bed temperature on metabolic rate indicates that optimum performance must employ a variable cooling configuration. A constant cooling configuration may be used at the cost of some off design operation. It is with respect to this problem that the use of ferrous sulfate as a catalyst appears desirable. Ferrous sulfate, an active catalyst with optimum performance at high cooling rate, low bed temperature conditions would provide overall performance almost equivalent to manganese oxide. The high cooling rate employed would result in a decrease in bed temperature sensitivity to metabolic conditions. A constant cooling configuration could be used. The final catalyst selection should therefore be made with respect to the complexity of conforming the system to the imposition made by the individual catalyst characteristics.

Total Flow Rate Variation

Two tests, MSC2-35 and 36, were conducted to evaluate the effect of total gas flow rate on lithium peroxide performance. Test number MSC2-35 was run at a flow rate of 5 ACFM while test MSC2-36 was run at 9 ACFM. The nominal gas flow rate for lithium peroxide testing is 7 ACFM.

5.4 Off Design Evaluation (Continued)

Results for this test series are presented in Figure 5-4-3. As seen from the CO₂ partial pressure curves, the CO₂ performance did not vary appreciably with total flow rate; the total amount of CO₂ removed and test duration being approximately the same for both tests.

The most pronounced effect of total flow rate variation is on the thermal operating characteristics of the chemical bed. Both tests were run with no internal cooling. Bed temperatures reached during low flow rate test MSC2-35 were over 500°F while the maximum bed temperature obtained during the high flow rate test was 350°F. This indicates that the high flow rate stabilizes the bed operating characteristics by transferring heat through the bed and eliminating local hot spots. It appears desirable then to operate at as high a flow rate as possible to obtain the maximum convective heat transfer through the chemical bed.

End Item Canister Evaluation

A new lithium peroxide canister was fabricated and tested to demonstrate the feasibility of a cooling technique for a lithium peroxide canister which would allow recharging of the canister assembly without the making or breaking of liquid cooling lines. This test canister was delivered as a contract end item at the completion of testing.

The canister assembly, shown in Figure 5-5-1, has internal dimensions equivalent to the present PLSS canister reservoir. Cooling of the reservoir is accomplished by coolant flow through the external coil assembly which in turn cools the canister wall. A coolant flow rate of 200 lb/hr. was maintained during testing with a coolant temperature of 50°F.

The cartridge assembly, shown in Figure 5-5-2, consists of a modified PLSS cartridge assembly. The end flange was modified to provide a new internal seal configuration and allow the use of a stronger handle. Twelve radial beryllium copper fins which run the length of the cartridge and extend from the center of the cartridge out through the cartridge wall were added. The outer ends of the radial fins were spring tempered so they would maintain contact with the canister wall when the cartridge was installed in the canister. Heat is transferred from the bed through the internal fins and is then transferred through the canister wall to the coolant flow loop.

The test objective for this configuration was to remove enough heat to maintain the chemical bed in the desired operating range. This operating range extends from 290°F to 430°F depending on the catalyst material and the desired operating characteristics of the chemical.

