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**Hamilton
Standard**

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DIVISION OF UNITED AIRCRAFT CORPORATION

CENTRAL WASTE PROCESSING SYSTEM

BY

FRANK L. KESTER

JUNE 1973

PREPARED UNDER CONTRACT NO. NAS 9-12730

BY

HAMILTON STANDARD

DIVISION OF UNITED AIRCRAFT CORPORATION

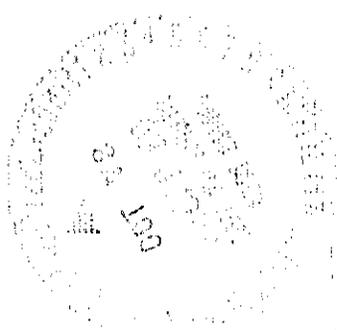
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FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

LYNDON B. JOHNSON SPACE CENTER

HOUSTON, TEXAS 77058



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ABSTRACT

CENTRAL WASTE PROCESSING SYSTEM

CONTRACT NO. NAS 9-12730

JUNE 1973

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FRANK L. KESTER

A new concept for processing spacecraft type wastes has been evaluated. The feasibility of reacting various waste materials with steam at temperatures of 538° - 760°C (1000° - 1400°F) in both a continuous and batch reactor with residence times (reciprocal of space velocity) from 3 to 60 seconds has been established. Essentially complete gasification is achieved. Product gases are primarily hydrogen, carbon dioxide, methane, and carbon monoxide.

Water soluble "synthetic" wastes are readily processed in a continuous tubular reactor at concentrations up to 20 weight percent.

The batch reactor is able to process wet and dry wastes at steam to waste weight ratios from 2 to 20. Feces, urine, and "synthetic" wastes have been successfully processed in the batch reactor.

A mathematical model of the type employed to correlate coal gasification rate data has been successfully extended to batch waste gasification.

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FOREWORD

This report has been prepared by the Hamilton Standard Division of United Aircraft Corporation for the National Aeronautics and Space Administration's Johnson Space Center in accordance with Contract NAS 9-12730, Central Waste Processing System.

Appreciation is expressed to the NASA Technical Monitors, Mr. Rex B. Martin and Mr. Ralph Drexel for their guidance and advice.

Hamilton Standard personnel responsible for the conduct of this program were Mr. F. H. Greenwood, Program Manager, and Mr. F. L. Kester, Program Engineer. Appreciation is expressed to Mr. J. S. Lovell, Chief, Advanced Engineering; Mr. V. Binks, Analytical Engineer; Mr. W. Coe, Assistant Project Engineer; and Mr. F. Sribnik, Analytical Engineer, whose efforts made the successful completion of this program possible.

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SUMMARY

A new concept for processing spacecraft wastes has been tested and evaluated. The feasibility has been established for the process of chemically reacting a variety of organic waste materials with steam at elevated temperatures.

Essentially complete gasification is achieved and product gases are primarily hydrogen, carbon dioxide, methane, and carbon monoxide.

Water soluble wastes such as methanol, glycine, and sucrose were readily processed through a 2.27×10^{-4} cubic meter (227 cm³) continuous reactor at weight concentrations in water up to 20 percent. Reactor contact times were 3 to 60 seconds at temperatures 538° - 700°C (1000° - 1400°F). Typical product gas composition was 45 percent hydrogen, 20 percent carbon dioxide, 5 percent methane, and 30 percent carbon monoxide.

A large-scale continuous reactor was designed and fabricated using data generated from the small-scale tests. After the slurry-feed system experienced severe reactor plugging, the continuous concept was abandoned in favor of a batch feed reactor with continuous steam flow. The continuous test rig was then modified to accept batch samples. The final configuration had two temperature zones and a volume of 4.183×10^{-4} cubic meter (4183 cm³).

The batch reactor was able to process wet and dry wastes at steam to waste weight ratios from 2 to 20. Feces, urine, paper, polyethylene plastic, cellulose, and a protein were processed in this dual-temperature-zone reactor. Wastes were gasified in a low temperature 200° - 538°C (500° - 1000°F) "gasifier" reactor and then converted to simple gases in a higher temperature 650° - 700°C (1200° - 1400°F) "converter" reactor. Product gas generation was very rapid once "ignition" had occurred, usually at temperatures between 260° - 430°C (500° - 800°F), depending on the chemical being tested and gasifier reactor heating rate. After ignition, gas generation rate drops rapidly. However, with some tests, the rate again increased as the sample was rapidly elevated to the converter temperature. Typical product gas composition was quite similar to that obtained in the continuous reactor tests. Water quality varied considerably with the test conditions. The highest quality water was obtained with 10 gram cellulose at a 4 second contact time (run 312). For this run, pH varied between 4.8 and 6.0 and electrical conductivity varied between 8 and 57 μ mho/cm, with product water clear/colorless and with almost no odor. Product water from a mixture of feces, urine, paper, and plastic (run 322) had a pH range of 2.2 to 7.8, electrical conductivity 20 to 10,000, and a cloudy yellow color and a slight odor.

Three types of tests were conducted with the batch reactor: "Synthetic" waste tests, Spacecraft waste tests, and Special tests. The "Synthetic" waste tests conducted on pure cellulose, methionine, or polyethylene yielded parametric data. Correlation

of the parametric data was possible for both the gasifier and the converter reactors. A kinetic model which has found success in coal gasification studies has been employed to describe the gasifier reactor. The converter reactor has been found to be readily correlated with a space velocity model for a continuous reactor.

The spacecraft wastes tests conducted on mixtures of fresh, untreated feces, urine, paper, and plastic established the feasibility of processing real wastes by steam reformation. The addition of a 5 percent by weight sodium carbonate catalyst promoted a 31.8 percent increase in the carbon gasified. Also, the addition of a 5 percent by weight ruthenium chloride catalyst lowered the effluent ammonia level from 106.0 grams/cubic meter of condensate (106 μ grams/ml) to 55.0 grams/cubic meter of condensate (55 μ grams/ml).

Special tests were conducted on polyethylene and cellulose in which gas products were sampled directly from the gasifier. Water was shown not to be of major importance in the gasification of polyethylene; however, water did enhance the gasification of cellulose.

It is recommended that research into the fundamental chemistry of waste gasification be investigated. In particular, the role of water vapor in the gasification and conversion process should be evaluated. Employment of a thermogravimetric apparatus to give simultaneous weight loss with analysis of gases generated will enable generation of detailed reaction rate data. It is also recommended that an overall mathematical model incorporating mass and heat transfer with reaction rate data be developed. Catalysts should be investigated to improve water and gas quality. Also, the possibility of extension of the basic process to municipal and agricultural waste gasification should be evaluated.

INTRODUCTION

The problem of safe handling and storage or reuse of spacecraft waste materials is one which must be addressed prior to proceeding with the long duration manned space missions planned for the future. Crew metabolic wastes, food wastes, and all other solid and liquid wastes must either be reclaimed to minimize vehicle penalties or stored in a reduced weight and volume condition, which also precludes safety hazards posed by the growth of microorganisms.

Included in the many schemes now under investigation for providing these conditions is the concept of steam reformation of organic waste materials. This candidate process is believed to offer potential for future missions because it performs many of the functions of both water and waste reclamation subsystems in a low pressure chemical reactor.

Steam reformation was originally developed as a process to product hydrogen from hydrocarbons in the chemical industry during the early 1930's, but has only recently been investigated for use in reducing organic waste materials to basic chemical compounds and elements. In a steam reforming waste processing system, the wide variety of organic compounds (hydrocarbons, carbohydrates, proteins, etc.) present are reacted with steam at atmospheric pressure at 540° - 760°C (1000° - 1400°F). The reaction produces product gases, which must be used in other spacecraft subsystems to eventually product oxygen and potable water. A sterile inorganic ash remains which represents a reduction in weight and volume of the original solid waste of approximately 40 to 1.

This process is in an early stage of definition for spacecraft application and many aspects require investigation. Among these are the equilibrium which results as a function of temperatures, the dependence of contact time upon temperatures, the processing technique for handling both soluble and insoluble waste materials, and the type of process which would be most suitable for spacecraft use (batch, continuous flow, combinations of both, etc.). It was the purpose of this program to develop an understanding of the process and to define equipment functions for a preliminary system design through analyses and tests with small and full-scale sized laboratory apparatus.

CONCLUSIONS

The results of this investigation to appraise the concept of gasifying and chemically reforming typical spacecraft waste materials to simple gases have led to the following conclusions:

- The feasibility of converting typical spacecraft waste materials to useful or more suitable forms by heating to elevated temperatures in steam was demonstrated.
- A batch feed reactor with continuous steam flow was the most successful test configuration. It consisted of a "dual temperature" reactor arrangement with a gasifier temperature of 260° - 371°C (500° - 700°F) and a converter temperature of 700°C (1400°F). A reactor contact time of about 12 seconds was most practicable.
- Sodium carbonate (catalyst) mixed directly with the feed enhanced the rate of carbon gasification.
- Ruthenium trichloride (catalyst) mixed directly with the feed lowered the ammonia level in the product gas.
- Water vapor is vital to the desired manner and rate of gasification of cellulose, but is of minor importance in the gasification of polyethylene.
- Gasification rate data was successfully correlated with a kinetic model adapted from coal gasification studies.
- A method for slurrifying and continuously feeding insoluble solid wastes into the reactor was not successfully developed.

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RECOMMENDATIONS

The recommendations listed below are derived from the analytical studies, test data, and the evaluation of the results of this program.

- The basic chemical mechanism of the gasification and conversion processes should be investigated. Specifically, it is recommended that:
 - High temperature thermogravimetric apparatus should be used in order to measure weight loss simultaneously with product gas generation. More quantitative data could be obtained in this manner and interpretation of the data could yield information on the basic chemical reactions occurring including the rates of these reactions.
 - Parametric tests should be conducted to obtain information on the effect of temperature, water vapor partial pressure, and space velocity on the reaction rates to enable derivation of appropriate rate equations.
- A quantitative mathematical model including heat and mass transfer and kinetic relationships should be developed to describe the process occurring in both the gasification and conversion phases of the process.
- An investigation should be conducted into the use of catalysts to improve the gasification phase of the process.
- An investigation should be conducted into the use of catalysts to improve the quality of product gases and water.

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PROCESS CHEMISTRY

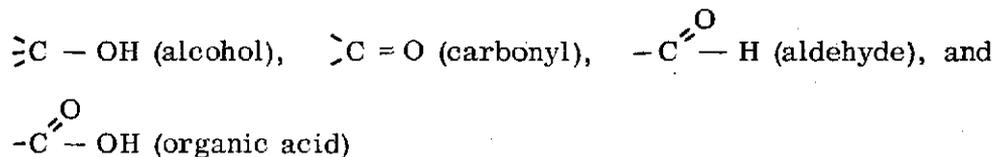
The purpose of this section is to provide some insight into the chemistry of waste gasification; however, a detailed description of all of the complex reactions occurring in both the continuous and batch reactors during the gasification and conversion processes would indeed be a very difficult task and was beyond the scope of this contract.

Organic waste materials of all types should be expected to show high reactivity to steam at elevated temperatures. This has been observed for cellulose, methionine, polyethylene, paper, feces, and urine in the batch tests, and in the continuous tests with sucrose, glycine, and methanol. Minerals remain behind as ash. Halogens are converted to acid gases and condensed in product water. Nitrogen also ends up in product water as ammonia. Sulfur compounds are found to be converted to hydrogen sulfide gas, and mercaptans (organic sulfides). The organic fraction is converted to hydrogen (H₂), carbon dioxide (CO₂), methane (CH₄), carbon monoxide (CO), and other simple gases depending on reaction conditions.

It was observed that cellulose, when heated in the absence of steam, generates some gases (water vapor and other simple gases) and a lot of char. When heated with steam, more gases are generated, with the char produced slowly gasified. If the flow exiting from the gasifier is directed into a heated reactor (the converter) and held at high temperatures for a number of seconds, even more gases are generated. Similarly, sucrose when introduced into the continuous reactor at a low feed rate is gasified rapidly, producing no char. However, at a high feed rate sucrose produces char, which can ultimately plug the reactor.

OVERALL CHEMICAL REACTIONS

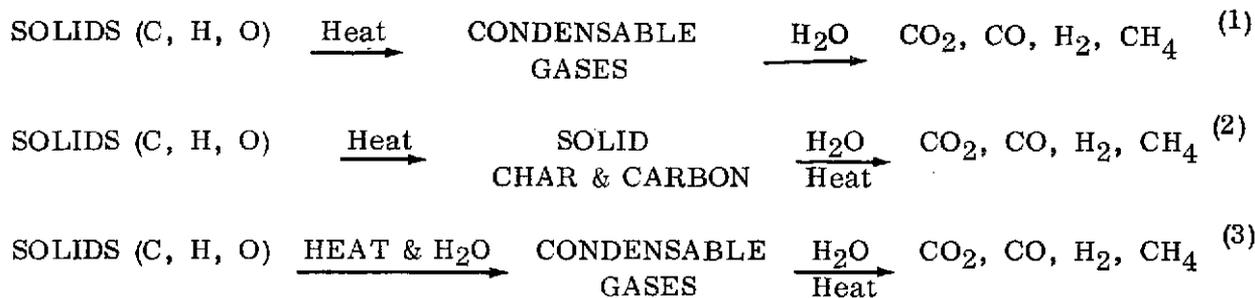
It is apparent that cellulose and sucrose can be described according to equation (1) as solids of a carbon, hydrogen, and oxygen reacting with heat only, producing what is termed a condensable gas. This condensable gas could contain reactive intermediates with chemical structures somewhere between complex sugars and simple gases. Typical chemical structures may contain:



groups in their chemical make-up. As these gases are passed through the heated converter, they react further with the water producing large quantities of H₂, CO₂, CO, and CH₂. Also, the char that is produced, equation (2), reacts with water at an elevated temperature to generate the simple gases. As water is shown to enhance the gas generation rate from the gasifier, reaction (3) is suggested.

The overall chemical reactions occurring can be summarized as follows:

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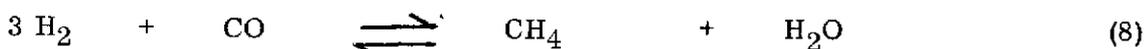
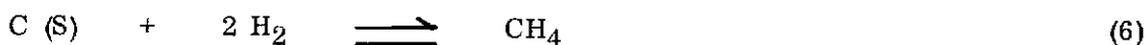
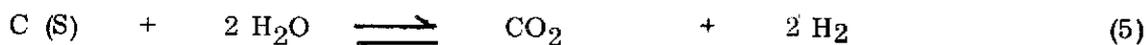
It was found that when heated in the presence of steam but in the absence of the converter, polyethylene generated no non-condensable gas, although sample weight loss was 100 per cent. (See Test Number 319, Page 54.) When heated in the converter in place, large quantities of simple gases were generated. These facts suggested reaction path (1). The reaction intermediates called condensable gases probably represent various stages of decomposition or cracking of the hydrocarbon structure. Polyethylene was not heated in the absence of steam. However, it is known to melt and gasify readily under these conditions.

Methionine and glycine were found to char but not as readily as cellulose. Presumably, their reaction paths are similar to the sugar.

Real wastes probably follow some or all of these and possibly other chemical routes to the final products.

DETAILED CHEMICAL REACTIONS

By analogy to char and coal gasification some of the following reactions may be important in waste gasification:



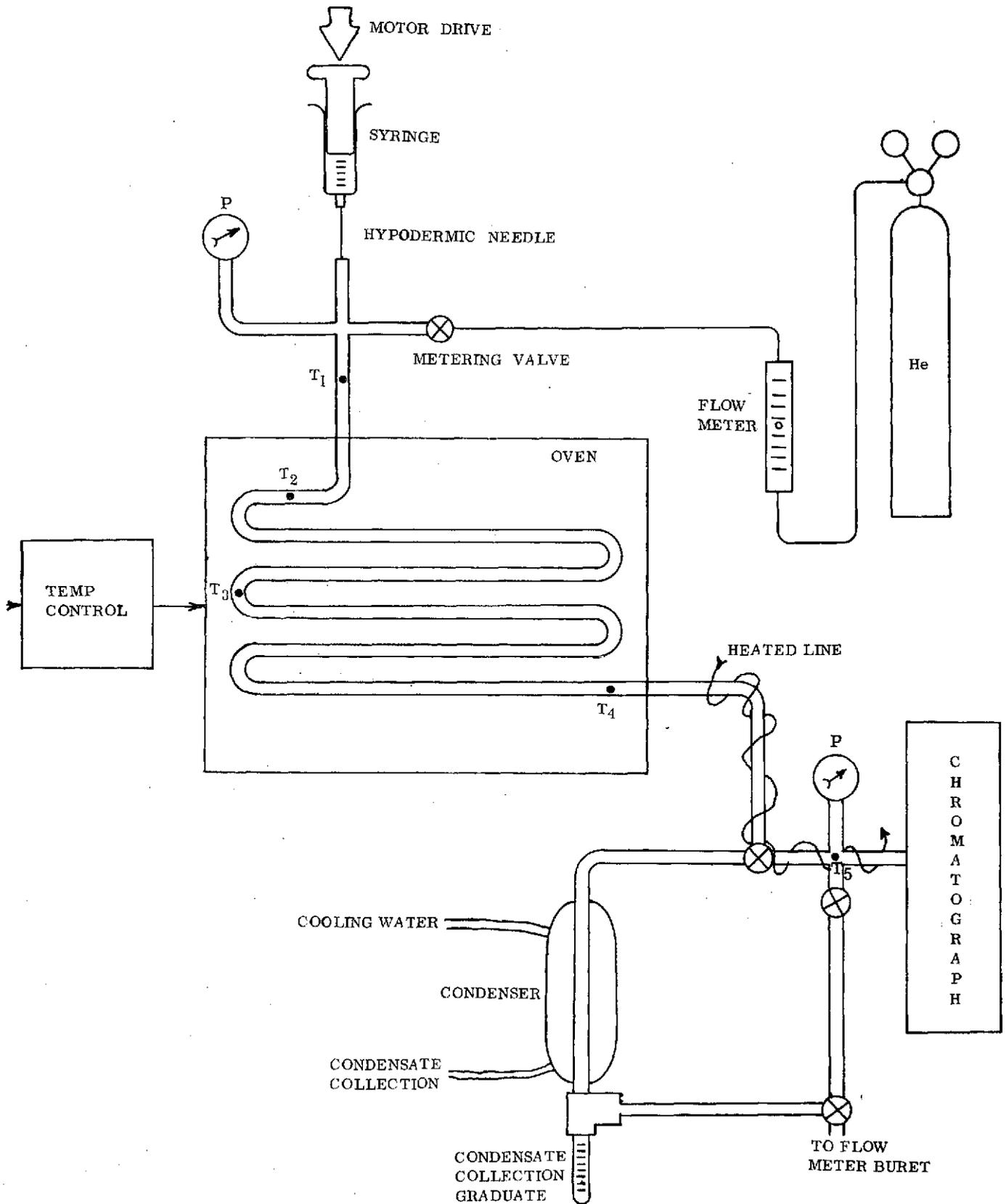
Reactions (4) and (5) are of importance in the steam gasification of coal and char and are probably of some importance as a source of CO and CO₂ in waste gasification. Reaction (6) is expected to be of only minor importance in that a high partial pressure of H₂ is needed to drive this reaction. For these tests, of course, the major gas is H₂O.

The last two reactions are of importance in the converter reactor. Reaction (7) is the well known "water gas reaction", which depending on condition of temperature and gas

partial pressures can generate quantities of H_2O and CO , or CO_2 and H_2 . The last reaction (8) may be of importance in generating the CH_4 observed. The extent of influence of these and other reactions on the final gas composition is a function of feed composition, reactor contact time, temperature and, of course, thermodynamic equilibrium.

Appendix A summarizes the equilibrium gas composition for various H_2O to waste ratios and for a number of waste materials, all as a function of temperature. This is the composition that would be expected to result if the mixture had an infinitely long time to react.

In practice, the composition observed was somewhat different than what was predicted. The gas concentrations that are greater than that predicted are probably due to the gasifier generating large quantities of perhaps CH_4 or CO , while the converter conditions, including residence time, do not favor reaching equilibrium before product recovery.



TEST APPARATUS SMALL SCALE
FIGURE I

SMALL SCALE TESTS - CONTINUOUS REACTOR

The objective of this task was to identify and evaluate the significant parameters of processing spacecraft wastes into simple usable gases with steam at high temperatures. A continuous reactor was selected to study the parameters because of its ease in data collection and interpretation.

Operation of the continuous reactor at steady state conditions offers precision in measuring feed flow and gas generation rates. Once steady state is achieved, interpretation of data is easy and straightforward, especially when collected at isothermal conditions. The space velocity model is used in data interpretation.

Space velocity is defined as the rate of steam flow (\dot{V}_{H_2O}) plus gas generation rate (\dot{V}_{GEN}) divided by the reactor volume (V_R), thus:

$$S_V = \frac{\dot{V}_{H_2O} + \dot{V}_{GEN}}{V_R}$$

contact or residence time (τ) is defined as reciprocal space velocity.

$$\tau = \frac{1}{S_V}$$

Plots of yield (carbon recovery) vs. contact time and temperature give a measure of reactor performance. A primary goal was to determine contact time and temperature giving the maximum yield. Here yield is defined as the maximum carbon recovered as CO₂ or CO product gases.

Candidates selected for evaluation were water solutions of glycine (NH₂CH₂CO₂H), sucrose (C₁₂H₂₂O₁₁), and methane gas (CH₄). A fourth chemical, methanol (CH₃OH), was used only for rig checkout and preliminary testing.

SMALL SCALE APPARATUS DESCRIPTION

The test setup used for the continuous reactor data collection consisted of a feed system, an oven/reactor, and analysis equipment. Figure 1 presents a schematic of the small-scale apparatus. The components employed in this testing as well as their range of use and precision of measurement are summarized in Table I.

Basically, the apparatus operates by feeding a water solution of known waste concentration at a constant rate into a reactor held at a constant temperature. As the water flash evaporates the waste gasifies and is then converted into simple gases carried along by the flowing stream. At the reactor exit, the water is condensed and the product gases are then sampled by the chromatograph and the total gas flow is measured with a gas buret.

TABLE I
SMALL SCALE TEST COMPONENTS

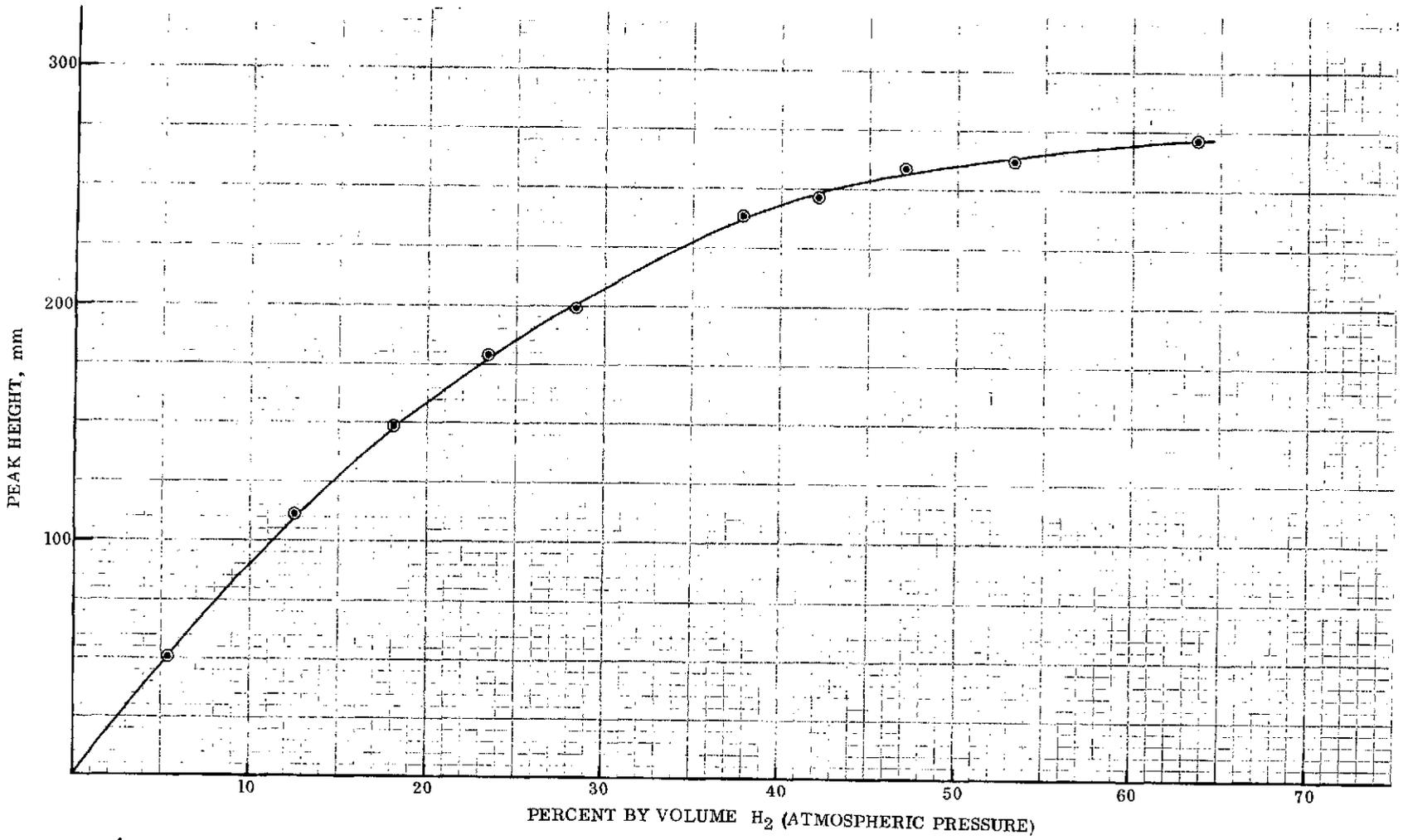
Feed System Components	Range of Measurement	Precision of Measurement
Sage Model 344 motor driven pump	2800 - 13,000 mm ³ /min (.0028 - 13 cm ³ /min)	±1% ±1%
Syringe	0 - 50,000 mm ³ /min (0 - 50 cm ³)	±1% ±1%
Flow Meter	0 - 30,000 mm ³ /min (0 - 300 cm ³ /min)	±5% ±5%
Oven/Reactor Components		
Reactor Coil, 347 Stainless Steel (.000635 meter (1/4") x 14.63 meter (48 feet))		
Sears L-15 Oven	24 - 1093°C (75 - 2000°F)	±14°C ±25°F
Leed & Northrup 8686-F Potentiometer with chromel alumel thermocouples	24 - 815°C (75 - 1500°F)	±8.3°C ±15°F
Analysis Components		
Glass Buret	0 - 50,000 mm ³ /min (0-50 cm ³)	±1% ±1%
Timer	0 - 1000 seconds	±1%
Bendix Model 6000 Process Chromatograph		
H ₂	0 - 65%	±5%
CO	0 - 70%	±5%
CH ₄	0 - 70%	±5%
CO ₂	0 - 60%	±5%

The chromatograph programmed for this test series was fitted with two 3.05 meter x 0.00318 meter (10 ft x 1/8 inch) inch stainless steel column, packed with Porapak "Q", in an oven temperature of 104°C (219°F) and helium carrier flow rate of 50 ml/min. The programming consisted of automatically sampling the stream flowing through the sample valve loop, eluting H₂, CO, CH₄, and CO₂ to a set of type W-2 thermal conductivity detectors through the two columns in less than 240 seconds (4 minutes), switching out the second column to elute H₂O from column one to the detector in less than 300 seconds (5 min.), backflushing the first column with helium to clean the column of any impurities, and at 480 sec. (8 min.) automatically introducing another sample for analysis. Individual detector signals were integrated and automatically gated, fed through a preset resistor pot (one for each constituent) and read out as a bar height on a Texas Instruments strip chart recorder. The bar height is a direct function of the partial pressure of the constituent in the gas stream. This measured height was fed into a data computer program for processing. Calibration was accomplished as outlined in the section "Calibration" which follows.

Calibration

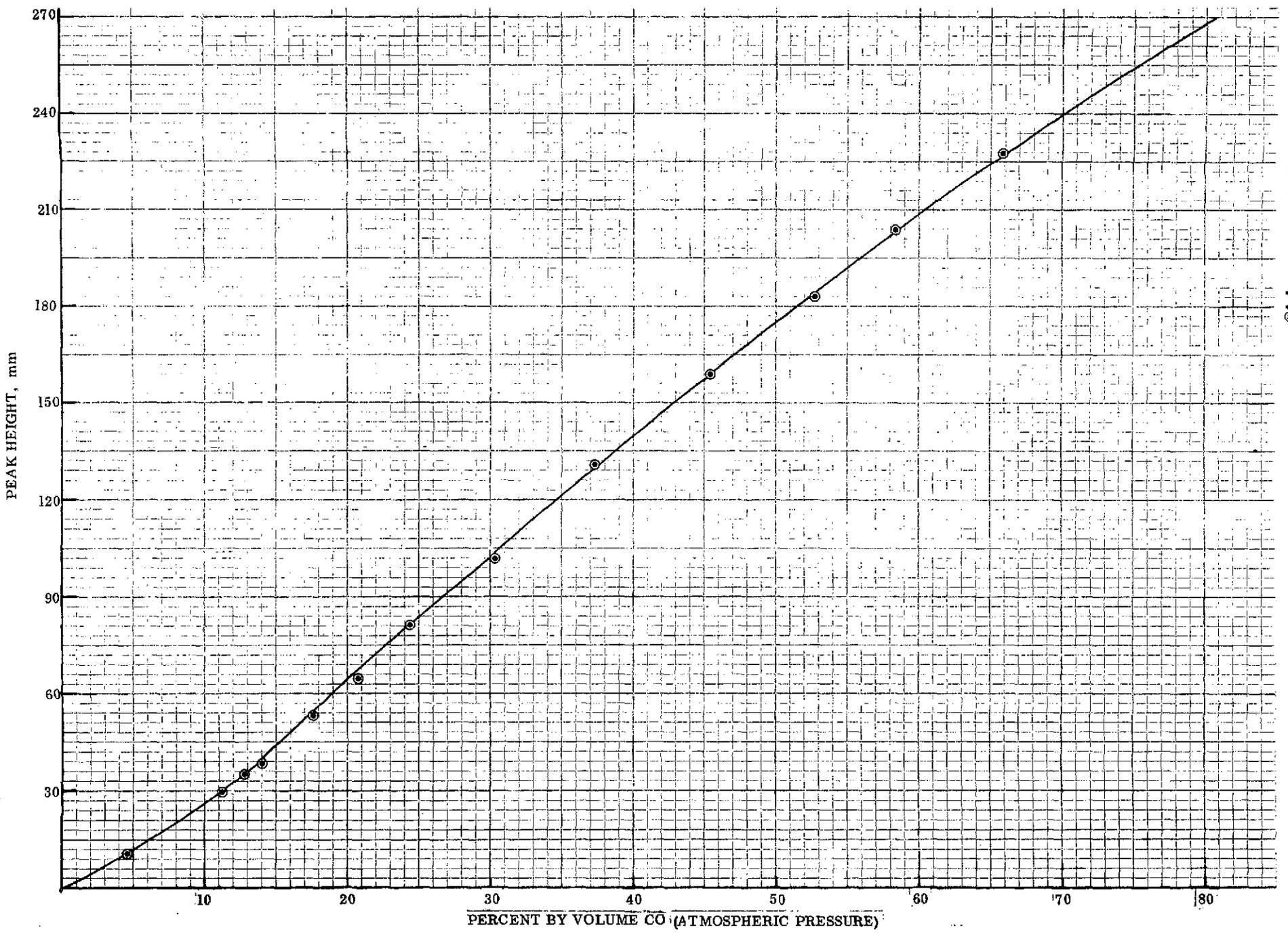
Chromatograph

This calibration was accomplished by two techniques. The first involved flowing calibration mixtures (from Scientific Gas Products) through the sample valve loop while holding the total pressure at $1.013 \times 10^{+5}$ Newton/meter² (atmosphere pressure). Standard samples were analyzed chromatographically until a set of at least six agreed within ± 1 mm. These were averaged and recorded. With some gases (e. g. CO) no standard mixtures were available, and with others (e. g. H₂) mixtures were not available over the entire range of interest. Here the second method was employed for calibration. A pure sample gas was fed into the sample valve loop in which a vacuum pump and precision absolute pressure gauge (Wallace and Tiernan Model No. FA 160, range 1 to 30 inches Hg equivalent to 1 atmosphere ($1.013 \times 10^{+5}$ Newton/meter²)) were connected so as to fill the sample loop to a desired pressure after evacuation. The second technique produced calibration curves displaying less scatter than the first. The final curves, Figures 2, 3, 4, and 5, represent a combination of the two techniques.



H₂ CALIBRATION CURVE, SMALL SCALE TESTS RUNS 1-38

Figure 2



CO CALIBRATION CURVE, SMALL SCALE TESTS RUNS 1-38

Figure 3

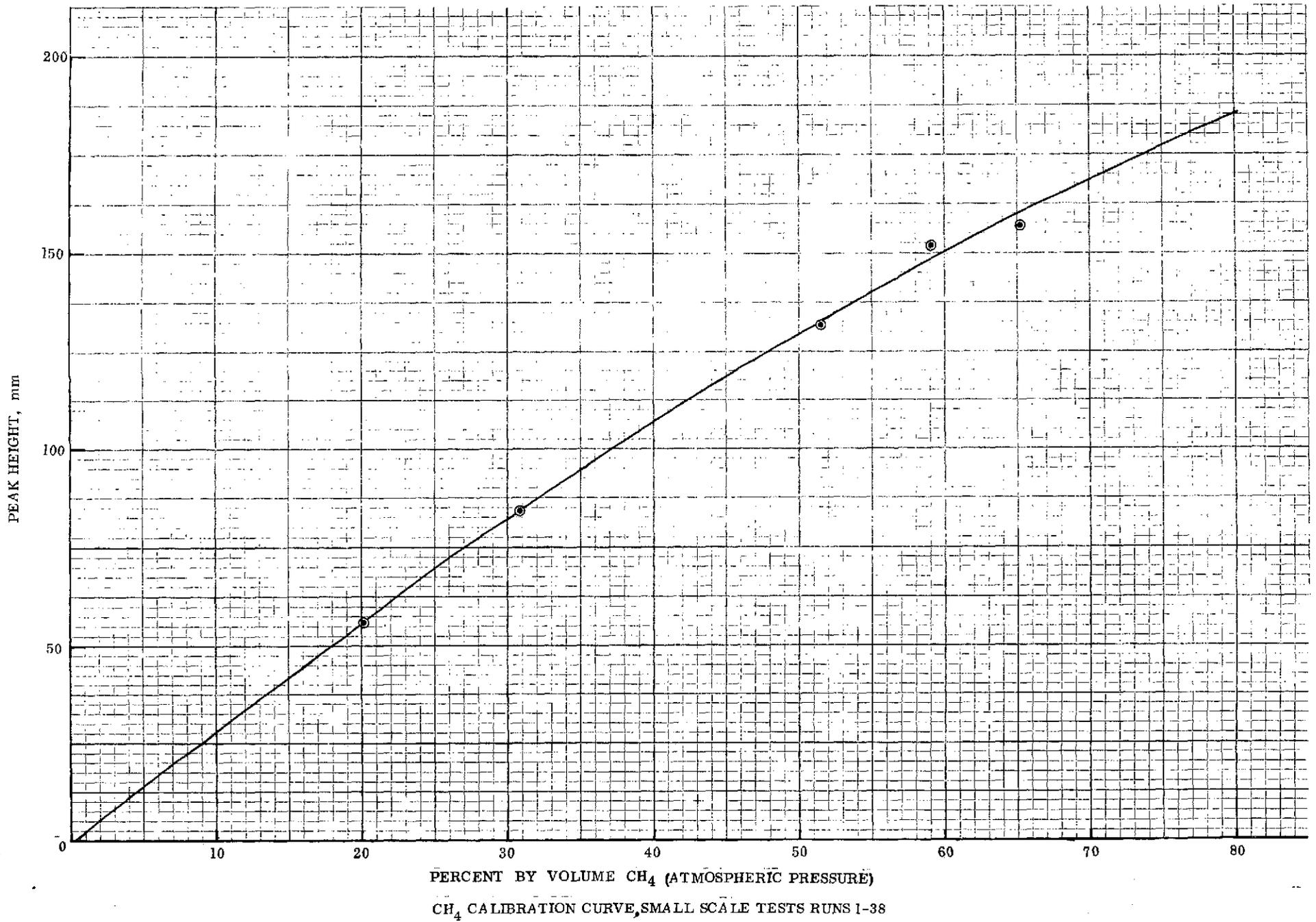
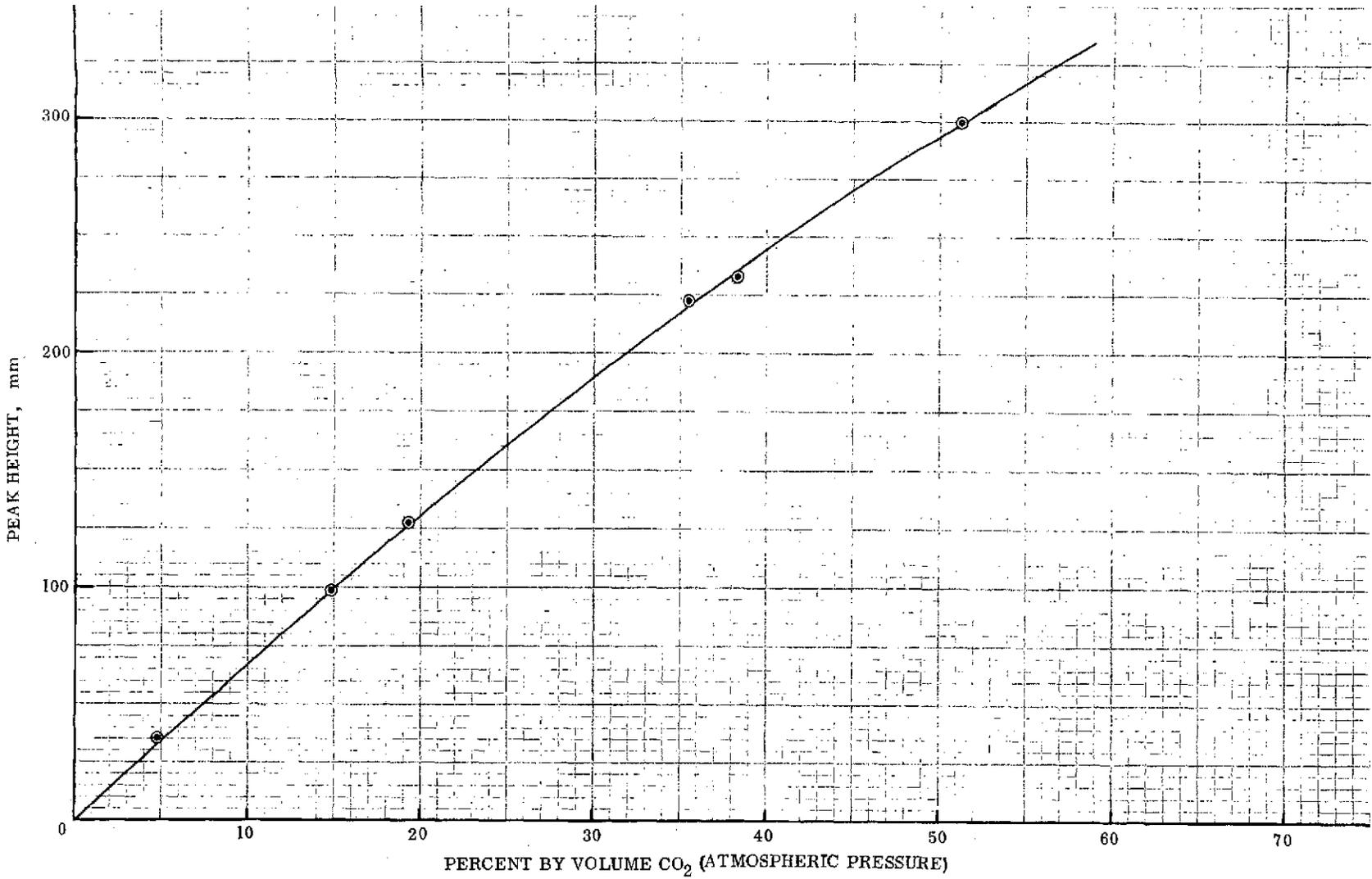


Figure 4



CO₂ CALIBRATION CURVE SMALL SCALE TESTS RUNS 1-38

Figure 5

Glassware

Scientific glassware was used for feed syringes, condensate collection, and gas generation rate measurement. The syringes and collection flasks were weighed empty and also when filled to a predetermined level with distilled water. Within the reliability limits, no corrections were necessary or applied. A buret modified to introduce soap bubbles was employed to measure gas generation rates. No corrections were applied to these generation rates.

Temperature

Millivolt readings of chromel-alumel thermocouples were checked at room temperature and 100°C (212°F). Thermocouples reading within 0.6°C (1°F) were selected and welded to various locations in the reactor oven. No high temperature calibration or corrections were applied.

Pressure Gauge

The Heise pressure gauge on the reactor outlet was set equal to the barometric pressure before each test run.

TEST PROCEDURE

Before this test series was started, the oven was brought to the desired temperature, and the chromatograph was checked. Before each run, a 50 x 10³ mm³ (50 ml) syringe of distilled water was slowly introduced by the syringe drive motor into the reactor, cleaning out any residue from the previous test. This was repeated until the gas generation rate was essentially zero. Then, the reactor and plumbing were flushed with a stream of helium to avoid chromatographic reading on residual gases. Next, a clean syringe was filled with a water solution of the desired waste, mounted in the drive motor assembly and positioned in the entrance port of the reactor.

The test run consisted of flowing the premixed water solution, at a preset rate, into the reactor where it was gasified, measuring the rate of gas product evolution, H₂O condensation and composition of the product gas. A complete set of test readings were recorded each time the chromatograph automatically sampled the existing gas stream, 480 seconds (8 minutes).

Test data was recorded on a number of variables, as necessary, to collect proper test data. Only a portion of this information was used in the data reduction program. This information is listed in Table II.

TABLE II

SMALL SCALE TEST DATA

Test Data Recorded		Test Data Used in Computer Reduction Program	
	Units		Units
Test sample	--	Test sample number	--
Run time	seconds	Run time	seconds
Reactor inlet pressure	psia	Reactor outlet pressure	psia
Reactor outlet pressure	psia	Reactor midpoint thermo- couple	°F
Reactor inlet thermocouple	°F		
Flash section thermocouple	°F		
Reactor midpoint thermocouple	°F		
Reactor exit thermocouple	°F		
Heat line thermocouple	°F		
Gas generation rate	ml/min	Gas generation rate	ml/min
Syringe feed volume	ml	Syringe feed volume	ml
Condensate volume	ml	Condensate volume	ml
Chromatographic peaks --		Chromatographic peaks --	
H ₂	mm	H ₂	mm
CO	mm	CO	mm
CH ₄	mm	CH ₄	mm
CO ₂	mm	CO ₂	mm
H ₂ O	mm	H ₂ O	mm

The test run was continued until steady state was achieved in gas production rate and gas composition. Typically, a run took 2 to 4 hours to complete. At this time, the syringe was removed, the reactor was flushed overnight with a low helium flow and then steam cleaned before starting the next test.

COMPUTER DATA REDUCTION

The test information collected in the small scale tests was input directly into a data reduction program. This program was able to display all test information on one single computer page for each test point, thus giving ease in the storage and retrieval of data useful for further interpretation. A typical computer print-out sheet is shown in Appendix B. In addition, the program performed many useful calculations employed in the final data treatment. These are summarized in Table III.

TABLE III
SUMMARY OF SMALL SCALE COMPUTER DATA REDUCTION PROGRAM

Data	Units	Remarks
Sample number	--	--
Run time	minutes	Calculated from input in seconds
Reactor temperature	°F	Temperature at reactor midpoint
Syringe volume	ml	Feed volume remaining in syringe
Volume collected	ml	Condensed water collected
Gas generation rate	ml/min	Measured with buret and stopwatch
Reactor volume	ml	Calculated volume of reactor
Density of steam	g/ml	Used in space velocity calculation
Set Conditions		
Liquid feed rate	ml/min	Rate set by syringe drive motor
Gas feed rate	ml/min	Measured feed rate if fuel is a gas
Space velocity	1/hours	Calculated as set liquid feed rate - multiplied by steam density at reactor temperature divided by reactor volume
Contact time	seconds	Reciprocal space velocity
Measured Conditions		
Liquid feed rate	ml/min	Calculated from syringe and run time. Reading from current & previous sample.
Liquid collect rate	ml/min	Calculated from volume collected & run time from current & previous sample
Space velocity		Calculated as summarized in section "Small Scale Tests - Continuous Reactor"
Contact time		Reciprocal measured space velocity
Reactor pressure	psia	Reactor outlet pressure - measured
Reactor pressure	atm.	Calculated from above

TABLE III (CONCLUDED)

Data	Units	Remarks
Chromatographic Peaks (for H ₂ , CO ₂ , CH ₄ , CO ₂ , H ₂ O)	mm	Measured values
Partial pressure	atm.	Calculated by linear interpolation of calibration curves--inputted as a table
Partial pressure total	atm.	The sum of the calculated constituent partial pressures
Mole fraction	--	Calculated as constituent partial pressure divided by the sum of the partial pressure
Predicted mole fractions	--	Values predicted from UAC thermodynamic equilibrium program
Predicted mole fractions (H ₂ O free)	--	Calculated from predicted mole fraction by summing all constituent mole fractions, except H ₂ O and dividing each by the sum
Predicted partial pressures	atm.	Predicted mole fractions multiplied by Reactor pressure
Predicted partial pressures (H ₂ O free)	atm.	Calculated from predicted partial pressures by subtracting predicted P _{H₂O} and equating sum of remain- ing terms to unity
Experimental/Predicted mole fractions		Ratio of experimental value to predicted
Experimental/predicted mole fractions (H ₂ O free)	--	Ratio of experimental value to predicted
Experimental/predicted partial pressures	--	Ratio of experimental value to predicted
Experimental/predicted partial pressures (H ₂ O free)	--	Ratio of experimental value to predicted
Rate of carbon generation	gm-atom/ min	Calculated from gas generation rate, ml/min multiplied by gm-atom/ml carbon obtained from chromatographic analysis
Carbon feed rate	gm-atom/ min	Calculated from measured liquid feed rate, and feed composition factors
(Generation/feed) ratio	percent	Ratio above two calculations. Termed "Carbon Recovery" in text.