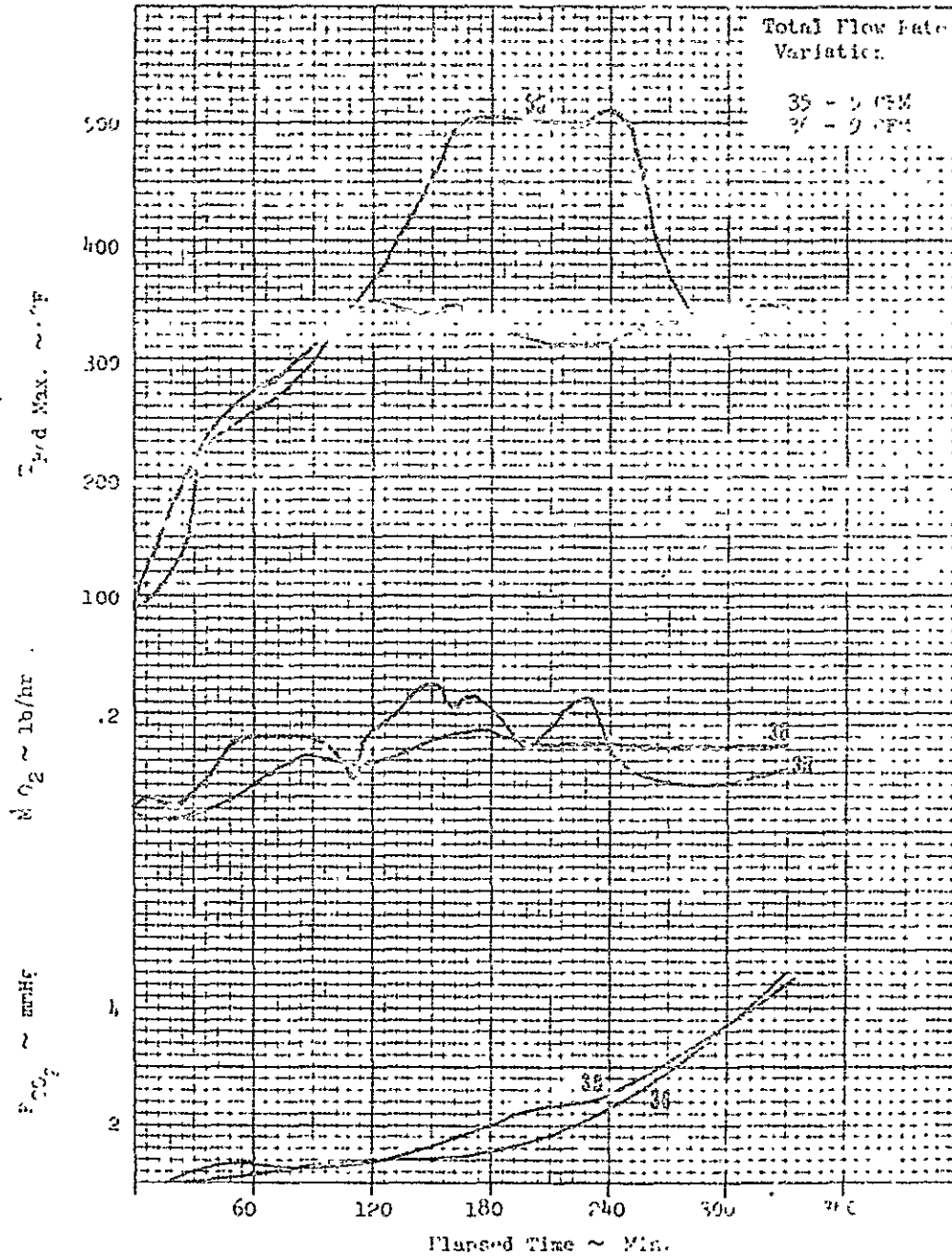


Figure 5-4-3

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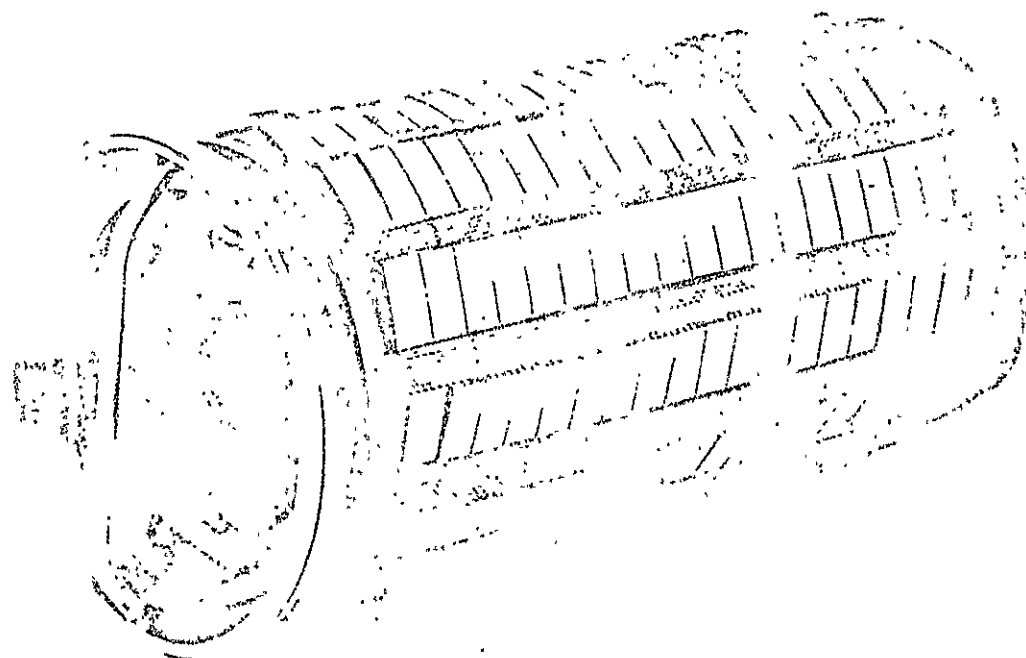
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Prototype Cooling Canister
(Canister #4)

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Prototype Cooling Cartridge
(Canister #4)

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End Item Canister Evaluation (Continued)

A total of seven tests was run during this series. It was demonstrated that at a maximum coolant flow rate of 200 l/hr. the bed temperature could be maintained below 325°F with a heat removal rate of 600 BTU/hr. The test cartridge was also run in a six fin configuration. The maximum bed temperature was 390°F with a heat removal rate of 600 BTU/hr. at maximum cooling flow for this configuration. Test data for this test series is presented in Table 5-5-1. The data verifies that the cooling concept which was tested is capable of removing sufficient heat to maintain the chemical bed at the desired operating temperature.

Very poor chemical bed utilization was encountered during testing. At first it was assumed that the poor bed utilization resulted from flow channeling along the radial fins. Several different loading techniques were tried to improve bed utilization but these were unsuccessful. In a further attempt to isolate the problem, spoilers were placed along each fin so that flow could not channel along the fin surface. Since this did not improve performance channeling along the fins was eliminated as the cause of poor bed utilization.

During testing it was observed that the pressure drop through the canister was only 2 mm Hg. This low pressure drop is not believed sufficient to provide adequate flow distribution through the chemical bed. It results from the running of lithium peroxide in a canister originally designed for lithium hydroxide with its inherently lower pressure drop requirement. This poor flow distribution is believed to be the main cause of the poor bed utilization noted during these tests.

A second related deficiency associated with using a modified PLSS canister results from the short bed length¹ and small flow area of the radial flow configuration. Previous Li₂O₂ testing has shown that poor bed utilization has resulted when bed lengths less than 2.5 inches are used. This probably results from the poor flow distribution associated with low pressure drop. In the PLSS cartridge used for these tests the bed length was 2.0 inches. Better bed utilization could be obtained using the same cooling configuration by running flow thru the cartridge axially rather than radially.

¹ Length as used here refers to the length of gas travel through the granule packed bed.

Canister Evaluation Test Summary

<u>Test No.</u>	<u>Wt. Lbs.</u>	<u>Time (Min.) To .5 - 1.0 - 4.0 mm Hg</u>	<u>Ave. O₂ (Lb/Lr)</u>	<u>Max. T °F</u>	<u>Heat Removal Rate BTU/Hr.</u>
37A	3.09	8-13-73	0.044	225	677
37E	3.11	30-47-89	0.071	182	693
38A	2.91	12-18-42	0.0375	150	551
38B	2.57	18-----	0.085	115	476
39C	3.78	7-12-40	0.041	175	537
39A	2.57	3- 8-82	0.096	390	663
39B	2.185	21-23-37	0.037	110	451

TABLE 1-1

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Li₂O₂ System Penalty Evaluation

In order to evaluate the total system penalty associated with using a lithium peroxide system, a comparison was made between a Li₂O₂/O₂ and LiOH/O₂ system. Test data used for comparison of the two systems was generated using the same test canister and test rig (Fig 21). The comparison is for a cooled LiOH or Li₂O₂ system and assumes the use of high pressure (7500 psia) oxygen.

The comparison is made for a four hour, 2000 PUF/hr. mission. This corresponds to a required oxygen supply rate of 0.5 lb/hr. and a CO₂ removal rate of 0.39 lb/hr. For a four hour mission a total of 1.44 lbs. of oxygen must be supplied and 1.56 lbs. of carbon dioxide must be removed.

The present development status of LiOH shows that for cooled LiOH the following utilization can be obtained: For a maximum allowable CO₂ outlet partial pressure of .5 mm Hg the utilization is $\frac{.40 \text{ lbs CO}_2}{1 \text{ lb LiOH}}$ and for a maximum allowable CO₂ outlet partial pressure of 4.0 mm Hg the utilization is $\frac{.6 \text{ lbs CO}_2}{1 \text{ lb LiOH}}$. This data is for the rectangular, axial flow canister with a maximum bed temperature maintained below 180°F.