TABLE IV

STEAM REFORMATION OF SUCROSE SOLUTION. 5% BY WEIGHT

Data Point	Reactor Temperature		Contact Time (sec.)	Gas Analysis Total (%)	Percent of Feed Constituents Recovered as Product Gases		
	°C	°F			Carbon	Hydrogen	Oxygen
18.03	642.8	(1189.)	2.15	101.04	40.0	43.0	50.6
18.04	643.9	(1191.)	2.91	99.14	46.2	50.0	55.2
18.05	647.8	(1198.)	2.38	99.27	38.1	43.5	46.2
Average	645.0	(1193.)	2.48		41.4	45.5	50.7
19.02	654.4	(1210.)	8.11	56.26	82.5	99.4	119.9
19.03	653.3	(1208.)	9.01	93.13	98.0	123.5	143.5
19.04	648.9	(1200.)	8.17	96.01	87.8	104.9	125.5
19.05	650.0	(1202.)	9.01	96.35	92.7	108.1	130.3
19.06	648.3	(1199.)	8.70	96.57	91.5	106.9	127.0
19.07	648.3	(1199.)	8.60	97.07	89.1	103.0	122.8
19.08	648.9	(1200.)	9.01	100.16	86.7	104.2	118.9
19.09	648.9	(1200.)	8.08	100.33	77.1	92.3	104.5
19.10	647.8	(1198.)	8.30	99.22	85.4	99.5	115.2
19.11	647.2	(1197.)	8.35	100.98	83.0	99.5	111.5
Average	649.4	(1201.)	8.53		87.4	104.1	121.9
21.03	645.0	(1193.)	20.1	96.60	95.8	121.2	137.0
21.04	645.6	(1194.)	19.4	97.98	96.1	119.6	134.8
21.05	645.6	(1194.)	20.2	98.57	99.2	121.8	138.5
21.06	646.1	(1195.)	19.4	100.19	91.5	113.4	127.6
21.07	646.6	(1196.)	20.2	99.69	102.5	125.5	143.2
21.08	647.8	(1198.)	19.4	99.96	98.9	120.7	138.2
21.09	647.8	(1198.)	20.2	99.44	104.7	126.2	146.8
21.10	647.8	(1198.)	19.6	101.75	104.7	130.9	146.4
21.11	647.2	(1197.)	20.1	99.52	94.2	113.5	131.7
Average	645.6	(1196.)	19.8		98.6	121.4	138.2
30.03	543.3	(1010.)	13.2	61.51	61.3	78.1	93.9
30.04	543.3	(1010.)	9.7	84.84	42.1	50.0	65.7
30.05	543.3	(1010.)	10.4	92.51	47.5	48.4	72.2
30.06	542.8	(1009.)	10.0	93.24	50.6	44.7	73.8
30.07	542.2	(1008.)	11.2	96.74	56.1	48.6	79.2
30.08	541.7	(1007.)	10.4	98.49	54.7	44.7	76.1
30.09	543.3	(1010.)	10.8	98.79	51.4	40.4	70.0
30.10	543.9	(1011.)	10.8	99.33	56.6	43.7	75.7
30.11	543.9	(1011.)	13.7	99.19	57.1	44.0	75.7
30.12	543.9	(1011.)	12.6	99.57	56.2	42.2	74.3

TABLE IV (CONCLUDED)

Data Point	Reactor Temperature		Contact Time (sec.)	Gas Analysis Total (%)	Percent of Feed Constituents Recovered as Product Gases		
	°C	°F			Carbon	Hydrogen	Oxygen
30.14	543.3	(1010.)	10.0	99.46	56.0	41.1	74.0
30.15	543.3	(1010.)	10.4	99.45	58.2	42.0	76.6
30.16	543.3	(1010.)	12.6	99.28	52.7	36.3	69.5
Average	542.8	(1009.)	11.2		53.9	46.5	75.1
31.03	537.8	(1000.)	35.7	30.04	100.0	92.2	133.9
31.04	537.8	(1000.)	38.0	51.13	94.4	94.6	126.7
31.05	538.3	(1001.)	35.3	64.80	98.6	94.1	131.8
31.06	539.4	(1003.)	39.0	75.34	100.8	95.6	133.0
31.07	540.0	(1004.)	36.6	82.83	97.8	93.0	128.3
31.08	540.6	(1005.)	39.0	85.82	106.5	96.7	139.1
31.09	540.6	(1005.)	36.6	90.80	101.6	95.7	131.4
31.10	540.6	(1005.)	32.1	93.67	100.3	95.7	128.9
Average	539.4	(1003.)	36.5		100.0	94.7	131.6

TABLE V

STEAM REFORMATION OF SUCROSE SOLUTION. 10% BY WEIGHT

Data Point	Reactor Temperature		Contact Time (sec.)	Gas Analysis Total (%)	Percent of Feed Constituents Recovered as Product Gases		
	°C	°F			Carbon	Hydrogen	Oxygen
25.03	646.1	(1195.)	36.9	62.68	97.6	135.9	154.8
25.04	644.4	(1192.)	37.7	84.04	101.8	145.2	162.2
25.05	646.1	(1195.)	36.7	90.81	103.8	144.4	166.5
25.06	646.6	(1196.)	35.7	91.62	105.9	139.9	169.0
25.07	649.8	(1198.)	37.8	95.47	103.5	140.6	164.9
25.08	647.2	(1197.)	33.0	95.96	102.1	137.5	162.4
25.09	643.3	(1199.)	33.2	97.21	98.2	134.1	156.6
25.10	650.0	(1202.)	37.5	97.66	107.8	146.4	171.2
25.11	648.3	(1199.)	36.2	97.61	105.0	142.6	167.5
25.12	648.9	(1200.)	33.5	96.62	99.4	132.1	158.5
25.13	648.3	(1199.)	35.0	96.61	101.1	133.8	161.7
25.14	648.3	(1199.)	30.6	96.78	106.6	140.6	170.4
Average	647.8	(1198.)	35.3		102.7	139.4	163.8
26.01	647.2	(1197.)	10.0	83.85	75.0	88.7	103.3
26.02	646.6	(1196.)	10.0	100.74	79.4	94.3	108.3
26.03	646.6	(1196.)	10.8	102.18	86.4	101.0	117.3
26.04	646.6	(1196.)	10.2	102.16	81.8	93.9	110.5
26.05	646.6	(1196.)	9.9	103.30	84.3	97.3	112.7
26.06	646.6	(1196.)	10.3	102.99	82.9	95.8	110.2
26.07	647.2	(1197.)	10.8	102.82	84.4	97.4	112.7
26.08	647.2	(1197.)	12.3	103.32	84.4	97.7	111.9
26.09	647.2	(1197.)	11.7	101.38	81.7	94.9	109.6
26.10	647.8	(1198.)	12.1	101.74	83.6	98.8	112.9
26.11	647.2	(1197.)	9.9	102.89	86.2	98.6	113.5
26.12	647.2	(1197.)	11.2	103.97	83.9	93.0	112.8
26.13	647.2	(1197.)	11.1	98.36	78.9	98.6	105.5
Average	647.8	(1198.)	10.8		82.5	96.2	110.9
28.03	649.4	(1201.)	1.87	97.94	61.9	63.5	80.3
28.05	650.0	(1202.)	4.14	99.31	58.3	69.2	80.1
28.07	650.0	(1202.)	3.64	99.84	66.6	71.6	84.7
Average	650.0	(1202.)	3.22		62.3	68.1	81.7
29.02	792.8	(1459.)	2.54	93.39	84.2	93.0	110.1
29.03	793.3	(1460.)	2.70	100.22	85.4	96.9	109.8
29.04	794.4	(1462.)	2.92	97.83	83.9	96.9	108.4
29.05	795.0	(1463.)	3.97	100.67	85.0	97.3	111.9
29.06	796.7	(1466.)	4.11	100.37	86.0	96.8	112.6
Average	794.4	(1462.)	3.25		84.9	96.2	110.6

TABLE VI

STEAM REFORMATION OF GLYCINE SOLUTION. 5% BY WEIGHT

Data Point	Reactor Temperature		Contact Time (sec.)	Gas Analysis Total (%)	Percent of Feed Constituents Recovered as Product Gases		
	°C	°F			Carbon	Hydrogen	Oxygen
32.02	652.8	(1207.)	3.73	21.98	26.3	23.2	37.0
32.03	651.7	(1205.)	3.47	61.90	24.5	23.3	36.3
32.04	651.1	(1204.)	2.88	80.47	25.3	21.8	37.3
32.05	651.1	(1204.)	2.44	85.32	27.3	22.0	39.3
Average	651.7	(1205.)	3.13		25.9	22.6	37.5
33.03	647.2	(1197.)	7.60	5.92	35.3	49.0	50.5
33.04	648.3	(1199.)	10.3	24.54	39.9	53.9	62.7
33.05	648.3	(1199.)	12.4	61.11	53.8	62.4	79.9
33.06	647.8	(1198.)	8.47	76.74	44.4	47.8	63.5
33.07	647.8	(1198.)	10.5	88.14	47.1	50.2	66.0
33.08	647.2	(1197.)	11.6	87.93	47.5	48.6	65.7
33.09	647.8	(1198.)	10.7	90.36	46.0	46.6	63.1
33.10	647.8	(1198.)	9.75	90.03	58.5	59.6	79.2
33.11	648.3	(1199.)	12.8	90.17	53.7	54.3	72.6
33.12	648.3	(1199.)	10.7	92.50	47.3	45.4	63.4
33.13	648.3	(1199.)	8.75	92.07	48.3	46.0	65.3
33.14	647.2	(1197.)	12.8	90.99	65.5	61.2	88.2
33.15	647.8	(1198.)	7.10	90.64	49.2	46.4	65.7
33.16	647.8	(1198.)	7.19	90.64	49.5	46.3	66.5
33.17	647.8	(1198.)	7.70	89.47	49.4	44.3	66.6
Average	647.8	(1198.)	9.89		49.0	50.8	67.9
34.03	652.2	(1206.)	31.5	14.20	80.8	97.2	110.8
34.04	652.2	(1206.)	32.1	27.14	83.6	90.9	121.5
34.05	652.8	(1207.)	31.8	44.25	82.7	86.4	123.3
34.06	652.8	(1207.)	31.8	57.31	80.7	89.0	119.3
34.07	652.8	(1207.)	37.0	63.08	85.1	88.2	125.3
34.08	653.3	(1208.)	37.6	68.52	84.4	87.9	123.8
34.09	653.3	(1208.)	37.2	70.34	86.0	86.2	125.9
34.10	653.3	(1208.)	26.8	70.84	85.5	81.7	126.2
34.11	653.3	(1208.)	30.8	71.79	88.2	82.1	130.1
34.12	653.3	(1208.)	30.7	72.40	89.0	82.0	130.4
Average	652.8	(1207.)	32.7		84.6	85.4	123.7

DATA ANALYSIS

Process Kinetics

A kinetic analysis was completed on the experimental tests collected with the small scale reactor. These tests were performed on an aqueous 5 percent by weight sucrose solution at 537.8°C (1000°F) and 648.9°C (1200°F). A few tests were run with five percent glycine at 648.9°C (1200°F) only. Also, two tests were performed on methane at 648.9°C (1200°F).

A summary of these runs is displayed in Tables IV, V, and VI. Gas contact times were calculated for each data point. The percent of the feed constituents (carbon, hydrogen, and oxygen) recovered in the product gases was calculated for each sample point. The results were then averaged for the total run, and the final value of carbon recovery as a function of contact time is shown plotted in Figure 6. Note in Tables IV, V, and VI that the recovery of hydrogen and oxygen as gaseous products in many tests exceeds 100 percent. It is presumed that the extra hydrogen and oxygen come from reactions involving consumption of feed water.

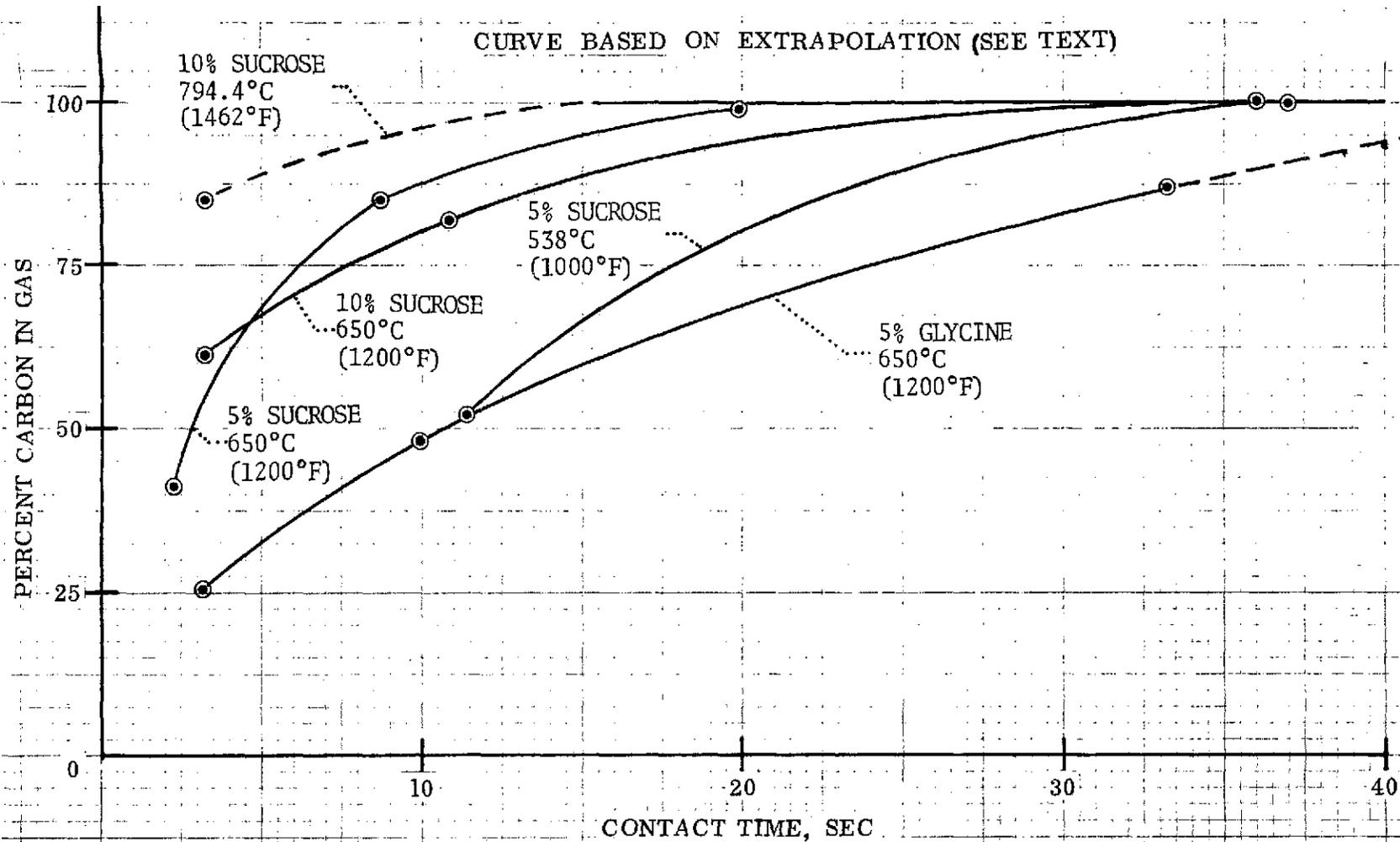
For most of the runs, contact times were obtained in which carbon recovery was quite close to 100 percent. However, with 5 percent glycine at 648.9°C (1200°F) and 10 percent sucrose at 794.4°C (1462°F), where the highest recoveries were only 85 percent, extrapolation was necessary to obtain contact times at 100 percent carbon recovery. In order to estimate the contact time for 100 percent carbon recovery with 10 percent sucrose at 794.4°C (1462°F), the 5 percent sucrose, 100 percent carbon recovery contact times were plotted versus temperature and then a parallel line was drawn from the 10 percent sucrose data point at 648.9°C (1200°F) Figure 7. Because of the approximations involved, the extrapolated contact time must be considered only an estimate. However, the result appears to be reasonable as shown by the broken line on Figure 6. The 5 percent glycine contact time was obtained by simple curve extrapolation. These contact times are summarized in Table VII below.

TABLE VII

CONTACT TIME REQUIRED FOR 100% CARBON RECOVERY AS PRODUCT GASES

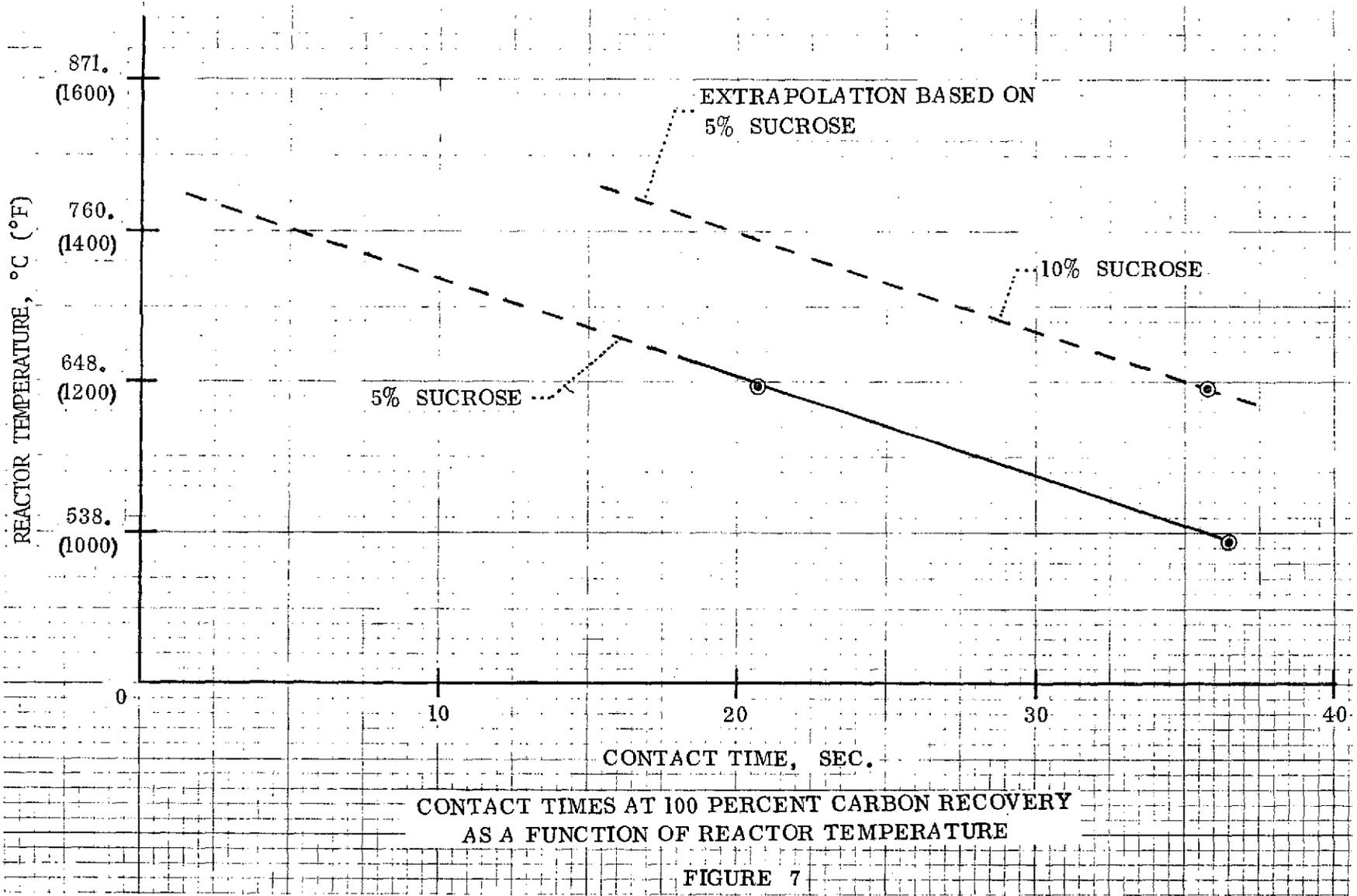
Test Sample	Reactor Temperature		Reactor Contact Times (sec.)
	°C	°F	
5% Sucrose	538	(1000)	36.5
5% Sucrose	650	(1200)	20.7
10% Sucrose	650	(1200)	35.3
10% Sucrose	794	(1462)	(15.)*
5% Glycine	650	(1200)	(45.)*

*Estimated values shown in brackets.



CONTACT TIMES VERSUS CARBON RECOVERY
FOR A NUMBER OF TEST RUNS
REACTOR VOLUME 227 ML

FIGURE 6



The 650°C (1200°F) methane steam reformation test indicated very little or no conversion to product gases when operated at 3 and 10 second contact times with H₂O to fuel weight ratios of 40 to 1 (2.5 percent by weight) and 99 to 1 (1 percent by weight) respectively. Processing of CH₄ and other simple hydrocarbons to CO and CO₂ is neither required nor desirable as it would be converted back to CH₄ in a Sabatier reactor subsystem.

In summary, sucrose appeared to be the easiest of the pure constituents to decompose, requiring contact times in the 15 to 36 second range. Glycine was about 4.5 times more difficult to decompose, requiring a contact time of 45 seconds, and methane is very difficult to decompose.

Material Mass Balance

In order to account for most or all of the feed constituents, a gas chromatograph analysis was conducted on each sample using the Bendix Process chromatograph described in the section on "Small Scale Test Apparatus Description". Water was condensed out before chromatographic analysis. This was occasionally operated on "scan" to look for gaseous constituents other than H₂, CO₂, CO, and CH₄. Very few other gases were found, and these were at low concentrations. Because of the manner in which the chromatograph was programmed, it was not possible to distinguish between CO and N₂. Accordingly, additional laboratory analysis was performed on samples from two test runs when nitrogen was present in the liquid feed. These were the glycine runs 33 and 34. Both of these runs employed 5 percent glycine as the feed. Analysis of both runs detected only a very small amount of N₂ and a smaller amount of O₂ and these were in the ratio found in air. It was concluded that the N₂ was a residual in the sample bottle after pretest purging and not from glycine reformation. The other trace impurities detected were acetone, possibly ethylene and two unidentified components.

Chemical analysis for NH₃ in the condensate was performed by both Nessler's colorimetric and Kjeldahl techniques. The Nessler's method was performed on run numbers 33 and 34; the Kjeldahl only on run 33. Because of the high dilution required in the Nessler's method (500 to 1), confidence in the Kjeldahl technique is higher. No non-volatile nitrogen was found from the Kjeldahl analysis of run number 33. Results for NH₃ are listed in Table VIII for runs with 5 percent glycine (actually measured as 5.18 percent). From these results, it is concluded that significant quantities of the nitrogen present in the glycine were converted into ammonia or soluble amines and collected as liquid constituents in the condensate, and not to N₂ gas as is predicted by Appendix A.

For all sample points, gas analyses of H₂, CO, CH₄, and CO₂ were used to establish a carbon, hydrogen, and oxygen "process feed" to "gas generation" mass balance. The computer program described in "Computer Data Reduction" was used to calculate the balances summarized in Tables IV, V, and VI. A carbon mass balance of close to 100 percent was obtained for most sucrose runs, indicating complete gasification of the carbon in sugar. For the hydrogen and oxygen present in the sugar, the picture is

not as clear because these values were at times much greater than 100 percent. One would expect the hydrogen and oxygen to be 100 percent for complete fuel gasification and to be greater than 100 percent if some of the water vapor present became chemically involved in the process. Also, the ratio of excess hydrogen to excess oxygen would be expected to, as in H_2O , be 2 to 1. Generally, the excess oxygen was greater than the excess hydrogen and was not equal to the ratio of 2 to 1.