The present development status for Li₂O₂ cooled at the rate of 350 BTU/hr. gives the following utilizations: For a maximum allowable CO₂ outlet partial pressure of .5 mm Hg the utilization is $\frac{.325 \text{ lb CO}_2}{1 \text{ lb Li}_2\text{O}_2}$ and for a maximum allowable CO₂ outlet partial pressure of 4.0 mm Hg the utilization is $\frac{.55 \text{ lb CO}_2}{1 \text{ lb Li}_2\text{O}_2}$. For a

Li₂O₂ system sized for CO₂ removal, an average usable oxygen generation rate of .18 lb/hr. can be obtained.

Using the above design parameters and considering only the components which will vary between a LiOH and Li₂O₂ system results in the following system comparison:

Outlet CO₂ partial pressure = 0.5 mm Hg

	LiOH/O ₂		Li ₂ O ₂ /O ₂	
	W (lb)	V (in ³)	W (lb)	V (in ³)
Chemical	3.9	--	4.8	--
Charcoal	0.36	--	0.36	--
Canister	2.20	390	2.20	4.
H ₂ O (Cooling)	1.90	--	1.15	--

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Fig. 1 LiOH system penalty evaluation (continued)

	LiOH/O ₂		Li ₂ O ₂ /O ₂	
	W (lb)	V (in ³)	W (lb)	V (in ³)
H ₂ O Tank	0.76	55	0.44	35
O ₂	1.44	--	0.72	--
O ₂ Tank	3.60	70	1.80	30
	<u>14.16</u>	<u>515</u>	<u>11.82</u>	<u>55</u>

Outlet CO₂ partial pressure = 4.0 mm Hg

	LiOH/O ₂		Li ₂ O ₂ /O ₂	
	W (lb)	V ² (in ³)	W (lb)	V (in ³)
Chemical	2.60	--	3.00	--
Charcoal	0.36	--	0.36	--
Canister	1.50	290	1.70	310
H ₂ O (cooling)	1.90	--	1.10	--
H ₂ O Tank	0.76	55	.44	35
O ₂	1.44	--	.72	--
O ₂ Tank	3.60	70	1.80	30
	<u>12.16</u>	<u>415</u>	<u>9.12</u>	<u>375</u>

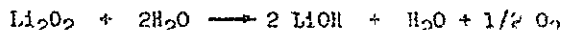
This data shows that the Li₂O₂/O₂ system is approximately 5.4 lbs. lighter and occupies the same volume as the LiOH/O₂ system for the 0.5 mm Hg CO₂ limit and is 3.0 lbs. lighter and 17.6 in³ smaller for the 4.0 mm Hg limit.

RECOMMENDATIONS

- 6.1 Three major areas requiring further development have come to light as a result of the present lithium peroxide test program. These are further catalyst study, the evaluation of the use of water vapor as a catalyst to oxygen production, and further study of test canister configuration.

6.1.1 Catalyst Evaluation

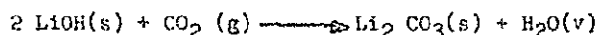
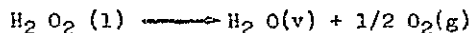
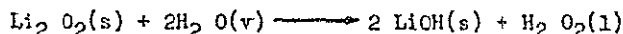
Present catalyst testing has not isolated a material which promotes oxygen evolution at low bed temperatures. These catalysts should promote oxygen generation through the water vapor reaction:



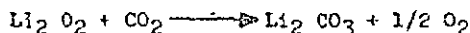
To determine this catalyst material, testing should be conducted similar to the small scale testing which was initiated during this phase of the lithium peroxide study. This test series should isolate a catalyst material which initiates the release of oxygen in the presence of water vapor at a temperature below 100°F.

6.1.2 Water Vapor Catalyst Approach

Present test results show a direct relation between lithium peroxide performance and inlet dewpoint condition. This suggests that chemical performance could be improved greatly by going to a very high inlet dew point condition. For a high dew point inlet condition it appears that the water vapor acts as a catalyst to oxygen generation through the reactions:



The net result for this process is:



with no net loss of water to the system.

A system could be built where the inlet air stream is conditioned to a very high dew point condition (100° - 150°F dew point) to increase the water vapor reaction. A condenser could then be used to reclaim the water vapor in the system for recycling. This would provide higher oxygen evolution and good CO₂ removal in a way similar to catalyst addition.

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6.2.1 Canister Configuration Testing

Flow distribution problems encountered during the present canister evaluation indicate that better flow distribution is needed utilizing a. To achieve this, the present radial flow cartridge should be modified to axial flow.