Gas compositions for each test run were compared with computer predicted H_2 equilibrium values. It was found, in general, that only 60 to 70 percent of predicted H_2 was present in these tests. This indicates that complete steam reformation is not occurring, although the hydrogen and oxygen fuel product gas mass balances indicate that steam reformation is taking place. Some pyrolysis might also be taking place in which water does not have an active chemical role but may serve primarily as fuel carrier and heat transfer agent. Water most likely serves to eliminate charring and carbon formation. Carbon monoxide formation was found to be 10 to 30 times in excess over predicted values, and CO_2 was only 30 to 40 percent of its predicted value. Methane, which was predicted to be almost zero, was found to be present at 3 to 8 percent by volume. Complete reformation to equilibrium products is not a necessary requisite for system design because a downstream Sabatier can process all these gases to the desired products.

TABLE VIII

CONDENSATE NITROGEN ANALYSIS

Sample Number	Contact Time, Sec.	Nessler's		Kjeldahl		
		% by Wt. NH_3	% of Fuel	% by Wt. NH_3	% of Fuel	PH
33	9.89	0.85	72.4	0.72	61.2	9.0
34	32.7	1.3	110.5	(1.10)*	(93.5)*	9.0

*Estimated from ratio Nessler's to Kjeldahl for Run 33.

LARGE SCALE - BATCH REACTOR

Three types of tests were performed in the batch test series, namely:

- Synthetic Waste Tests
- Spacecraft Waste Tests
- Special Tests

The synthetic waste tests were performed on four classes of pure compounds. Representative pure compounds were selected first, to establish the feasibility of steam reforming spacecraft wastes; second, to characterize the critical parameters in the process; and third, to generate quantitative data on which to build a rate model for gasification.

The spacecraft waste tests were performed on mixtures of real wastes. These tests were designed to confirm the data of the synthetic tests and also, to determine whether any process enhancement results from the addition of two catalysts. One catalyst was employed to enhance char gasification, while another was selected to reduce the ammonia level in the exit gas.

Three special tests were performed with selected synthetic wastes. These tests were designed to provide some understanding of the chemical nature of the gasification and the role of water vapor in the reaction process.

SYNTHETIC WASTE TESTS

The synthetic waste tests were designed to provide a better understanding of the steam gasification processes and to generate data with which to build a quantitative gasification model. The materials selected and tested were:

- Cellulose. - $(C_6H_{10}O_5)_n$ Nine batch tests were conducted with FMC Avicel PH-102 microporous alpha-cellulose powder. Alpha-cellulose is a biologically active sugar substance readily degraded by active enzymes. This material was selected because for these tests, it represented both a typical sugar and a "paper like" waste product.
- Protein. - Three tests were conducted on methionine $(C_5H_{11}O_2NS)$. Reagent grade powder was selected to represent products of feces and nitrogen wastes.
- Hydrocarbon. - Three tests were conducted on polyethylene $(C_2H_4)_n$ plastic. Selected for these tests was a clear "Saran" material, 0.2 mm (8 mils) thick, which is normally used to make plastic bags. This waste was used without further processing except to cut the material to convenient sizes for weighing into the sample boat.

- Halogen. - One test was performed to determine the effect of a halogen on the steam gasification process. Since the iodate ion (IO_3^-) is an ingredient in urine sterilization, the effect of potassium iodate (KIO_3) on cellulose processing was examined.
- Mixture. - Lastly, a mixture of cellulose, methionine, polyethylene and potassium iodate was processed.

SPACECRAFT WASTE TESTS

The spacecraft waste tests were conducted to evaluate the performance of real wastes in the steam reformation process. The information collected was the same as with the individual and mixture synthetic waste tests. These tests were designed to provide a comparison with the results obtained with the synthetic waste tests. The waste composition evaluated consisted of equal parts by weight of fresh feces, fresh urine, paper toweling (Mosinee Type 417), and polyethylene plastic.

These tests were conducted first with and then without the two catalysts--sodium carbonate (Na_2CO_3) and ruthenium chloride ($\text{RuCl}_3 \cdot x \text{H}_2\text{O}$).

Sodium carbonate was selected as it is known to enhance char gasification. Ruthenium chloride was tested as a candidate to lower the effluent ammonia level.

The chemical model selected to approximate the composition of real waste as well as the mixture of synthetic wastes tested was:

Carbon	51.50%
Hydrogen	7.98%
Oxygen	32.96%
Nitrogen	0.60%
Sulfur	1.37%
Iodine	5.60%

This was the input composition for the computer predictions of the spacecraft wastes summarized in Appendix A. For some tests, this composition was considered to be only an approximate guideline.

SPECIAL TESTS

Three special tests were conducted to provide some understanding of the chemical processes occurring in the gasifier and converter reactors. The effect of water vapor on the processes was of particular interest in this study.

A sample of the same polyethylene material evaluated in the synthetic tests was heated in a manner to analyze the products directly from the gasifier reactor. Cellulose was heated in the presence and absence of steam while gases evolving directly from the gasifier were measured.

LARGE SCALE APPARATUS DESCRIPTION

An apparatus was designed and constructed to process water-waste slurries. The equipment included a slurry feed pump, reservoir and flash boiler. After preliminary checkout tests encountered severe injection port plugging, the apparatus was modified to accept batch samples.

The batch test apparatus consisted of four types of equipment:

- Gasifier and converter reactors
- Heating and temperature control equipment
- Steam generation equipment
- Sampling and measuring equipment

Batch Reactor

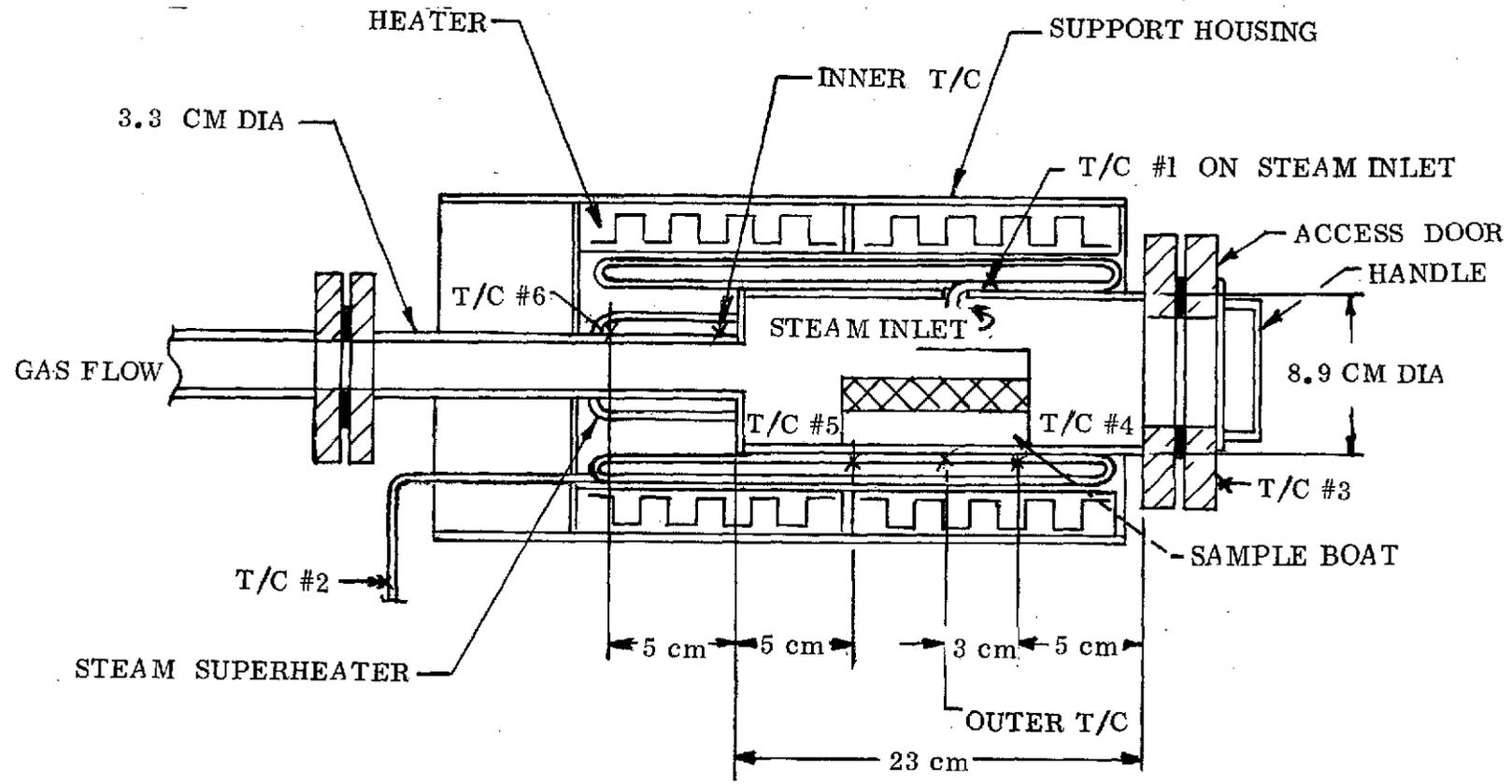
The test set-up was modified for the batch tests by adding a welded "gasifier" section to the converter reactor described below. The gasifier section, Figure 8, fabricated from AISI 316 stainless steel, includes a 100 millimeter (4 in.) pipe flange access port for insertion and removal of a sample boat at the access port with an asbestos-steel gasket. The gasifier reactor was contained in a muffle oven and bolted to the "converter reactor".

The "converter reactor" was fabricated out of 25.4 millimeter (1 inch) diameter thin wall AISI 316 stainless steel pipe. Flanges were welded to both ends of three 1.83 meter (six foot) sections of the reactor. These were bolted together giving a total reactor length of 5.5 meters (18 feet). The volume of the reactor was calculated to be $4.1835 \times 10^6 \text{ mm}^3$ (4183.5 cm^3). At the reactor exit port was an ash trap, water condenser, and water collector arrangement, Figure 9.

The configuration and dimensions of the sample boat are illustrated in Figure 10.

Heating and Temperature Control

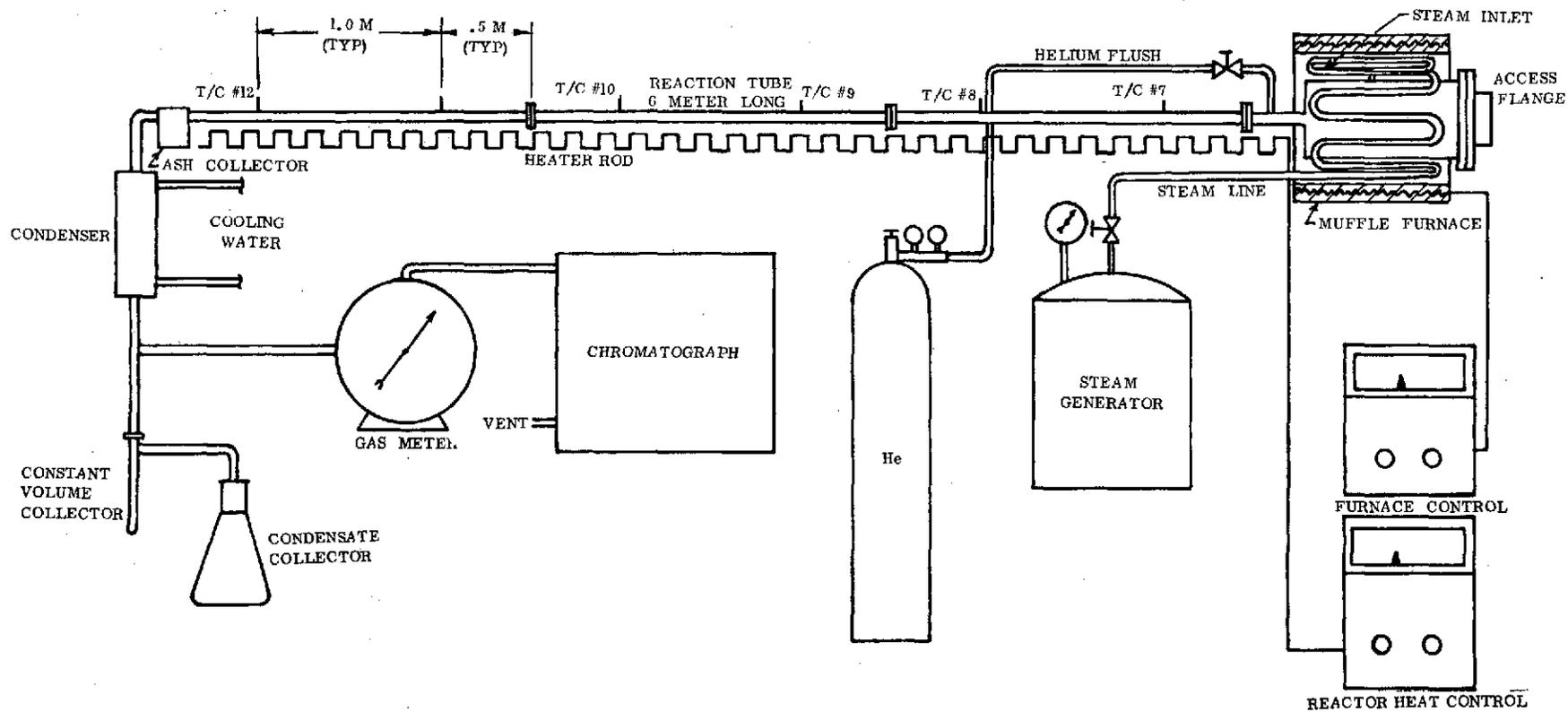
Four semi-circular "Lindberg Heavi-Duty" heaters were mounted in a steel housing so as to provide approximately uniform heating to the gasifier reactor enclosed within the heaters. Two heater sections were mounted radially and connected electrically to provide two separate radially heated zones along the axis of the gasifier reactor. Two Honeywell on-off temperature controllers were employed to supply current to each of the two reactors of the muffle oven, through 20 amp, 120 volt variable powerstats. Controller thermocouples were welded to the reactor at locations as shown in Figure 9. Desired heating rates were obtained by varying the potentiometric setting.



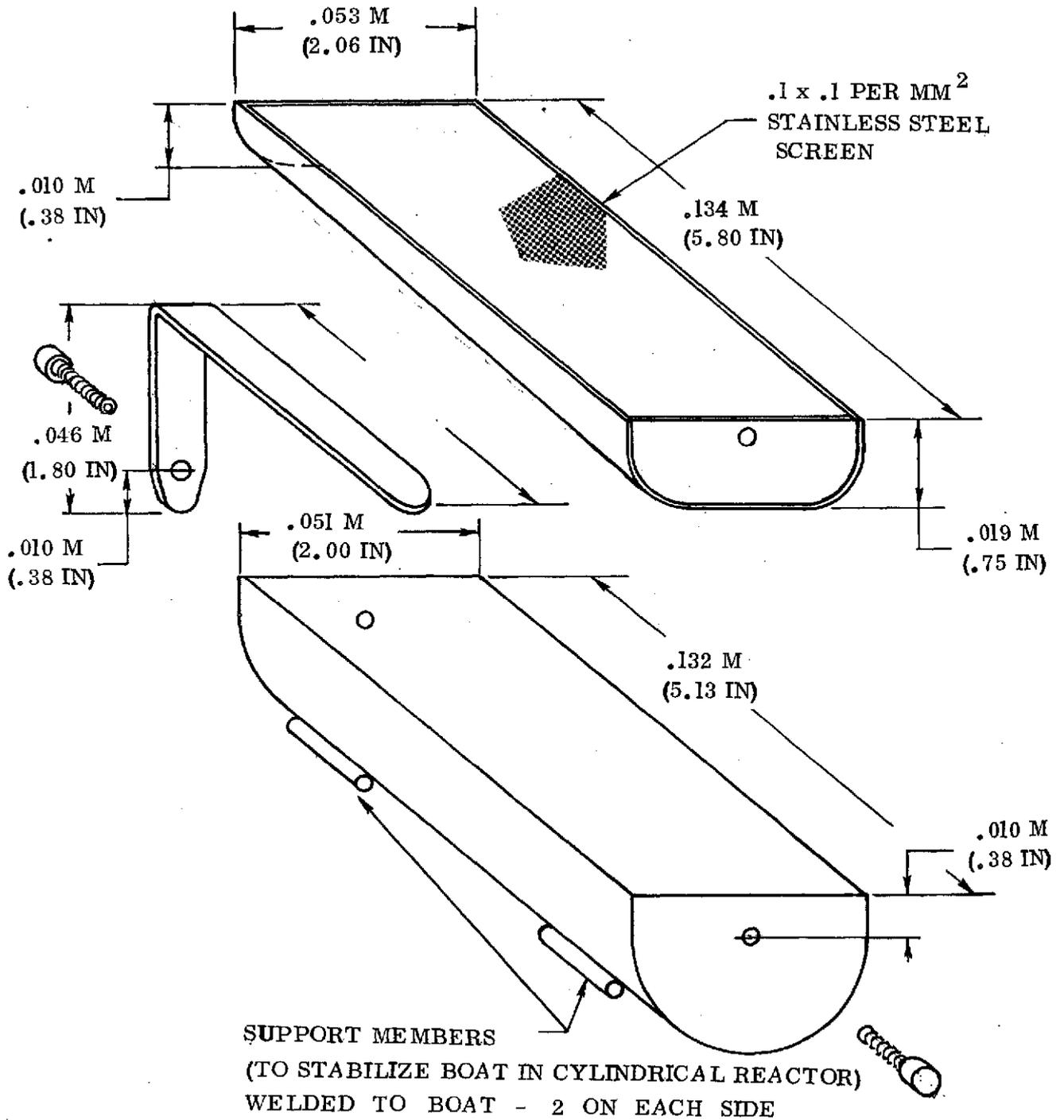
GASIFIER SECTION OF BATCH REACTOR

FIGURE 8

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BATCH REACTOR TEST APPARATUS
 FIGURE 9



MATERIAL - TYPE AISI 316

BATCH REACTOR SAMPLE BOAT

FIGURE 10

Each of the three conversion reactor sections was fitted with three contact heater rods of 3 amp, 120 volt ratings. Three 10 amp powerstats were employed to control the temperatures of the conversion reactor sections. Two chromel-alumel thermocouples were welded to each reactor as shown in Figure 9. These thermocouples generally read within $\pm 37.8^{\circ}\text{C}$ (50°F) during a test run. The oven and reactor sections were wrapped with 0.0254 to 0.051 meters (1 to 2 inches) of Kaowool ceramic fiber insulation.

Steam Generator

The flow of steam into the gasifier reactor was provided by a converted 0.029 cubic meter (30 quarts) auto-clave. The steam flow rate was maintained by flowing steam through a precalibrated Hoke micro-metering valve. Constant boiler steam pressure was maintained by carefully controlling boiler steam temperature with a West SCR stepless temperature controller. Steam pressure was maintained in this manner to $\pm 6.9 \text{ KN/meter}^2$ ($\pm 1 \text{ psi}$) at 170 KN/meter^2 (10 psig). Flow lines to the oven were maintained at 148°C (300°F) with heating tape. Inside the oven the AISI 316 stainless steel line steam flow was heated to reactor temperature by flowing through 1.83 meter (6 feet) of line situated in the oven but outside of the reactor as shown in Figure 8. Steam entered the reactor directly above the sample boat, then swept through the gasifier, into the conversion reactor and ultimately was condensed at the reactor exit port.

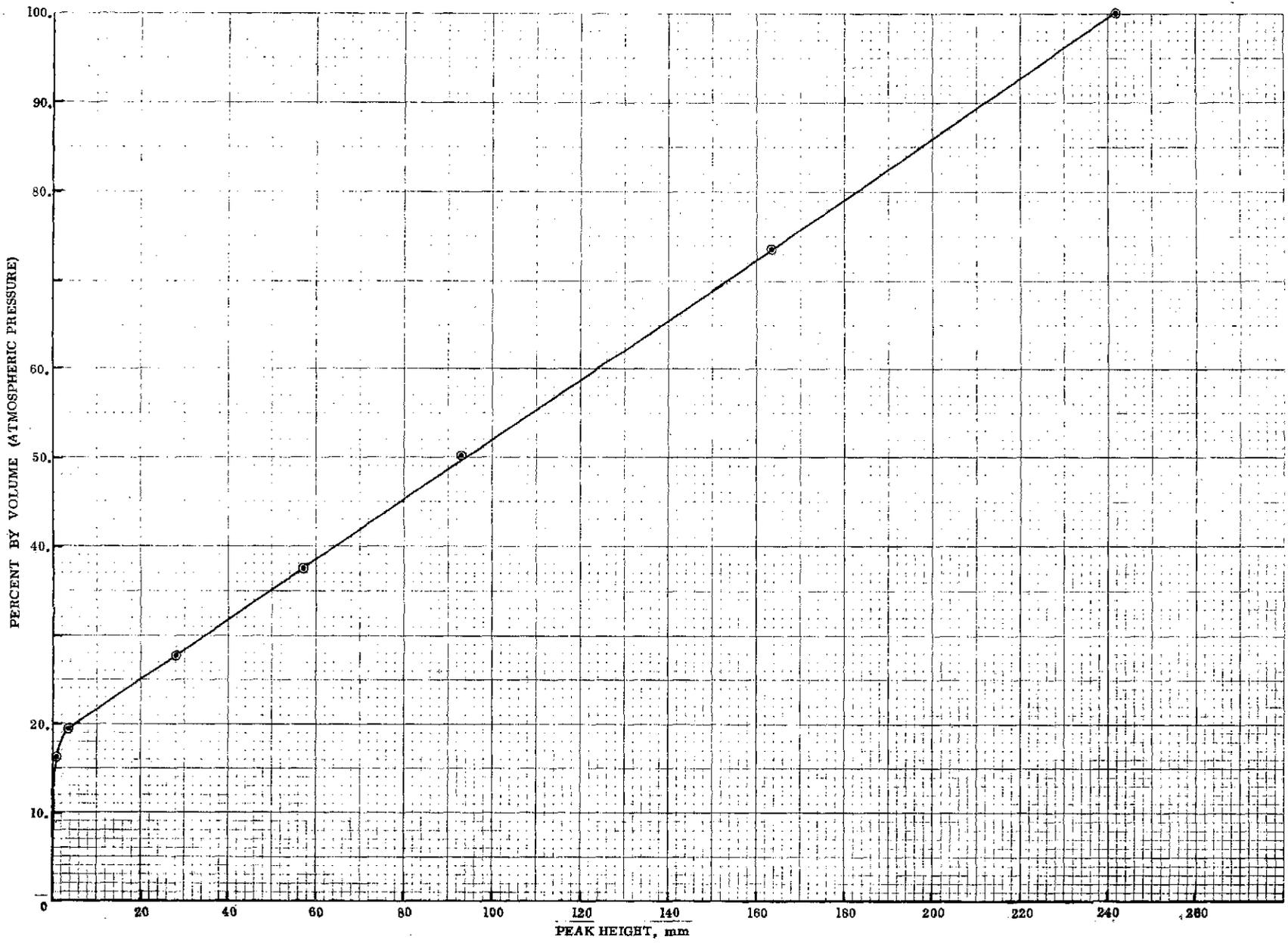
Sampling and Measuring Equipment

Gas Analysis

A Bendix model Chroma Trend 100T process chromatograph was employed for gas analysis. This instrument differed in many ways from the unit used for the "small scale tests". The three principle differences were:

- Use of molecular sieve column vs. Porapak "Q" to resolve oxygen and nitrogen
- Sample analysis time of 800 seconds (13.3 minutes) versus 480 sec. (8 minutes)
- Processing of detector output through an analog integrator vs. a non-integrated output

The elution order was H_2 , CO_2 , O_2 , N_2 , CH_4 , and CO . An analysis for water vapor was not made as it was condensed out at the reactor exit port. Calibration and data reduction were handled in the same manner as with the small scale tests. The chromatographic calibration curves for H_2 , CO_2 , O_2 , N_2 , CH_4 , and CO are shown in Figures 11 through 16 respectively.