6.2 Test Program

The proposed test program which has a sum total of 114 tests, is composed of the following set of test series.

6.2.1 Catalyst Evaluation - Small scale tests will be conducted on fifty (50) different catalyst materials in order to identify catalyst which produce oxygen generation for use in the future reactions at low temperatures.

A total of 100 tests, two per catalyst material, will be run. These tests will consist of flowing conditioned oxygen through catalyzed lithium peroxide. Oxygen evolution will be measured as a function of chemical bed temperature for each catalyst material with the objective of finding a catalyst material which will provide 100% conversion of lithium peroxide to lithium hydroxide at low temperature through the reaction:



6.2.2 High Dew Point Catalyst Evaluation - Nine (9) full scale tests will be run to determine if a high dew point system can be designed to increase lithium peroxide performance.

Testing will consist of evaluating the performance of lithium peroxide run at 100°, 125°, and 150°F dew point and with maximum bed temperatures of 200°, 350°, and 600° for each of the three dew points evaluated.

6.2.3 Canister Configuration Evaluation - A total of five (5) tests will be run during this series. The present cooling evaluation cartridge will be reworked to run in an axial flow mode and the cooling evaluation tests conducted during the end item canister evaluations (Section 3.7) will be repeated.

7.0 APPENDIX

7.1 Specification for Lithium Peroxide (Li₂O₂) Manufacture

7.1.1 Scope

This specification establishes the process requirements for the manufacture of catalyzed granular lithium peroxide for environmental use as a carbon dioxide adsorbent and oxygen source. Care must be exercised because of the toxicity of lithium peroxide as well as maintaining cleanliness and contaminant free conditions.

7.1.2 Requirements

The manufacturing process shall take place in an inert dry argon environment to minimize atmospheric contamination of the lithium peroxide.

7.1.2.1 Equipment

The following equipment shall be required:

- A. Dry argon chamber (0°F dew point or less)
- B. Granulator
- C. Mixing container
- D. Drying oven (equipped with dry argon purge)
- E. Sifting screen (Standard No. 8 mesh)

7.1.2.2 Materials

The following materials shall be required:

- A. Virgin lithium peroxide powder (minimum Li₂O₂ content of 97.0%)
- B. Inorganic inert binder
- C. Catalyst - manganese oxide (MnO)
- D. Anhydrous methyl alcohol
- E. One gallon, screw top, wide mouth, polyethylene bottles
- F. Polyethylene bag liners
- G. One inch vinyl tape

7.1.3 Required Procedures and Operations

7.1.3.1 The amount of lithium peroxide granules to be made in each process batch shall be limited to approximately one (1) pound to preserve safe manufacturing conditions.

7.1.3.2 The granulator, mixing container, and specified materials shall be placed in the dry argon chamber.

- 7.1.3.3 The lithium peroxide powder, inert binder and catalyst shall be thoroughly mixed in the mixing container to provide a homogeneous powder mixture of the following proportions by weight:

Lithium peroxide powder	93% min.
Catalyst	$2.0 \pm 0.1\%$
Inert binder	$5.0 \pm 0.2\%$

- 7.1.3.4 Anhydrous methyl alcohol shall be blended into the powder mixture until a thick slurry is formed.

- 7.1.3.5 The slurry shall be processed through the granulator which shall force the mixture through a Standard No. 8 mesh screen. The granules are then formed by cutting the slurry extruder to a length of 3/16 of an inch.

- 7.1.3.6 When the entire slurry has been granulated, the particles shall be removed from the argon chamber and placed on drying trays in the argon purged oven. A maximum of ten (10) one (1) pound batches may be processed and placed in the oven before the drying operation is begun.

- 7.1.3.7 The drying process is initiated by turning the oven on and maintaining 120°F to 140°F to evaporate the methyl alcohol processing aid.

- 7.1.3.8 The drying process shall be maintained until the moisture content of the granules is less than one (1) per cent by weight. (Approximately 8 to 10 hours). The termination of drying shall be determined when a random 2 to 3 gram sample of particles taken from the drying trays exhibits less than a one (1) per cent weight change when subjected to a high temperature oven (180°F to 190°F).

- 7.1.3.9 On completion of drying, the granules shall be placed in the argon chamber where any fine particles or dust shall be sifted away. The liners shall be placed in the polyethylene bottles and purged with argon before filling with lithium peroxide granules. When each bottle is filled the liner shall be sealed closed with vinyl tape. The bottle cap shall be screwed on tightly and sealed by wrapping circumferentially with vinyl tape.