H₂ CALIBRATION CURVE, 300 SERIES TESTS - BATCH TEST

Figure 11

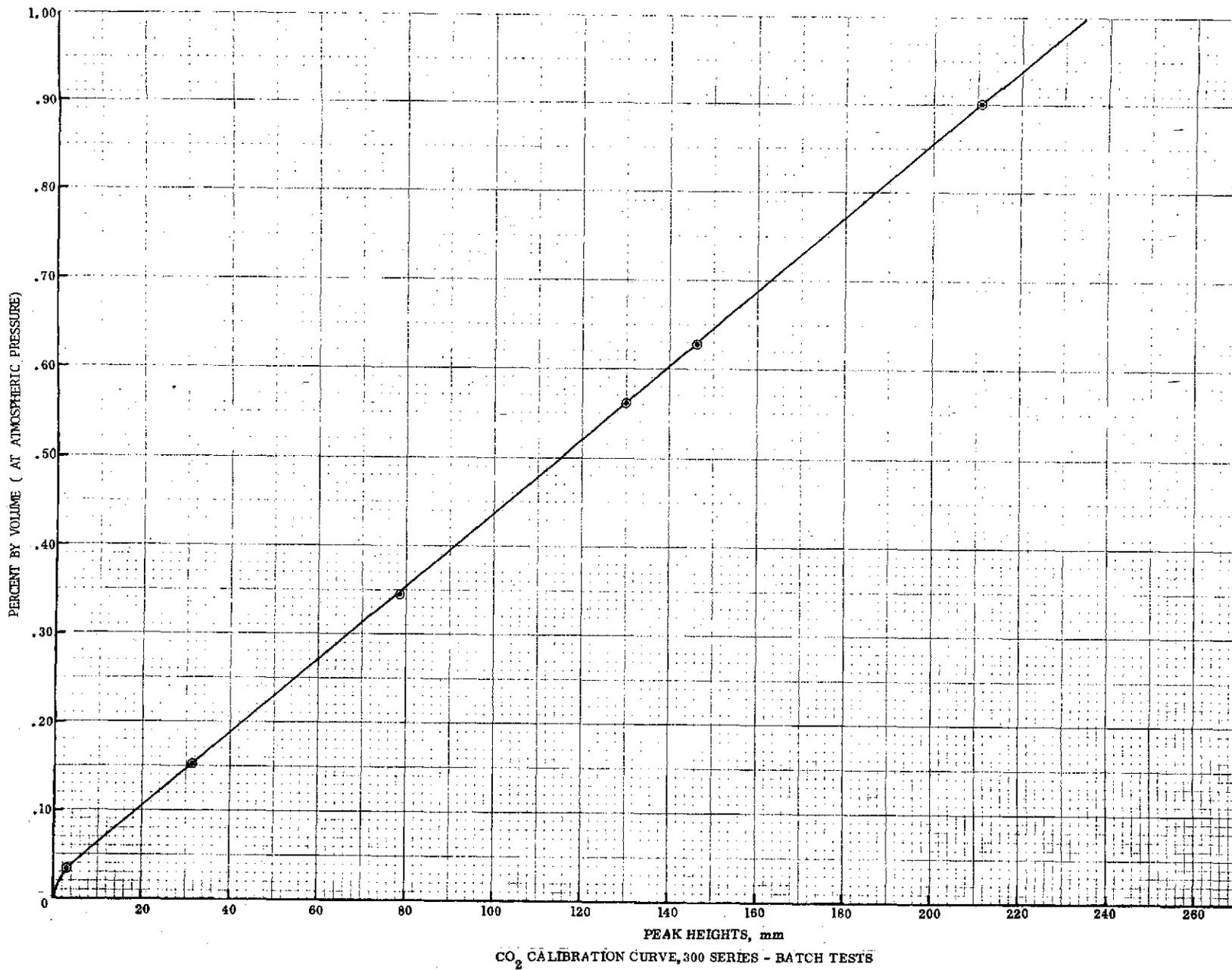
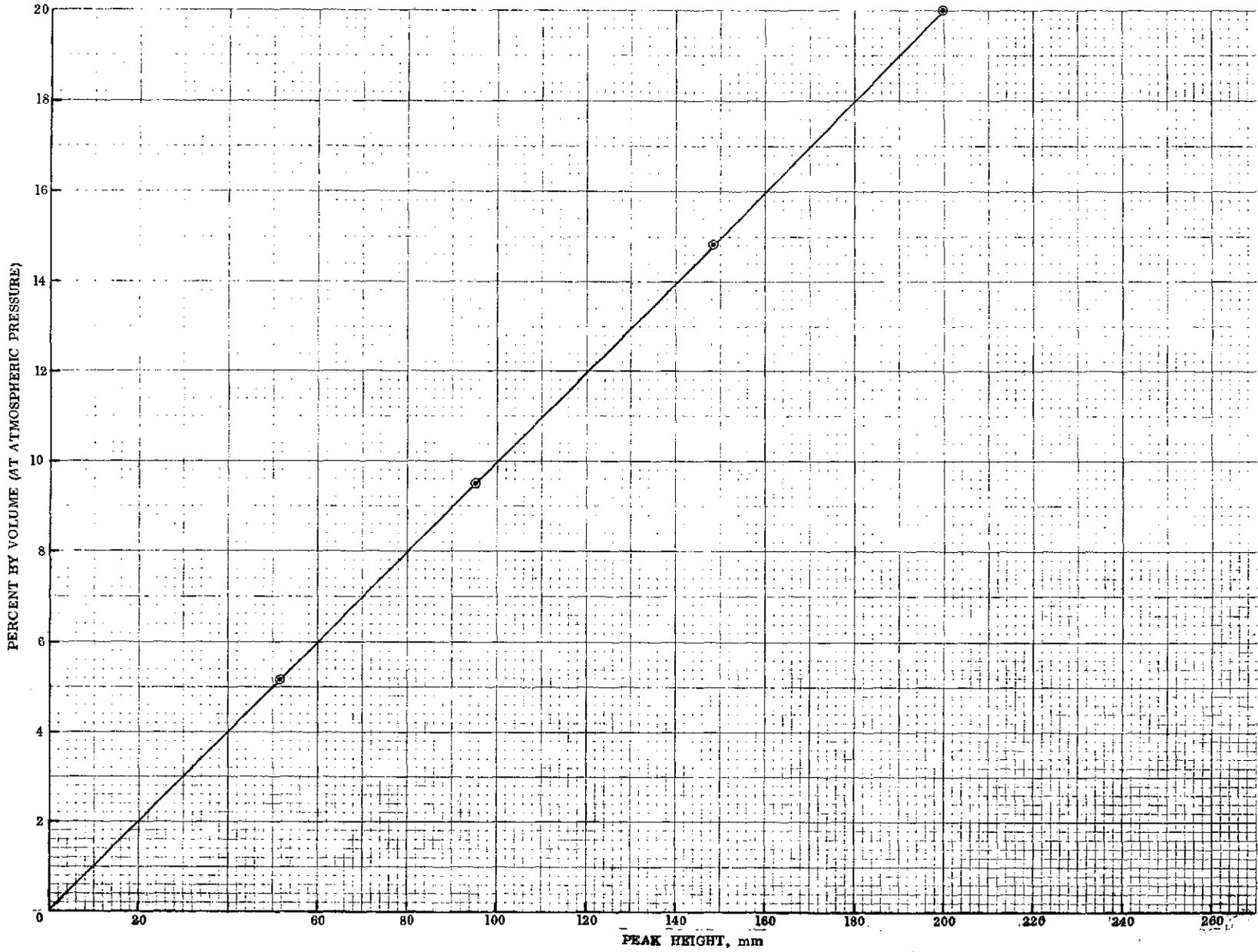
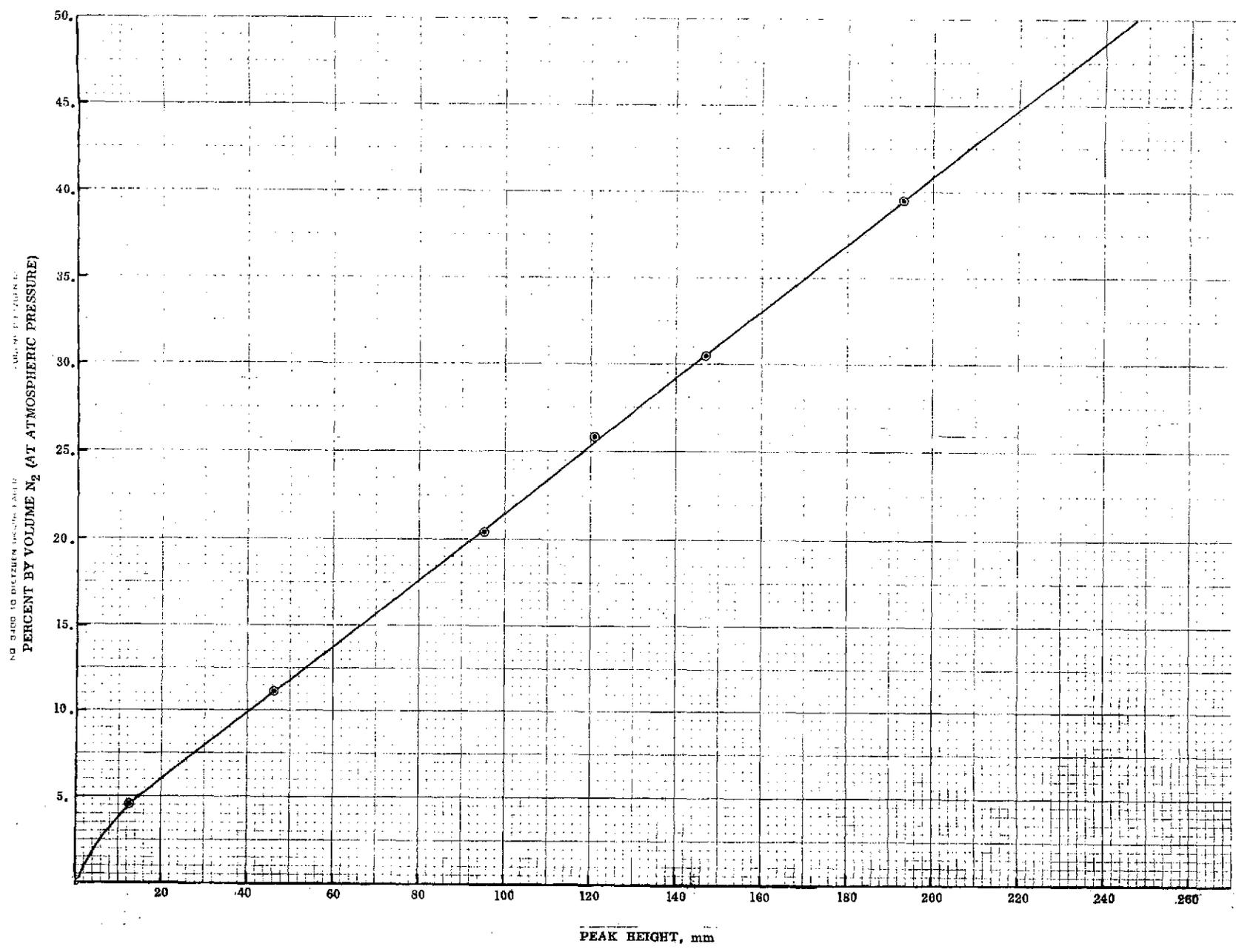


Figure 12



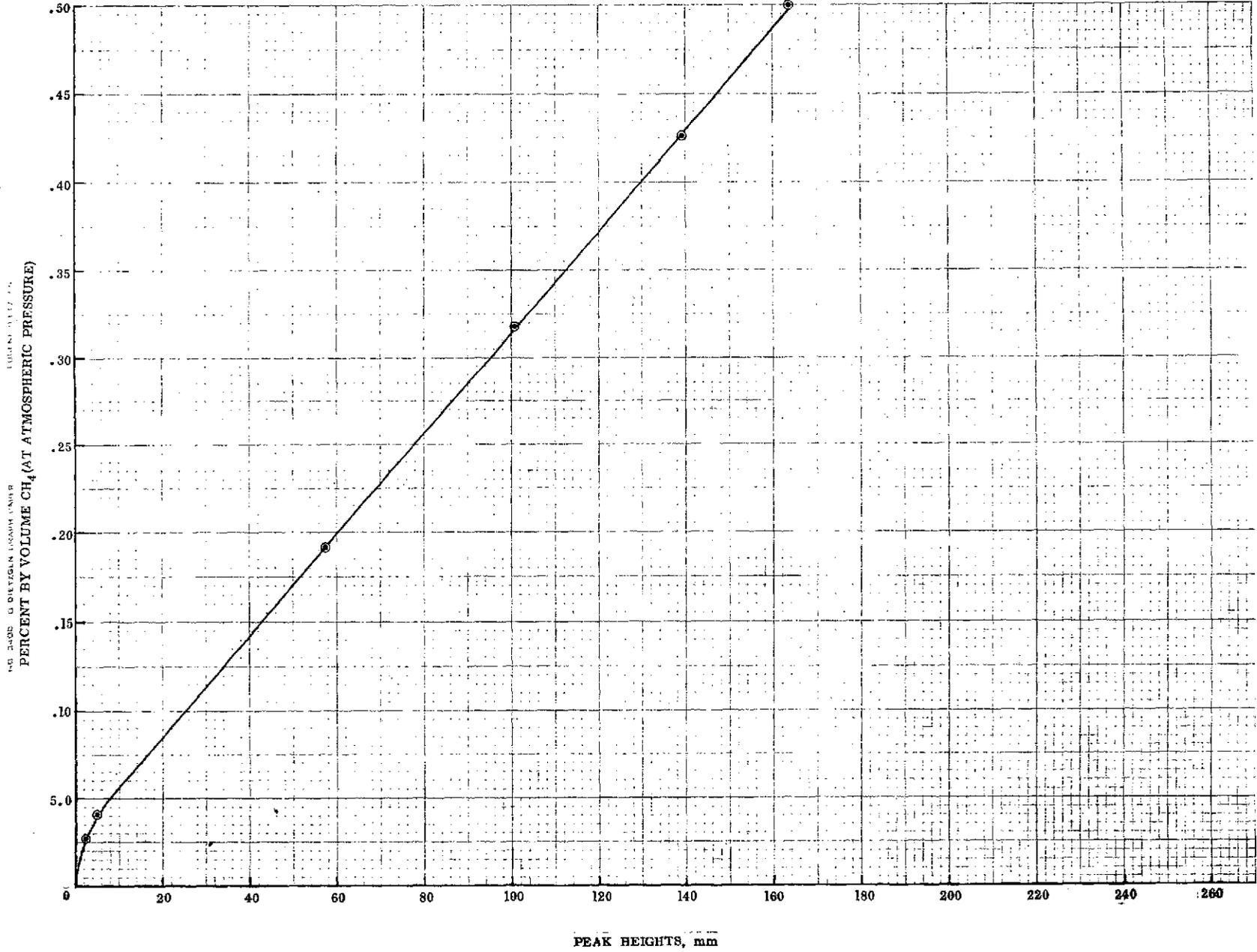
O₂ CALIBRATION CURVE, 300 SERIES - BATCH TEST

Figure 13



N₂ CALIBRATION CURVE, 300 SERIES - BATCH TEST

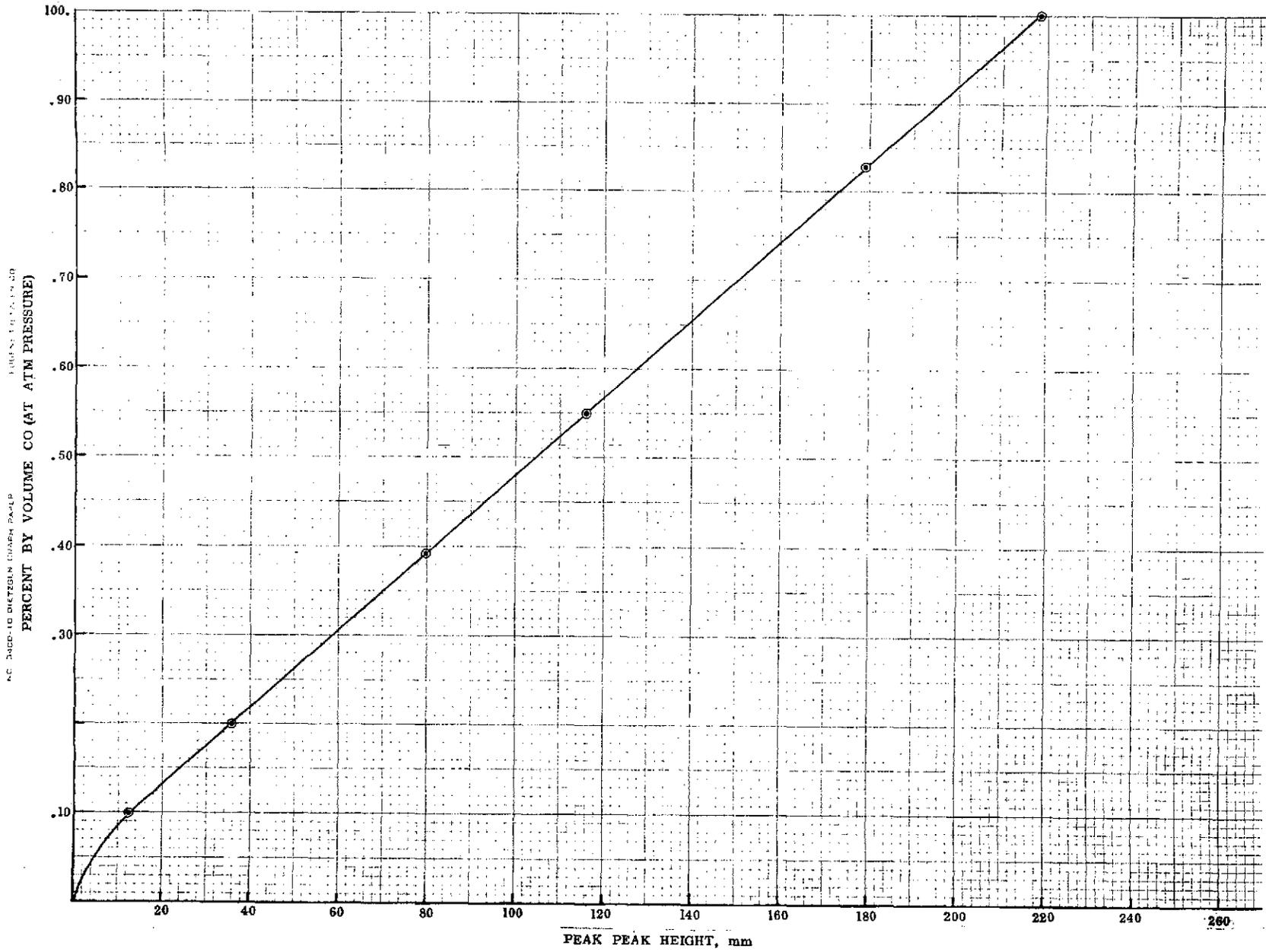
Figure 14



100-3400 G. DIEZELIN LINARIM CANIER
EUGENE DIEZEL S.A.
PERCENT BY VOLUME CH₄ (AT ATMOSPHERIC PRESSURE)

CH₄ CALIBRATION CURVE, 300 SERIES - BATCH TEST

Figure 15.



AC 3462-10 DIRECT-READING ANALYZER
SERIES 300

CO CALIBRATION CURVE, 300 SERIES - BATCH TEST

Figure 16

Temperature Measurement

Chromel - alumel thermocouples were used for temperature measurement. The selection of thermocouples was performed in the same manner as with the small scale tests. The thermocouples then welded to the locations indicated on Figure 9. Temperature readout was performed on a Bristol Dynamaster 0 to 1500°F multi-point recorder.

Other Analysis Techniques

In addition to the routine chromatographic gas analysis, numbers of other analytical techniques were employed as listed below.

- Hydrogen sulfide (H₂S) detection - Performed by Mine Safety Appliances colorimetric sample tube.
- Mercaptan (RSH) detection - Performed by Mine Safety Appliances colorimetric sample tube.
- Sulfur dioxide (SO₂) analysis - Performed by Mine Safety Appliances colorimetric sample tube.
- Ammonia (NH₃) analysis - Performed on water condensate by a Nessler's solution spectrophotometric technique.
- Carbonate (CO₃⁼) analysis - Performed on water condensate by a barium chloride solution forming a barium carbonate precipitate.
- Halogen identification - Performed on water condensate with silver nitrate solution producing silver halogen precipitate. Silver carbonate interferences were avoided by acidification.
- pH measurement - Performed on water condensate by a Leads and Northrup Model No. 740-1 pH meter. pH calibration standards as well as pH paper served to check meter operation.
- Electrical conductivity measurement - Performed on water condensate by an Industrial Instruments, Inc., Model No. RC 1632 conductivity bridge. Meter performance was checked periodically with deionized water.

TEST PROCEDURE

Before each test run the reactor was "steam cleaned" to remove the residue of the previous run. After the product gas generation rate had reached essentially zero a helium flush was added to the reactor to reduce the chromatographic reading. Next the temperature in the gasifier was lowered to 204°C (400°F), the boat from the previous run removed, and a weighed sample, usually either 10 gm or 40 gm,

TABLE IX
LARGE SCALE TEST PARAMETERS RECORDED FOR EACH SAMPLE POINT

Total Test Data Recorded	Units	Test Parameters Used In Data Reduction Program	Units
Test Number	-	Test Number	-
Run Time	Seconds	Run Time	Seconds
Reactor Outlet Pressure	PSIA	Reactor Outlet Pressure	PSIA
Reactor Thermocouple #1 (See figure 14 for location)	°F		
Reactor Thermocouple #2 (See figure 14 for location)	°F		
Steam Feed Line			
Thermocouple	°F		
Reactor Thermocouple	°F	Reactor Thermocouple #4 (See figure 14 for location)	
Reactor Thermocouple #5 (See figure 14 for location)	°F		
Reactor Thermocouple #6 (See figure 14 for location)	°F		
Converter Reaction Section #1			
Thermocouple No. 7	°F		
Thermocouple No. 8	°F		
Converter Reactor Section #2			
Thermocouple No. 9	°F		
Thermocouple No. 10	°F		
Converter Reactor Section #3			
Thermocouple No. 11	°F		
Thermocouple No. 12	°F		
Test Meter Volume	Ft ³	Test Meter Volume	Ft ³

TABLE IX
LARGE SCALE TEST PARAMETERS RECORDED FOR EACH SAMPLE POINT
(Continued)

Total Test Data Recorded	Units	Test Parameters Used In Data Reduction Program	Units
Volume Water Condensed	ml	Volume Water Condensed	ml
Condensed water pH	-	Condensed water pH	-
Condensed Water Conductivity	mho/cm	Condensed Water Conductivity	mho/cm
Laboratory Temperature	°F	Laboratory Temperature	°F

contained in a screen boat was installed in the gasifier reactor. The access door was then quickly replaced and the test started. The gasifier was heated to the conversion reactor temperature, which usually took a period of two to three hours. The boat and sample were held at the conversion reactor temperature for up to 48 hrs. while steam continued to pass through the reactor.

Test data was recorded every 800 seconds (13.3 minutes) on a number of test variables. This was necessary to collect proper experimental data, although only a portion of this information was used in the data reduction program. Table IX lists all the test parameters which were recorded and also those which were used as input information for the computer data reduction program.

COMPUTER DATA REDUCTION

The same program used for data reduction in the small scale continuous tests was modified for usage in the batch tests. Modifications included elimination of the comparing of feed to production rates as the carbon recovery calculational technique. Instead, carbon recovery was calculated by a ratio of feed sample weight and a determination of the percent of carbon recovered at each sample point. Sample point data was collected every 800 seconds (13.3 minutes) and input into the computer program for calculations. Generally, 8 to 15 sample points constituted a test run. Information printed out in the data program is given in Table X. A sample of the computer print-out sheet is shown in Appendix B.

TABLE X
SUMMARY OF LARGE SCALE - BATCH COMPUTER DATA REDUCTION
Note: Sample printout sheet is given in Appendix B

Data	Units	Remarks
Sample Number	-	-
Run Time	Hours	Calculated from input in seconds.
Reactor Temperature	°F	Temperature at reactor midpoint.
Volume Collected	ml	Condensed water collected.
Gas Generation Rate	ml/min	Measured with wet test meter.
Reactor Volume	ml	Calculated volume of reactor.
Density of Steam	g/ml	Used in space velocity calculation.

TABLE X
SUMMARY OF LARGE SCALE - BATCH COMPUTER DATA REDUCTION (CONT'D)
Note: Sample printout sheet is given in Appendix B

Data	Units	Remarks
Set Conditions		
Liquid feed rate	ml/min	Steam rate set by metering valve.
Space velocity	1/hour	Calculated as liquid feed rate - set multiplied by steam density at reactor temperature divided by reactor volume.
Contact Time	seconds	Reciprocal space velocity.
Measured Conditions		
Liquid feed rate	ml/min	Same as for set conditions.
Liquid collect rate	ml/min	Calculated from volume collected and run time from current and previous sample.
Space velocity		Calculated as summarized in section "converter" reactor rate treatment"
Contact Time		Reciprocal space velocity-measured.
Reactor Pressure	psia	Reactor outlet pressure - measured.
Reactor Pressure	atm	Calculated from above.
Chromatographic Peaks (for H ₂ , CO ₂ , CH ₄ , H ₂ O)		Measured values.
Partial Pressure	atm	Calculated by linear interpolation of calibration curves - inputted as a table.
Partial Pressure Total	atm	The sum of the calculated constituent partial pressures.

TABLE X

SUMMARY OF LARGE SCALE - BATCH COMPUTER DATA REDUCTION (CONT'D)

Note: Sample printout sheet is given in Appendix B

Data	Units	Remarks
Mole Fraction	-	Calculated as constituent partial pressure divided by the sum of the partial pressure.
Predicted Mole Fractions	-	Values predicted from UAC thermodynamic equilibrium program.
Predicted Mole Fractions (H ₂ O Free)		Calculated from predicted mole fraction by summing all constituent mole fractions, except H ₂ O and dividing end by the sum.
Predicted Partial Pressures	atm	Predicted mole fraction multiplied by expartial total pressure.
Predicted Partial Pressures (H ₂ O Free)	atm	Calculated from predicted partial pressures by dividing partial pressure by partial pressure sum, excluding H ₂ O.
Experimental/Predicted Mole Fraction	-	Ratio of experimented value to predicted multiplied by 100.
Experimental/Predicted Mole Fractions (H ₂ O Free)	-	Ratio of experimented value to predicted multiplied by 100.
Experimental/Predicted Partial Pressures	-	Ratio of experimental value to predicted multiplied by 100.
Experimental Predicted Partial Pressures (H ₂ O Free)	-	Ratio of experimental value to predicted multiplied by 100.

TABLE X
SUMMARY OF LARGE SCALE - BATCH COMPUTER DATA REDUCTION (CONT'D)
Note: Sample printout sheet is given in Appendix B

Data	Units	Remarks
BATCH REACTOR		
Rate of Carbon Generation	gm atom/min	Calculated from gas generation rate, ml/min multiplied by gm-atom/ml carbon obtained from chromatographic analysis and conversion factors.
Total Carbon Fed	gm-atom	Calculated from feed composition rate, and composition factors.
(Generation/Feed) Ratio	percent	Ratio above two calculations. Termed "Carbon Recovery" in text.
Rate of Hydrogen Generation	gm-atom/min	Calculated from gas generation rate, ml/min multiplied by g-atom/ml hydrogen obtained from chromatographic analysis and conversion factors.
Total Hydrogen Fed	gm-atom	Calculated from feed composition and feed factors.
(Generation/Feed) Ratio	percent	Ratio of above two calculations. Termed "Hydrogen Recovery" in text.
Rate of Oxygen Generation	gm-atom/min	Calculated from gas generation rate, ml/min multiplied by g-atom/ml hydrogen obtained from chromatographic analysis and conversion factors.
Total Oxygen Fed	gm-atom	Calculated from feed composition and rate factors.
(Generation/Feed) Ratio	percent	Ratio of above two calculations. Termed "Oxygen Recovery" in text.

TEST RESULTS

Synthetic Wastes

The synthetic waste tests as well as the other batch test runs are summarized in Table X. Listed after the test run number is the chemical tested. The weight of sample given represents the dry weight of waste introduced into the reactor. For the spacecraft wastes the number represents the total weight of feces, urine, paper, and plastic. The converter reactor temperature is given in the next column. For these tests the gasifier was heated during the test run from 204°C (400°F) to the converter reactor temperature. Contact times calculated for each sample point were averaged to obtain the values in the table.

The percent of sample weight loss after a total run time of usually 48 hours is shown in the next column. The carbon recovery represents the percent of carbon in the feed that is recovered as gases collected over a time period indicated in the column "analysis time". Note that only 20 to 75 percent of the feed carbon was recovered in the 2 to 4 hour run time. In most tests carbon recovery closely approached 100 percent after 24 to 48 hours run time. The composition of gases generated represents the percent of gases obtained for sample point taken at run time equal to the "analysis time". The percent of theoretical composition represents a comparison of actual composition to that which is predicted by equilibrium values obtained from a United Aircraft Computer Program.

Indicated in the next column, "analysis time" is the time into the run where the rate data analysis was conducted. (See "converter reactor rate treatment"). Product water quality is given in last two columns as odor and color.

Computer plots of twelve test and data components are presented in Appendix C. All 300 series tests are summarized in this Appendix. The components listed in Table XIII are plotted versus run time (in hours).

The tests were conducted in the order listed in Table XII, with the 650°C (1400°F) runs conducted last because of uncertainties in rig and heater element life time at elevated temperatures. A test run was conducted approximately every other day, with the day in-between used for data reduction. The tests were conducted over a period of about 2 to 5 months.

Elevation of the converter and final gasifier temperature to 760°C (1400°F) increased the total sample weight loss. (See Runs 301 to 310 versus 311 to 317 (except 315) in Table XI.) However, only a small change is noted for these in the composition of gases generated and the percent carbon recovery. Methane and carbon monoxide demonstrated the greatest change in composition with temperature elevation.

TABLE XI

TEST RUN SUMMARY - 300 SERIES TESTS

BATCH TEST REACTOR

Test Run No.	Chemical	Converter Reactor Temp.		Average Contact Time (sec)	Sample Weight Loss (%) ⁽³⁾	Carbon Recovery ⁽⁴⁾ as Gases (%)	Composition of Gases ⁽⁴⁾ Generated (%)					Percent of Theoretical Composition ⁽⁴⁾					Analysis Time (hr)	Water Quality	
		°C	°F				H ₂	CO ₂	N ₂	CH ₄	CO	H ₂	CO ₂	N ₂	CH ₄	CO		Color	Odor
301	43.1g cellulose	650	1200	61.4	82.8	35.6	41.6	9.6	9.2	12.4	19.6	64.8	33.4	--	11383.	278	3.29	Clear	Slight
302	44.8g methionine	650	1200	45.7	100.	44.0	41.1	10.0	6.2	9.4	1.6	14.1	46.0	218.	60.2	24.3	2.49	Brown	Strong
303	37.8 cellulose & 2g KIO ₃	650	1200	57.2	84.4	38.8	51.7	12.8	10.8	3.6	7.6	80.6	44.6	--	3308.	108.	3.46	Clear/brown	Slight
304	40g polyethylene	650	1200	60.0	100.	98.6	30.8	20.1	6.5	17.7	0	43.7	143.	--	542 ⁽⁵⁾	0.	3.24	Clear	Slight
305	10g cellulose	650	1200	47.9	100.	35.1	41.8	19.9	13.6	9.21	1.6	63.5	62.9	--	∞	66.5	3.18	Clear	Very slight
306	10g cellulose	650	1200	14.2	88.9	47.8	45.9	16.8	16.8	8.7	9.6	63.1	46.3	--		2032.	3.34	Clear	Slight
307	10g polyethylene	650	1200	12.	96.0	31.5	29.3	22.4	6.1	14.1	0	39.2	92.4	--		0.	3.11	Clear/brown	Very slight
308	40g cellulose	650	1200	12.6	96.0	36.3	52.6	13.0	5.9	11.4	0	79.1	39.4	--		0.	3.21	Clear/slightly cloudy	Slight
309	10g methionine	650	1200	46.6	99.0	68.4	35.0	14.0	6.2	13.9	.8	53.8	56.9	232.		36.9	2.45	Clear/slightly yellow	Moderate
310	40g cellulose	650	1200	4.51	90.4	27.7	42.8	7.3	7.8	18.2	22.9	64.6	22.6	--		2032.	2.79	Clear/slightly yellow	Slight
311	40g cellulose	760	1400	3.90	99.0	38.1	52.6	11.3	5.2	12.0	8.5	80.0	35.2	--		468.	2.90	Clear/colorless	Almost none
312	10g cellulose	760	1400	3.78	97.0	42.3	38.3	14.6	9.2	6.9	18.5	57.7	44.6	--		31.8	2.45	Clear/colorless	Almost none
313	10g polyethylene	760	1400	3.93	99.0	35.5	36.9	17.8	13.3	15.1	17.6	40.6	93.6	--		104.	3.11	Clear/slightly yellow	Slight
314	10g methionine	760	1400	3.50	99.0	76.3	45.6	6.4	10.2	10.9	4.4	69.7	24.8	385.		6.9	2.45	Clear/cloudy	Slight/moderate
315	10g spacecraft mixture (1)	760	1400	12.1	99.0	34.6	51.9	12.8	10.5	5.2	1.6	79.9	40.5	6939.		603.9	3.34	Clear/colorless	Slight
316	10g cellulose	760	1400	11.9	99.0	38.6	44.3	21.3	8.2	7.0	6.0	66.8	64.9	--		1033.	2.45	Clear/colorless	Very slight
317	40g cellulose	760	1400	11.3	98.3	41.1	54.9	9.9	1.8	7.6	6.4	84.0	32.2	--		1651.	2.93	Clear/yellow	Moderate
318	40g synthetic mixture (2)	760	1400	10.5	100.	38.3	52.1	14.8	1.47	0	11.5	80.3	48.1	920.4		782.	2.90	Clear/yellow	Strong
319	40g polyethylene	150	300	12.0	100.	.69	10.9	5.0	2.0	8.9	0	14.9	26.0	--		0	2.45	Clear	Slight
320	40g cellulose	150	300	12.0	100.	23.4	51.4	8.7	.4	2.2	20.7	77.5	26.4	--		3560.	3.34	Dark brown	Sweet carnal
321	40g cellulose	150	300	--	78.5	0	0	0	0	0	0	0	0	--		0	2.66	--	--
322	40g spacecraft mixture (1)	760	1400	10.8	100.	31.7	56.5	17.8	0	0	0	87.1	58.0	0		0	3.34	Clear/cloudy/yellow	Slight
323	40g spacecraft mixture (1)	760	1400	10.6	97.5	24.8	53.5	18.8	0	0	0	82.5	61.3	0		0	3.34	Clear/cloudy/yellow	Strong

- (1) 25% by weight each of fresh feces, urine, paper toweling, and plastic. Note: Run 322 contained 2gm Na₂CO₃ and 2gm. RuCl₃ x H₂O (40% Ru)
- (2) 3.6 gm methionine, 24 gm cellulose, 12 gm polyethylene, 0.4 gm potassium iodate.
- (3) After 24-48 hours.
- (4) Results based on a run time shown in column headed "Analysis Time".
- (5) ∞ indicates some gases generated though none was predicted.
- (6) N₂ observed through test series is believed due to air impurity entering reactor when loading sample. Very little O₂ was observed.
- (7) Steam generation was not employed in Test 321 in order to compare gasification with (Test 320) and without (Test 321) steam.
- (8) These tests conducted at a higher than average heating (to reach converter temperature in approximately 1 hour versus about 2 to 2 1/2 hours for the other tests.

TABLE XII
TEST SUMMARY COMPONENTS COMPUTER PLOTTED AND LISTED
IN APPENDIX C

Test Component	Units
Gasifier temperature (sample boat)	°F
Gas volume generated	liters
Gas generation rate	ml/minutes
Total pressure analyzed gas	atom
Percent carbon recovery	-
Percent hydrogen recovery	-
Percent oxygen recovery	-
Product water - electrical conductivity	mho/cm
Gm-atom carbon generated per minute	gm-atom/minute
Gm-atom hydrogen generated per minute	gm-atom/minute
Gm-atom oxygen generated per minute	gm-atom/minute
Product water pH	
These components are plotted versus:	
Run time	hours

Cellulose

Cellulose was selected as the base material for extensive testing, as it possessed reactivity similar to what might be expected with real waste mixtures. Without a catalyst, cellulose was found to gasify rapidly, to form some non-volatile char, and to liberate water vapor when subjected to reactor conditions. The fact that cellulose liberates water from its chemical structure when heated, was of interest as water from the interior of the chemical itself would be available for reaction. However, water from the steam boiler would be required to diffuse through the surface of the sample to react. Thus, dry cellulose might be expected to react in a manner similar to a wet solid waste sample.

Cellulose is suggested as a possible guideline for interpretation and design usage. For example, the sample weight loss over 24 to 48 hours was 98.3 percent for cellulose (Run 317) and 97.5 percent (Run 323) for the spacecraft mixture. However, the percent carbon recovery for the spacecraft mixture was lower than that obtained for cellulose (24.8 versus 41.1). Therefore, caution is indicated in the use of test data.

The runs conducted on pure, dry cellulose indicated high reactivity in the presence of steam, producing gases and char once "ignition" had occurred. This occurred at 354°C (670°F) for run 301 (40 gram sample). For next 40 gram cellulose sample tested, run 308, "ignition" occurred at a lower temperature of 288°C (550°F) (see test summary, Appendix C). This lower ignition temperature is believed due to the slower sample heating rate employed in all but the first tests. A higher heating rate was used in the first four tests than the latter ones. The slower sample heating rate generally means lower gas generation rate from the gasifier, longer converter contact time, resulting in higher quality product water and gas. Run 312, run at 760°C (1400°F), produced the highest quality water collected which was clear, colorless and had almost no odor.

All cellulose runs, except run 314, displayed larger quantities of CO than predicted. It appears that increasing water also lowers the CO level. Presumably, CO initially is formed from char as indicated in reaction 4, then when given the time and temperatures to react, the CO is converted to CO₂ as suggested by reaction 7.

Polyethylene

Polyethylene always demonstrated high volatility in these tests. All runs indicated a sample weight loss of 97 to 100 percent, with most results very close to 100 percent. With run 304 the carbon recovery, evaluated at a run time of 3.24 hours, was 98.6 percent. These conditions appear to be optimum for polyethylene. Note the heating rate was fairly high for this run (see Appendix C) as compared with the later polyethylene runs. Also, the average contact time was 60 seconds and the final temperature was 650°C (1200°F). Product water was clear and with a slight odor. Under these conditions, 43.7 percent of the predicted H₂, 143 percent of the predicted CO₂, and 549 percent of predicted CH₄ was observed. Carbon monoxide was predicted to be present although none was observed. However, when the temperature is elevated to 760°C (1400°F), slightly more than the predicted amount of CO was observed, 104 percent (Run 313). Presumably, the carbon monoxide observed is due to the reverse of reaction 7 at the higher temperature.

Cellulose + Potassium Iodate (KIO₃)

The addition of KIO₃ to cellulose can be seen in run 303 versus 301. A slightly greater sample weight loss was observed (84.4 versus 82.8 percent) when KIO₃ is present. Also, a slightly greater carbon recovery (38.8 versus 35.6 percent) was observed in the 3.3 hour run time. It may be possible that KIO₃ has some catalytic activity with cellulose. It is known that when KIO₃ is heated to 100°C (212°F), it is decomposed to

KI, liberating O₂ gas. When KI is heated to 560°C (1040°F), it melts and could be evaporated from the boat. As iodine ion (I⁻) was detected in the product water, it was, therefore, carried through the reactor as KI gas or other iodine compound. It can be seen in Table X that the yield of H₂ and CO₂ is increased when KIO₃ is present. The H₂ percent of theoretical composition increased from 64.8 to 80.6 percent and CO₂ from 33.4 to 44.6 percent, which CO₂ was reduced from 278 percent to 108 percent. These results suggest that some chemical form derived from KIO₃ is influencing the final reaction products. In particular, the lower level of CO and higher level of H₂ and CO₂ suggests a catalytic effect upon "the water gas shift" reaction 7.

Methionine

Methionine demonstrated high reactivity in all tests. All runs with pure methionine indicated at least a 99 percent sample weight loss over the 24 to 48 hour run time. Water quality was always lower for the methionine than other test materials which was attributed to the sulfur and nitrogen in its chemical makeup.

The chromatographic analysis for N₂ is shown in Table XI. This number varied almost randomly for the 300 series runs whether or not nitrogen was present. As air is able to enter the reactor when the sample is introduced, the nitrogen readings are assigned as an impurity from air. Methionine runs always displayed a low value for the percentage of the predicted CO. It is possible that CO is not formed directly from the solids in the boat, but rather from gases reacting in the converter.

Sulfur Analysis

The effluent product gas stream was analyzed periodically for sulfur compounds. Mine Safety Appliances colorimetric detector tubes were used to measure hydrogen sulfide (H₂S), sulfur dioxide (SO₂), and mercaptans - organic sulfides. The mercaptan was analyzed as butyl mercaptan; however, a mixture of mercaptans was present.

In all of the runs containing the sulfur bearing protein methionine (C₅H₁₁O₂NS), the H₂S level was greater than the maximum measurable concentration of the detector tube (3200 ppm). The real spacecraft mixture displayed lower maximum level ranging from 20 to 200 ppm. As can be seen in Table XIII, the feed sulfur levels were considerably greater for the methionine than the values estimated for the real spacecraft mixture, which were calculated from a NASA document (Reference 1).

No SO₂ was observed in any test runs. Some mercaptan (100 to 160 ppm) was observed for these tests. The thermodynamics predict that essentially all of the sulfur should be present as H₂S in the product stream, with no SO₂ or mercaptans present.

TABLE XIII
 MAXIMUM SULFUR LEVELS DETERMINED IN PRODUCT GAS STREAM

Run Number	Chemical	Sulfur Level in Feed, Grams	Hydrogen Sulfide ppm	Sulfur Dioxide ppm	Mercaptan ppm
309	10 gm methionine	2.15	3200	*	*
314	40 gm methionine	8.60	3200	*	2
315	10 gm ⁽¹⁾ Spacecraft mixture	0.0025 ⁽⁵⁾	200	0	100
318	40 gm ⁽²⁾ Synthetic mixture	0.77	3200	0	160
322	40 gm ⁽³⁾ Spacecraft mixture	0.01	20	0	100
324	44 gm ⁽⁴⁾ Spacecraft mixture	0.01	35	0	100

* Not conducted

- (1) - 2.5 gm feces, 2.5 gm urine, 2.5 gm paper toweling, 2.5 gm polyethylene, 0.5 gm $\text{RuCl}_3 \times \text{H}_2\text{O}$ (40% Ru)
- (2) - 3.6 gm methionine, 24 gm cellulose, 12 gm polyethylene, 0.4 gm potassium iodate
- (3) - 10 gm feces, 10 gm urine, 10 gm paper toweling, 10 gm polyethylene 2 gm N_2CO_3 , 2 gm $\text{RuCl}_3 \times \text{H}_2\text{O}$ (40% Ru)
- (4) - 10 g feces, 10 gm urine, 10 gm paper toweling, 10 g polyethylene
- (5) - Estimated from Reference (1).

Halogen Analysis

Product water from test runs containing halogen compounds in the feed makeup was analyzed for the presence of chloride ion. A white precipitate of AgCl or AgI would be formed by the addition of a silver nitrate solution (AgNO_3) to the water samples. Table XIV summarizes the results for runs containing halogens. Note that all these tests confirmed the presence of chloride ion. Also, the product water in these runs was acid ($\text{pH} < 7$). It is concluded that the halogen is converted to the acid gas (HCl or HI) and then condensed in the product water. Thermodynamic predictions indicate iodine to be split about equally between HI and I_2 . No chemical measurements were conducted on I_2 .

TABLE XIV
TESTS FOR PRESENCE OF HALOGEN COMPOUNDS

Run Number	Chemical	Halogen Level in Feed, gm	Halogen Present in Product Water	Lowest pH Reading of Product Water
303	37 gm Cellulose 2 g K103	1.2	Yes	4.8
315	10 gm Spacecraft ⁽¹⁾ mixture	0.224	Yes	2.5
322	40 gm Spacecraft ⁽²⁾ mixture	2.22	Yes	2.2
3.23	44 gm Spacecraft ⁽³⁾	0.07 ⁽⁴⁾	Yes	5.5

- (1) - 2.5 gm feces, 2.5 gm urine, 2.5 gm paper toweling, 2.5 gm polyethylene, 0.5 gm $\text{RuCl}_3 \times \text{H}_2\text{O}$ (40% Ru)
- (2) - 10 gm feces, 10 gm urine, 10 gm paper toweling, 10 gm polyethylene, 2 gm Na_2CO_3 , 2 gm $\text{RuCl}_3 \times \text{H}_2\text{O}$ (40% Ru)
- (3) - 10 gm feces, 10 gm urine, 10 gm paper toweling, 10 gm polyethylene
- (4) - Estimated from Reference (1)

Spacecraft Waste Tests

Alkali carbonates have been studied as coal (2), char (3), and waste (4) gasification catalysts. As char was observed to be formed during the earlier cellulose test runs, two grams of sodium carbonate, Na_2CO_3 , were added to the feed mixture for run 322 for catalyst evaluation. This was compared with Run 323 which contained no Na_2CO_3 . Table XV summarizes the results of these two runs.

TABLE XV
CHAR GASIFICATION CATALYST TEST RESULTS

Run	Catalyst	Percent Fed Carbon Recovered as Gases*	Percent Rate Increase in Char Gasification
322	5% by wt. Na ₂ CO ₃	31.7	31.8
323	None	24.8	-

* In run time of 3.34 hours

As can be seen from Table XV, Na₂CO₃ increases the rate of gasification (percent of fed carbon recovered) by 31.8 percent.

It is worthwhile to note that Appell(3) suggested a gasification mechanism for the alkali carbonate catalyst of char requiring the presence of a liquid water phase. However, this work as well as the research of Cox(4) demonstrates carbonate catalysis in the absence of a liquid phase.