7.2 Specification for Lithium Peroxide (Li₂O₂) Storage

7.2.1 Scope

This specification establishes the requirements for the storage of granular lithium peroxide to minimize chemical degradation over prolonged periods of time.

7.2.2 Requirements

General Requirements

The chemical shall be packaged and stored in an area which shall have a controlled environment suitable for maintaining to the fullest extent the original chemical properties.

7.2.2.1 Detail Requirements

Packaging

- a. The chemical shall be packaged in a dry argon environment in one (1) gallon, screw cap, wide mouth, polyethylene bottles.
- b. The chemical in each bottle shall be contained in a polyethylene bag liner which shall be purged with dry argon and sealed with one (1) inch vinyl tape.
- c. The bottle screw cap shall be tightened and sealed to the container by circumferential wrapping with one (1) inch vinyl tape before removal from the dry argon atmosphere.
- d. Weight of the chemical shall not exceed five (5) pounds per bottle.

7.2.2.2 Storage Environment

- a. The chemical storage area shall be maintained between 60-80°F.
- b. Relative humidity of the storage area shall not exceed 40 percent.
- c. The storage area shall have temperature and relative humidity indicators which shall be monitored daily.
- d. Exposure of the chemical to direct sunlight shall be prevented.
- e. The chemical shall be kept remote from combustible materials, water and steam.

7.2.3 Handling

Exposure of the chemical to an uncontrolled environment, while being moved between controlled areas, shall not exceed twenty four (24) hours in duration. When exposed to an uncontrolled environment, the following limitations are in effect.

- a. The chemical bottles shall be packaged in cardboard containers sealed with masking tape with no more than four (4) bottles per carton.

7.2.3 Handling (Continued)

b. Vibration of the chemical shall be held to a minimum.

7.2.4 Shipment

The chemical shall be shipped in a temperature controlled vehicle to maintain exposure within the specified limitations.

7.2.5 Receiving

The chemical shall not be exposed to a receiving area whose environmental conditions do not satisfy the storage area specifications for more than eight (8) hours.

7.3 Specification for Canister Charging Lithium Peroxide (Li_2O_2)

7.3.1 Scope

This specification establishes the requirements and procedures to be followed when charging a lithium peroxide canister. Care should be exercised when charging the canister because of the toxicity of lithium peroxide as well as maintaining cleanliness and contaminant free conditions.

7.3.2 Requirements

7.3.2.1 Equipment

The following equipment shall be required:

A. Nitrogen chamber equipped with:

1. Purge system (using dry nitrogen gas - 0° dew point or less)
2. Vacuum cleaning system
3. Purge hose with adapters as required
4. Loading fixture with pneumatic vibrator

B. Li_2O_2 canister

7.3.2.2 Materials

Those materials listed below shall be required:

- A. Li_2O_2 chemical
- B. Non-waxed paper cup

7.3.3 Required Procedures and Operations

- 7.3.3.1 Weigh and record the weight of the unopened Li_2O_2 containers to the nearest 0.01 lb.

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- 7.3.3.2 Place the equipment and materials of paragraph 2.3 and 2.7 in the nitrogen chamber.
- 7.3.3.3 Attach nitrogen purge hose to pneumatic vibrator.
- 7.3.3.4 Close chamber door and initiate flow of nitrogen through chamber. Maintain flow for 10 ± 2 minutes before opening Li_2O_2 containers to allow chamber to approach dry nitrogen conditions.
- 7.3.3.5 Fill the canister approximately one third ($1/3$) full of Li_2O_2 by pouring granules directly from polyethylene container. Use steel rod to level particles assuring even distribution.
- 7.3.3.6 Turn on pneumatic vibrator and adjust nitrogen supply pressure to increase or decrease the vibration frequency as required to obtain good packing of Li_2O_2 . Good packing may be attained when the Li_2O_2 granules tumble uniformly and slide slowly to occupy voids in the bed. Do not permit the Li_2O_2 bed to assume a boiling effect during vibration loading.
- 7.3.3.7 Allow canister to vibrate for 10 ± 1 minutes.
- 7.3.3.8 Load remaining two one third ($1/3$) sections while the canister is vibrating; allowing 10 ± 1 minutes of vibration after each one third section is loaded.
- 7.3.3.9 Close canister and seal inlet and outlet flanges with tape.

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