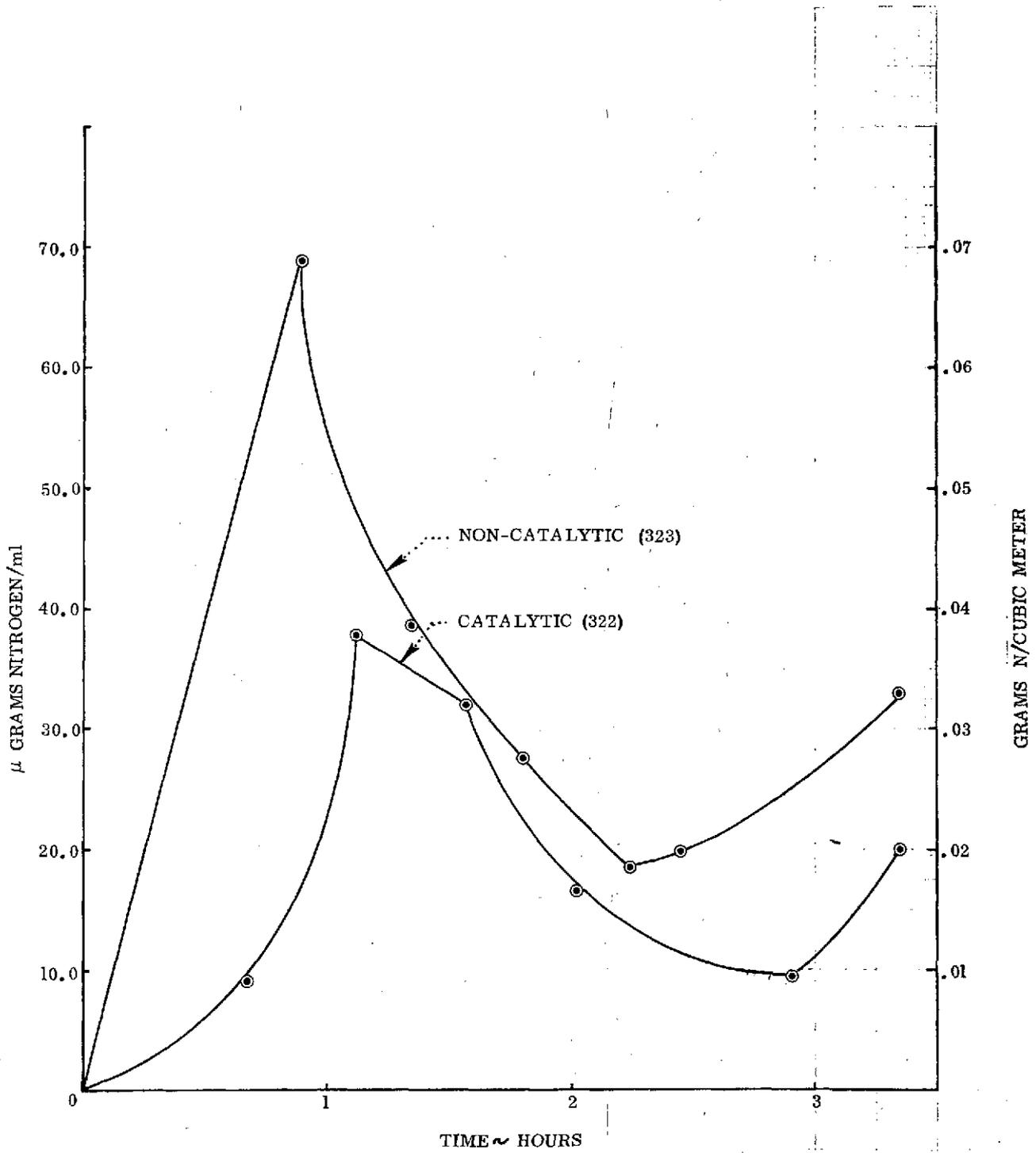
Post Run Analysis

After the test run, overnight heat at 760°C (1400°F), the residue was found to weigh 2.6 grams. All but 0.8 grams of the residue was readily soluble in water. The insoluble residue was a grey metallic substance. As two grams of RuCl₃ x H₂O (40 percent Ru), or 0.8 grams Ru, had been added in the feed it appears the only insoluble residue was metallic ruthenium (see following section).

The remaining water soluble 1.8 grams probably represent the original 2.0 grams of Na₂CO₃, with possibly some loss as NaOH (a liquid at these temperatures). The solution resulting from dissolving the residue contained carbonate, as demonstrated by a precipitation reaction with a BaCl₂ solution, and the pH was 12, which is consistent with that of a concentrated Na₂CO₃ solution.

Ammonia Catalyst

Two grams of RuCl₃ x H₂O (40.0 percent Ru) were mixed with the spacecraft mixture and tested in Run 322 at 12 second contact time and 204°C (400°F). Test Run 323 did not contain the ruthenium catalyst. Liquid condensate samples taken periodically during both runs were analyzed for ammonia with Nessler's solution. The concentration of ammonia, expressed as grams nitrogen/cubic meter of solution (μ grams nitrogen per ml) is shown in Figure 17 for these two runs. The area under these curves multiplied by the total volume of water condensed during this time period, represents the total grams of nitrogen generated (analyzed as NH₃). For both runs, the total volume



AMMONIA ANALYSIS OF RUNS 322 AND 323

FIGURE 17

of condensed water was 9.72×10^6 mm³ (972 ml). This gives 0.0538 gram nitrogen in the condensate for the catalyzed reaction and 0.103 grams for the uncatalyzed reaction.

The estimated nitrogen level in the feed was calculated as follows using average nitrogen levels given by reference (1) as:

Average nitrogen in feces = 0.7 percent by weight

Average nitrogen in urine = 1.1 percent by weight

Therefore, a 40 gram sample of waste consisting of 10 grams feces and 10 grams urine would contain a total of $0.07 + 0.11 = 0.18$ grams nitrogen. As seen in Table XVI, the catalyst was able to lower the level of NH₃ from 0.103 to 0.0535 presumably converting it to N₂ gas. This yields a catalyst efficiency of 51.9 percent in reducing the NH₃ level when used at 2 grams catalyst per 40 grams waste, or 5 percent ruthenium by weight.

Most chromatographic traces, including runs where no nitrogen was present in the feed, displayed trace levels of N₂ and many times trace levels of O₂. This is because the batch reactor had to be opened after the helium flush to introduce the sample, thus permitting air to enter the reactor. Also, the nitrogen gas generated would be expected to be less than 0.16 percent by volume (see Appendix A). Since the chromatograph was programmed to read nitrogen in the high percent range, this reading would be below its detectability limit.

Special Tests

Three tests were conducted near the end of the large scale tests to provide an understanding of the mechanisms of waste gasification and conversion. It was of primary interest to clarify the role of water in (1) gasification of waste, and (2) its effect on conversion.

Special Test Number 319

To measure the gas products evolving directly from the gasifier, the temperature of the conversion was lowered to 150°C (300°F) thus serving only to transport gases to the exit of the condenser for analyses, and not to change them chemically. Therefore, any non-condensable gas produced in the gasifier would be transported directly to the chromatograph for analysis; condensable gases would be deposited on the wall of the converter and/or condensed in the product water. The polyethylene sample was tested because it contains no oxygen in its makeup; therefore, oxides of carbon generated would identify water as being involved in the initial gasification.

TABLE XVI

AMMONIA CATALYST - TEST RESULTS

Run No.	Catalyst	Area Under Curve (1)		Volume H ₂ O Condensed		Grams Nitrogen (as NH ₃) in H ₂ O	Estimated Nitrogen in Waste	% Nitrogen Recovered as NH ₃	Catalyst Efficiency (%)
		gm Nitrogen/ cu. meter soln.	(gm Nitrogen/ ml soln.)	cu. meters	(ml)				
322	RuCl ₃ · x H ₂ O	55.	(55.)	9.72 x 10 ⁻⁴	(972)	.0535	.18	29.7	51.9%
323	None	106.	(106.)	9.72 x 10 ⁻⁴	(972)	.103	.18	57.2	--

(1) Calculated for a run time of 3.34 hours.

Test Run No. 317 was repeated as Run 319 with the converter at low temperature, but with the usual steam flow rate of 4.85 g/min. Table XI indicates almost no gas generation and very little CO₂ and no CO as products. The CO₂ was observed only after the gasifier had reached 700°C (1400°F). Essentially no gases were generated in the 315 to 400°C (600 to 750°F) range as observed in Run 317.

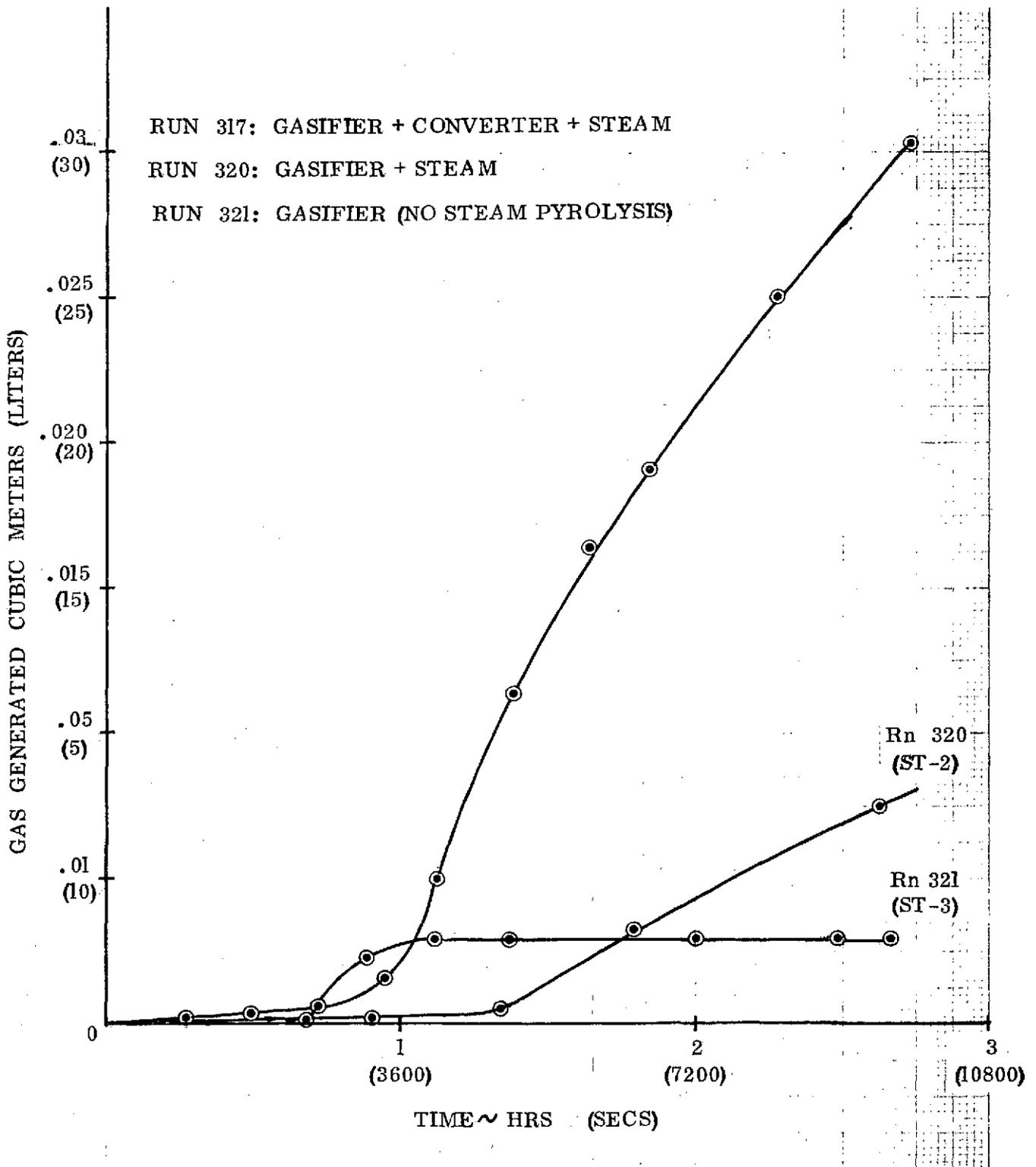
Because CO and CO₂ are observed when the converter is at reactor temperature, water must be chemically involved in the high temperature reaction. When the converter temperature is lowered to 200°C (400°F), no CO or CO₂ is observed; therefore, lowering the temperature quenched the role of water. As essentially no gases were generated, even when all the sample was volatilized from the boat, it is concluded that water has no significant role in the gasification of polyethylene in the temperature range of 260 to 538°C (500 to 1000°F).

Special Tests Number 320 and Number 321

Because polyethylene in the presence of water vapor demonstrated only condensable gas generation from the gasifier, testing polyethylene in the absence of steam was of less interest than testing another material. Therefore, for the last two special tests cellulose was heated in the presence (Run 320) and then the absence (Run 321) of steam. As before, the conversion reactor was held at a low temperature, 150°C (300°F), so not to chemically influence the gasifier reaction products.

For comparison, Run 317 (cellulose at 12 seconds contact time) produced 30,300 mm³ (30.3 liters) of gas at 2.71 hours run time. Table XVII shows that cellulose without the converter reactor produces only 7500 mm³ (7.5 liters) or 24.8 percent as much gas in the same run time. Pyrolysis of cellulose, Run 321 produced only 2290 mm³ (2.29 liters) or 1.31 percent of the gases in this run time. These facts suggest that water is involved in the role of cellulose gasification to some extent.

With pyrolysis, the reaction rate falls to zero when the sample has been raised to only 316°C (610°F) (Run 321, Appendix C). No more gas is generated even as the temperature is raised to 760°C (1400°F). But, both Run 317 and Run 320, with steam present demonstrated the rapid low temperature reaction (see Runs 317 and 320, Appendix C) as well as some reaction occurring at higher temperatures in the range of 427 to 650°C (800 to 1200°F). In fact, Run 320 demonstrated more gases formed at the higher temperature than at the lower temperature. Figure 18 shows that the quantity of non-condensable gases generated for Run 317 was always significantly greater than that for Run 321. In fact, with Run 321 the quantity of gas generated, 2290 mm³ (2.29 liters), was less than the volume required to flush the purge helium from the 4183 mm³ (4.183 liters) reactor. Therefore, curves for Run 321 in Appendix C displayed only the gas generation rate and the gas volume generated. Since no reaction gases reached the chromatograph, no information is recorded regarding the pressure and percent carbon, hydrogen, and oxygen recovery. Table XI indicates almost complete weight loss when cellulose is heated in the presence of steam (Run 317). However, when heated



GAS GENERATION VOLUME FOR CELLULOSE SPECIAL TESTS

FIGURE 18

TABLE XVII
 SUMMARY OF SPECIAL TEST NUMBERS (320) AND (321) - 40 GM CELLULOSE
 HEATED TO 1400°F

Test Number	Reactor Conditions/ Configurations	Run Time Hours	Volume Generated		Percent Gases Gen. versus Normal Test (317)
			Cubic Meters	(Liters)	
317	Gasifier in place, converter in place with steam flow	2.71	0.303	(30.3)	100
320	Gasifier in place, converter not used with steam flow	2.67	0.075	(7.5)	24.8
321	Gasifier in place, converter not used, no steam flow	2.66	0.0229	(2.29)	1.31

in the absence of steam (Run 321), only 78.5 percent of the cellulose was gasified. It is concluded that when processing cellulose, water serves to:

- Enhance the gasification of cellulose from the boat
- Convert non-condensable gases to condensable gases
- Ultimately gasify the char formed upon heating cellulose

Note also in Figure 18 that Run 321 (pyrolysis) gasified more rapidly and at lower temperatures, though the total gas generated was much less. As cellulose when heated, is known to char and to chemically split-off water and other simple gases, heating in the presence of steam more likely suppresses the water split reaction while enhancing production of other simple gases.

DATA ANALYSIS

Converter Reactor - Space Velocity Model

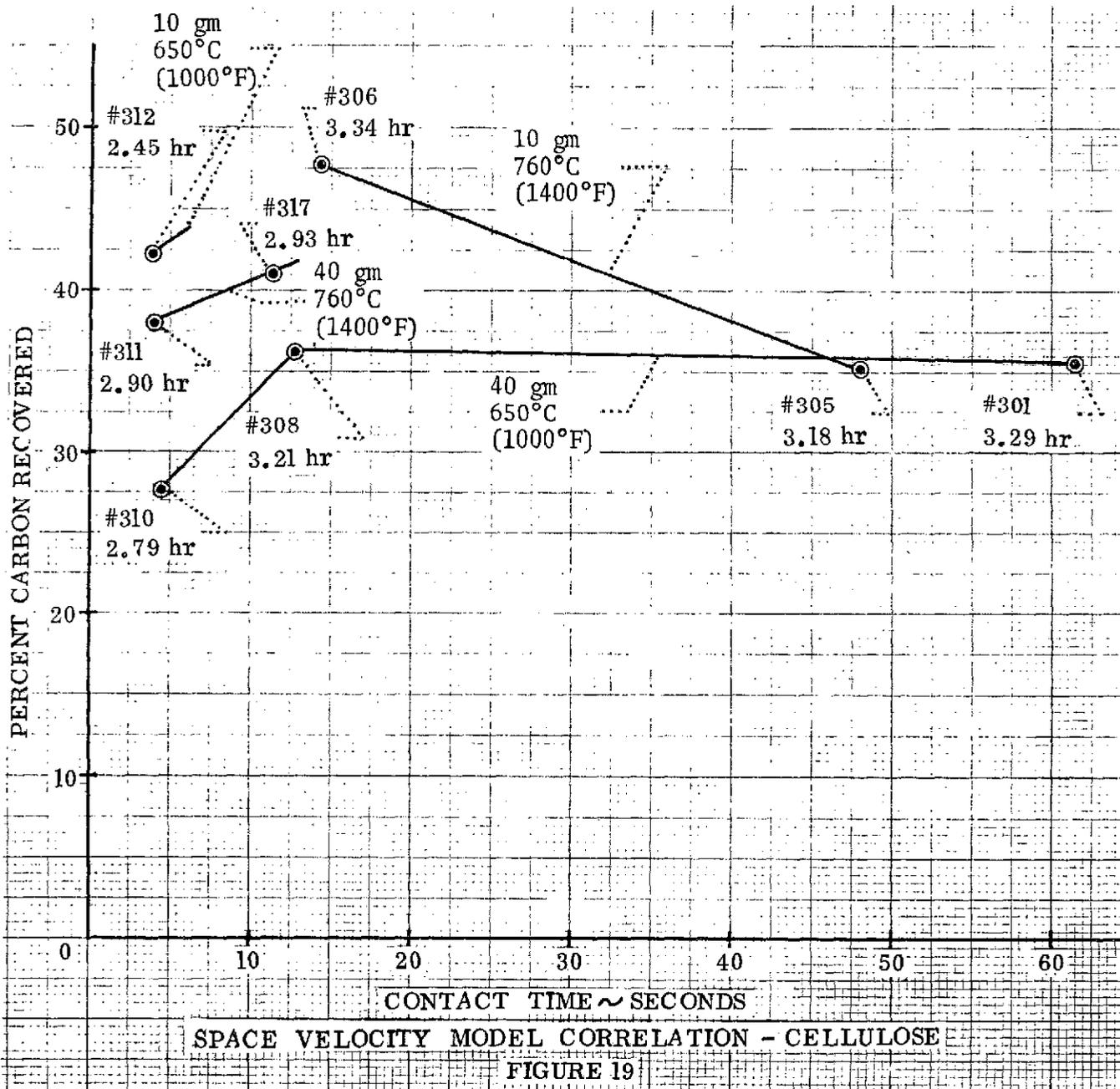
For this test series, the data treatment is handled in a manner similar to the small scale tests. Since gases are processed from the gasifier to the converter at a rate approximately proportional to the steam flow rate and because the reactor is isothermal, a space velocity estimate can be employed to establish reaction times. Space velocity and contact time have been defined in the section, "small scale tests - continuous reactor".

Steam flow rate is calculated from the volume of liquid water condensed at each sample point divided by run time between samples. Both the steam and the gas generation flow rates are calculated at reactor temperature by gas density calculations. Generally, the gas flow rate was small compared to the steam flow rate. Contact times were averaged for each sample point and then plotted against carbon recovery. An effort was made to compare the data between tests by using the same total run time. For the curves discussed below, the run times for data analysis are indicated, in hours, beside each point plotted.

Correlation

Figure 19 describes an approximate estimate of carbon recovery versus contact time in which the maximum recovery occurs for both the 10 gm and the 40 gm cellulose samples within 30 seconds. However, since only three data points generate these curves, the contact time for maximum carbon could be as low as 12 seconds, which is the maximum data point.

The effect of temperature is evident for the 40 gm cellulose run on Figure 19. The carbon recovery increases 5 to 10 percent as the converter temperature is elevated from 650°C (1200°F) to 760°C (1400°F).



The curve for 10 grams cellulose also plotted in Figure 19 is generated from data points taken at 650°C (1200°F), except Run 312 which was at 760°C (1400°F). Here, the maximum carbon recovery is shown to have increased up to 10 percent when the sample weight was reduced from 40 grams to 10 grams.

Polyethylene, Figure 20, approached 100 percent carbon recovery at a somewhat longer contact time. The methionine data, Figure 21, are very scattered, and do not support generalizing.

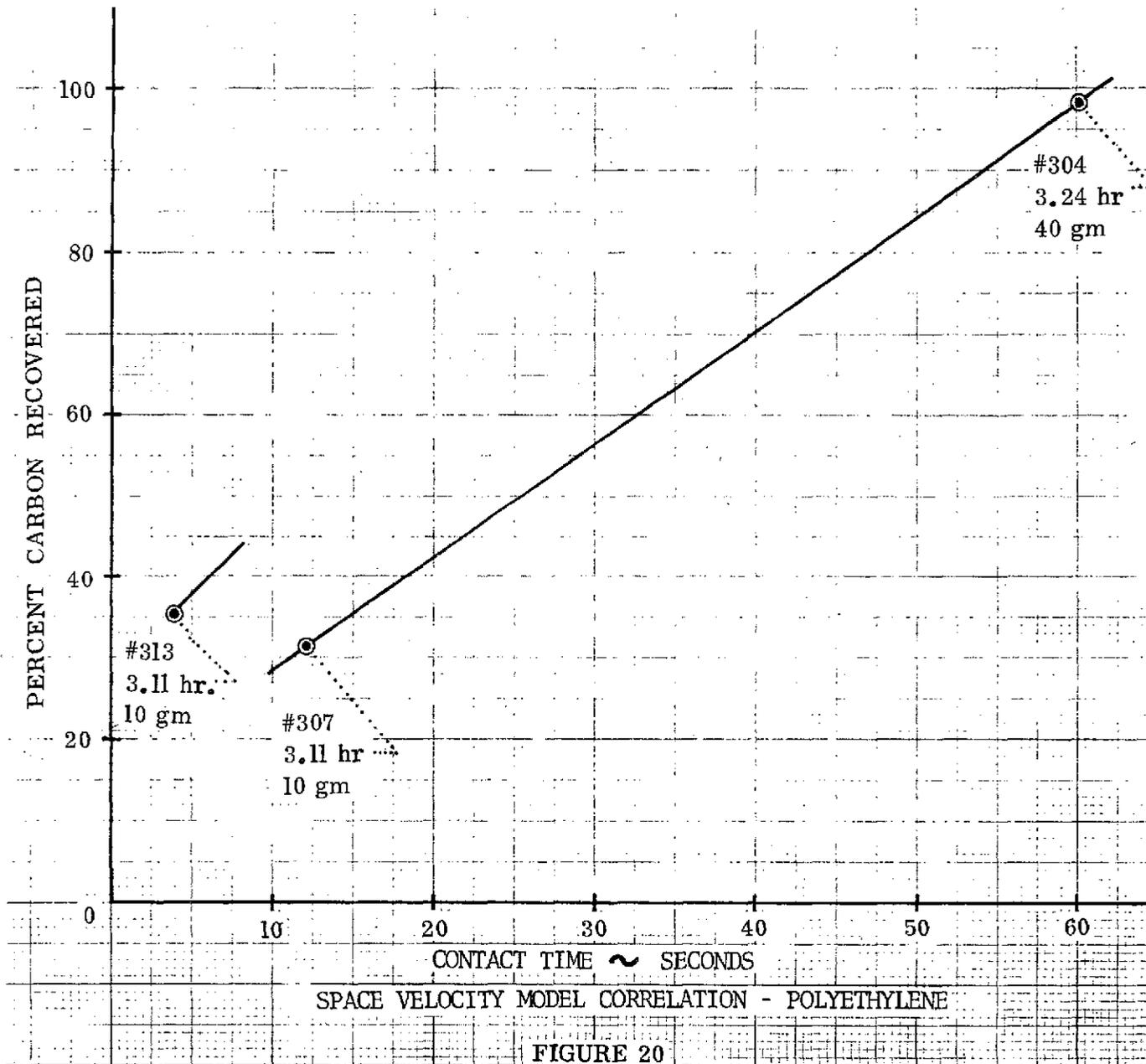
A general conclusion can be drawn from Figures 19 and 20 that carbon recovery for a given temperature is a function of contact time, and that maximum carbon recovery from cellulose and polyethylene occurs with contact times of 60 seconds or less and is perceptibly less for cellulose. Caution is expressed in the use of these curves because of the few data used to generate them. The general rationale developed from these data are: First, increasing the time a complex molecule resides at reaction temperature increases the probability of converting it to simple carbon compounds. However, in order to increase contact time the steam flow rate must be reduced. A lower flow rate means a lower water to waste ratio, which generally favors a lower equilibrium value for carbon recovery. More important, a lower water to waste ratio means a lower reaction driving force (P_{H_2O}) which, in turn, reduces the reaction rate for conversion. In summary, the net result of these two effects is seen in the cellulose curve, Figure 19.

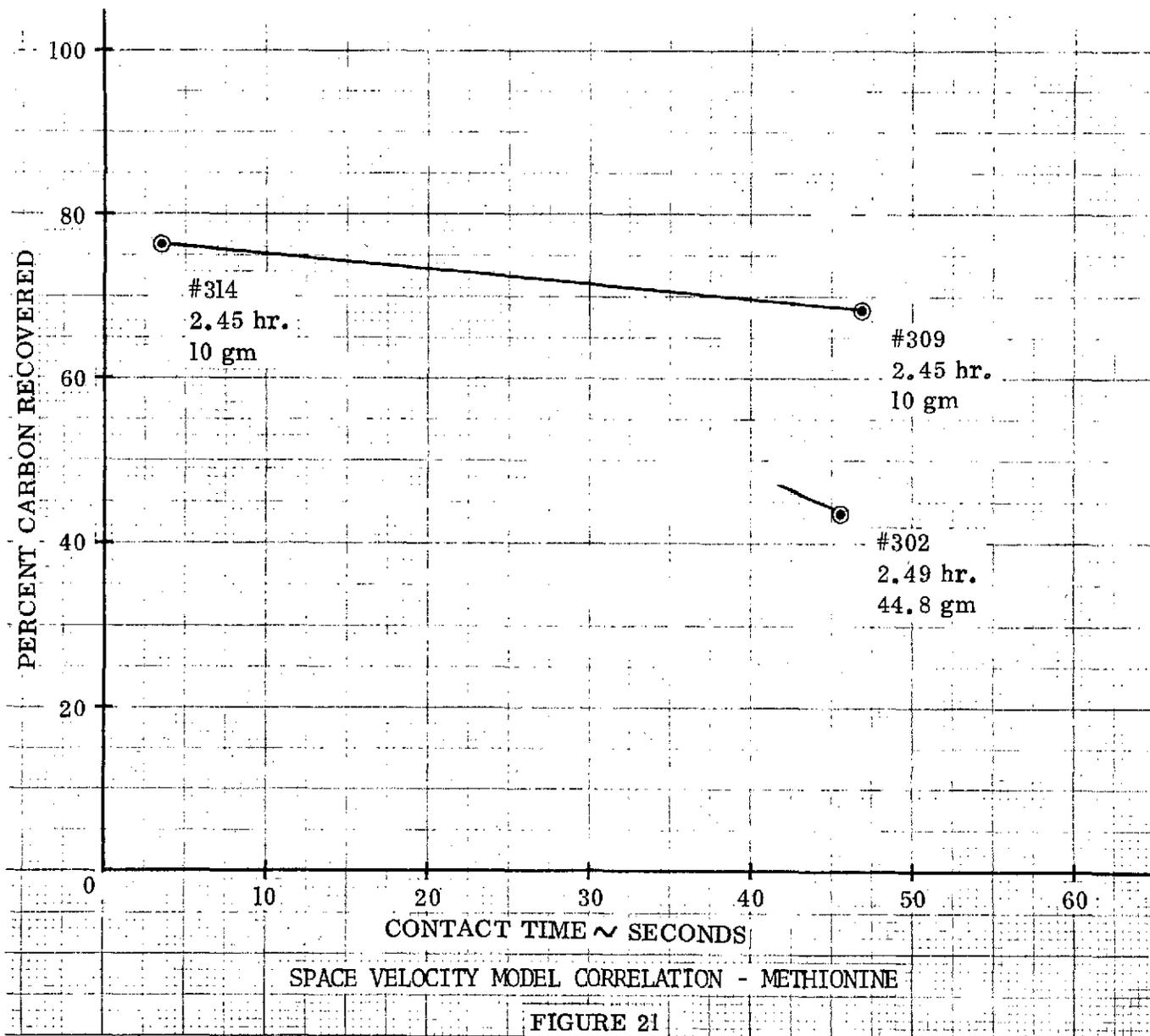
Polyethylene data, Figure 20, did not display a maximum, probably due to so few data, but it may be assumed that it is more difficult to decompose than cellulose. Evidence for this is seen by examining the gas volume generated as a function of run temperature for polyethylene versus cellulose and methionine (Appendix C). Polyethylene's maximum gas generation rate generally occurred at a temperature of approximately 425°C (800°F). However, both cellulose and methionine achieved their maximum gas rates at lower temperatures of 260 to 315°C (500 to 600°F).

Gasifier Reactor - Kinetic Model

All test runs in the batch series (see curves, Appendix C) have shown the on-set of the gasification reaction ("ignition") at a threshold temperature followed by a very rapid rise in gasification rate and then followed by a much slower reaction regime where the rate slowly diminishes as the waste is depleted. This unusual type of reactivity has been observed by workers in the fields of coal gasification and hydro-gasification. Results of rate correlation have recently been presented by H. Garner, et al (5) at the 165th National Meeting of the American Chemical Society in Dallas, Texas.

In contrast to a homogeneous reaction where the reaction activation energy is independent of reaction rate, Garner proposes a kinetic model in which the activation energy is clearly a function of the extent of the reaction. Since the waste gasification process studied in this program is so similar to coal gasification, a similar kinetic model might apply. Following Garner's notation, the steam gasification rate equation is given as:





$$-\frac{dX}{dt} = V_n P_{H_2O}^n (1-X) e^{-\Delta H^0/RT} \quad (1)$$

where X = fraction conversion of carbon
 V_n = frequency factor
 n = order of reaction
 ΔH^0 = activation energy for gasification
 R = gas constant, 1.987 cal/degree Kelvin/g-mole
 T = absolute temperature, degree Kelvin

Garner suggests the activation energy is related to X, the fraction of carbon converted, by the following equation:

$$\Delta H^0 = E_a + \alpha X \quad (2)$$

where E_a = initial activation energy
 α = sensitivity factor

Other workers, see reference 6, in related studies, have found ΔH^0 proportional to X^2 , indicating an even greater rate to concentration relationship.

In summary, the reaction rate for this process decreases exponentially with time. This is in strong contrast to the usual first order chemical reaction where the concentration decreases exponentially with time.

Simplifying equation (1) for correlation of this data, with rate as $-dX/dt$ and since P_{H_2O} was held constant at atmospheric pressure for the series it may be included in the proportionality constant, k. Also, substituting equation (2) in equation (1) results in

$$-dX/dt = k(1-X) e^{-(E_a + \alpha X)/RT} \quad (3)$$

Rearranging and taking logs gives

$$\ln \frac{dX/dt}{1-X} = \ln k - \frac{E_a + \alpha X}{R} \left(\frac{1}{T} \right) \quad (4)$$

Now the equation is in a form for correlation.

Correlation

A review of the batch tests summarized in Appendix C indicated only a small temperature dependence of the gasification rate after the "ignition" of the reactors. This suggests that the activation energy is quite small. Therefore, E_a was set equal to zero which generated a mathematical form easy to correlate by available computer least squares analysis program:

$$\ln \frac{-dX/dt}{1-X} = \ln k - \frac{\alpha}{R} \left(\frac{X}{T} \right) \quad (6)$$

Table XVIII summarizes the results of the first four tests conducted in the 300 run series. These tests showed better correlation than other tests attempted. To obtain correlation, all sample points before "ignition" were deleted from the analysis.

In Table XVIII, the exponent constant α shown above each test run was determined from the slope generated from the least squares calculation. Using this, the equation (3) was rearranged and the rate constant then calculated for each sample point and then averaged.

Curves generated from plotting $\ln (-dX/dt / (1 - X))$ versus $X/T^\circ K$ are shown in Figures 22, 23, 24 and 25. These curves show acceptable correlation for cellulose, methionine, cellulose + KIO_3 , and polyethylene.

Correlation was not attempted on test runs involving real spacecraft wastes; however, it is believed such a model should apply. Also, correlation was not attempted with E_a included in the treatment; it is believed that E_a should be quite small.

TABLE XVIII
KINETIC MODEL CORRELATION

STEAM REFORMATION RATE TREATMENT
EQUATION OF THE FORM RATE = K EXP-(EA+AXI/RT)(1-X)

RUN 301 - CELLULOSE

INPUT ACT. ENERGY = 0.0 KCAL/MOLE
EXPONENT CONSTANT = 77.698

TEST NUMBER	ABSCISSA X(NP) (X/DEG K)	ORDINATE Y(NP)	REACTOR TEMP. (DEG F)	REACTOR TEMP (DEG C)	PERCENT RECOVERED	REACTION RATE GM-ATOM/MIN	CALCULATED RATE CONSTANT
301.02	0.00030354	-4.6338	932.	500.	23.464	.7438E-02	0.1388E 04
301.03	0.00034197	-6.2267	1103.	595.	25.683	.1389E-02	0.1268E 04
301.04	0.00034199	-6.3385	1164.	629.	30.844	.1222E-02	0.1135E 04
301.05	0.00035333	-6.7995	1164.	629.	31.866	.7593E-03	0.1115E 04
301.06	0.00036148	-7.2306	1159.	626.	32.501	.4887E-03	0.9966E 03
301.07	0.00036068	-7.0970	1183.	639.	32.910	.5552E-03	0.1104E 04
301.08	0.00036136	-7.1002	1203.	651.	33.374	.5496E-03	0.1130E 04
301.09	0.00035794	-7.3296	1242.	672.	33.833	.4340E-03	0.7859E 03
301.10	0.00036783	-7.5566	1214.	657.	34.196	.3439E-03	0.9222E 03
301.11	0.00037182	-7.4418	1210.	654.	34.484	.3841E-03	0.1205E 04
301.12	0.00037550	-7.3734	1209.	654.	34.805	.4092E-03	0.1495E 04
301.13	0.00038033	-7.5784	1204.	651.	35.147	.3316E-03	0.1471E 04
301.14	0.00038658	-7.9347	1190.	643.	35.424	.2312E-03	0.1315E 04
301.15	0.00039155	-8.3386	1178.	637.	35.618	.1539E-03	0.1067E 04
301.17	0.00040448	-8.7101	1160.	627.	36.390	.1049E-03	0.1220E 04

AVG. RATE CONSTANT = 0.11749E 04

RUN 302 - METHIONINE

INPUT ACT. ENERGY = 0.0 KCAL/MOLE
EXPONENT CONSTANT = 78.170

TEST NUMBER	ABSCISSA X(NP) (X/DEG K)	ORDINATE Y(NP)	REACTOR TEMP. (DEG F)	REACTOR TEMP (DEG C)	PERCENT RECOVERED	REACTION RATE GM-ATOM/MIN	CALCULATED RATE CONSTANT
302.04	0.00042485	-5.6510	1125.	607.	37.396	.2200E-02	0.6375E 05
302.05	0.00044175	-6.3408	1150.	621.	39.497	.1067E-02	0.6218E 05
302.06	0.00045178	-6.4707	1152.	622.	40.444	.9693E-03	0.8518E 05
302.07	0.00045855	-6.4755	1162.	628.	41.305	.9043E-03	0.1053E 06
302.08	0.00046427	-6.7568	1172.	633.	42.078	.6736E-03	0.9948E 05
302.09	0.00047112	-7.0899	1172.	633.	42.699	.4776E-03	0.9337E 05
302.10	0.00047932	-6.9521	1160.	627.	43.123	.5441E-03	0.1480E 06
302.11	0.00047674	-7.1316	1197.	642.	43.606	.4508E-03	0.1117E 06
302.12	0.00048525	-7.4287	1173.	634.	44.007	.3326E-03	0.1160E 06
302.13	0.00048583	-7.7356	1182.	639.	44.302	.2434E-03	0.8730E 05
302.14	0.00048880	-7.6712	1180.	638.	44.519	.2586E-03	0.1047E 06
302.15	0.00049132	-7.8525	1180.	638.	44.748	.2144E-03	0.9640E 05
302.16	0.00049402	-8.4341	1178.	637.	44.939	.1197E-03	0.5992E 05
302.18	0.00049924	-8.6557	1180.	638.	45.470	.9495E-04	0.5898E 05
302.19	0.00050110	-8.9452	1183.	639.	45.723	.7075E-04	0.4750E 05

AVG. RATE CONSTANT = 0.69312E 05

RUN 303 - CELLULOSE + KIO₃

INPUT ACT. ENERGY = 0.0 KCAL/MOLE
EXPONENT CONSTANT = 56.598

TEST NUMBER	ABSCISSA X(NP) (X/DEG K)	ORDINATE Y(NP)	REACTOR TEMP. (DEG F)	REACTOR TEMP (DEG C)	PERCENT RECOVERED	REACTION RATE GM-ATOM/MIN	CALCULATED RATE CONSTANT
303.04	0.00035228	-6.1122	1136.	613.	31.224	.1524E-02	0.5052E 02
303.06	0.00038186	-6.7221	1152.	622.	34.185	.7924E-03	0.6374E 02
303.07	0.00038543	-6.8571	1173.	634.	34.954	.6842E-03	0.6165E 02
303.08	0.00038824	-7.1852	1192.	644.	35.619	.4878E-03	0.4811E 02
303.09	0.00039193	-7.2835	1220.	660.	36.567	.4357E-03	0.4844E 02
303.10	0.00039862	-7.3453	1230.	666.	37.413	.4041E-03	0.5510E 02
303.11	0.00040699	-7.6421	1230.	666.	38.198	.2965E-03	0.5196E 02

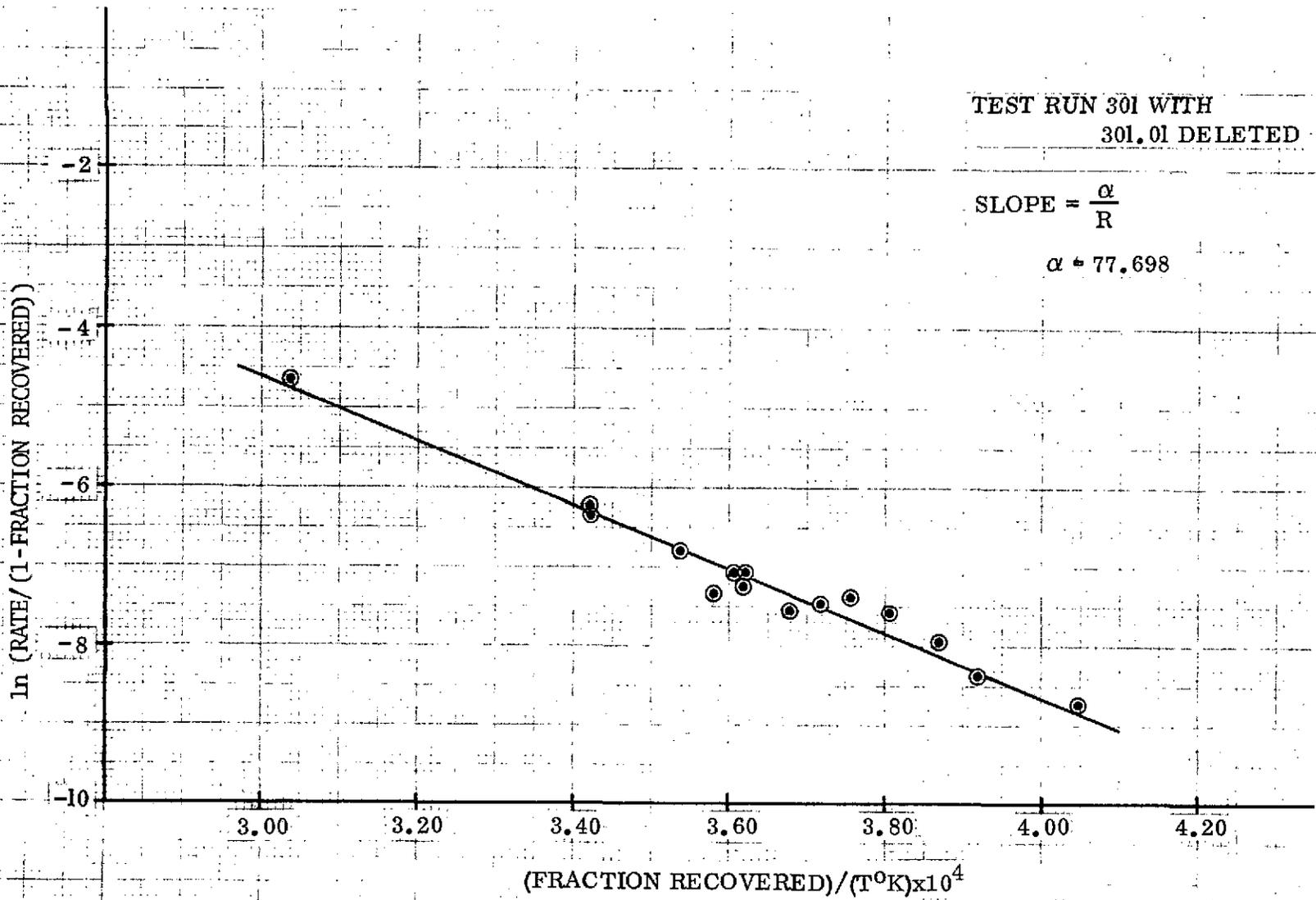
AVG. RATE CONSTANT = 0.54217E 02

RUN 304 - POLYETHYLENE

INPUT ACT. ENERGY = 0.0 KCAL/MOLE
EXPONENT CONSTANT = 19.007

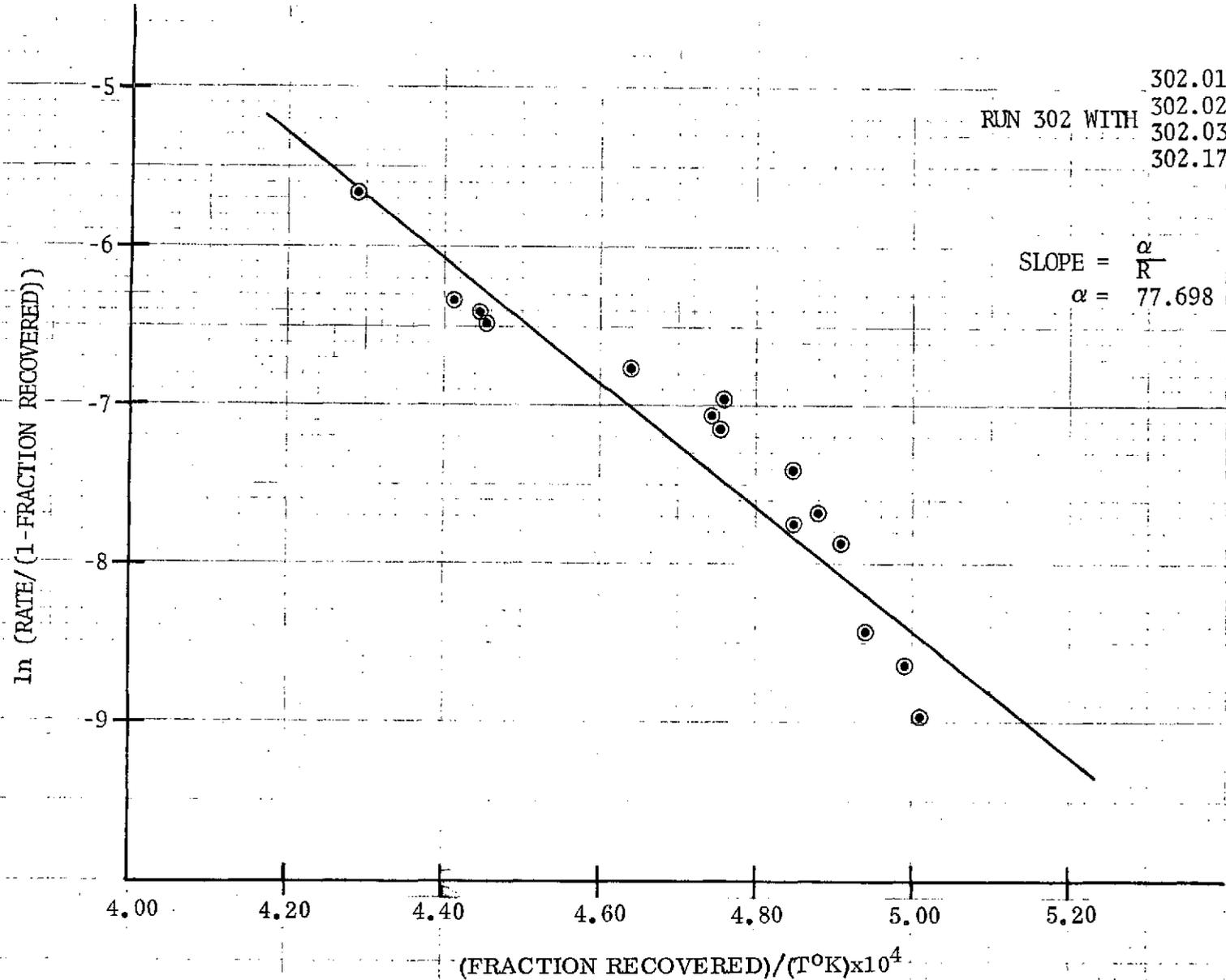
TEST NUMBER	ABSCISSA X(NP) (X/DEG K)	ORDINATE Y(NP)	REACTOR TEMP. (DEG F)	REACTOR TEMP (DEG C)	PERCENT RECOVERED	REACTION RATE GM-ATOM/MIN	CALCULATED RATE CONSTANT
304.06	0.00100773	-3.8864	1140.	616.	89.542	.2146E-02	0.3152E 03
304.07	0.00104267	-4.3101	1150.	621.	93.226	.9099E-03	0.2882E 03
304.08	0.00107823	-4.7827	1123.	606.	94.788	.4364E-03	0.2525E 03
304.09	0.00109575	-4.9711	1110.	599.	95.537	.3095E-03	0.2473E 03
304.10	0.00107887	-4.4639	1170.	632.	97.662	.2693E-03	0.3494E 03
304.11	0.00109853	-4.5641	1156.	624.	98.587	.1472E-03	0.3815E 03

AVG. RATE CONSTANT = 0.30567E 03



RATE EQUATION PLOT - CELLULOSE

FIGURE 22



RATE EVALUATION PLOT - METHIONINE

FIGURE 23

RUN 302 WITH 302.01, 302.02, 302.03, 302.17

DELETED

SLOPE = $\frac{\alpha}{R}$
 $\alpha = 77.698$

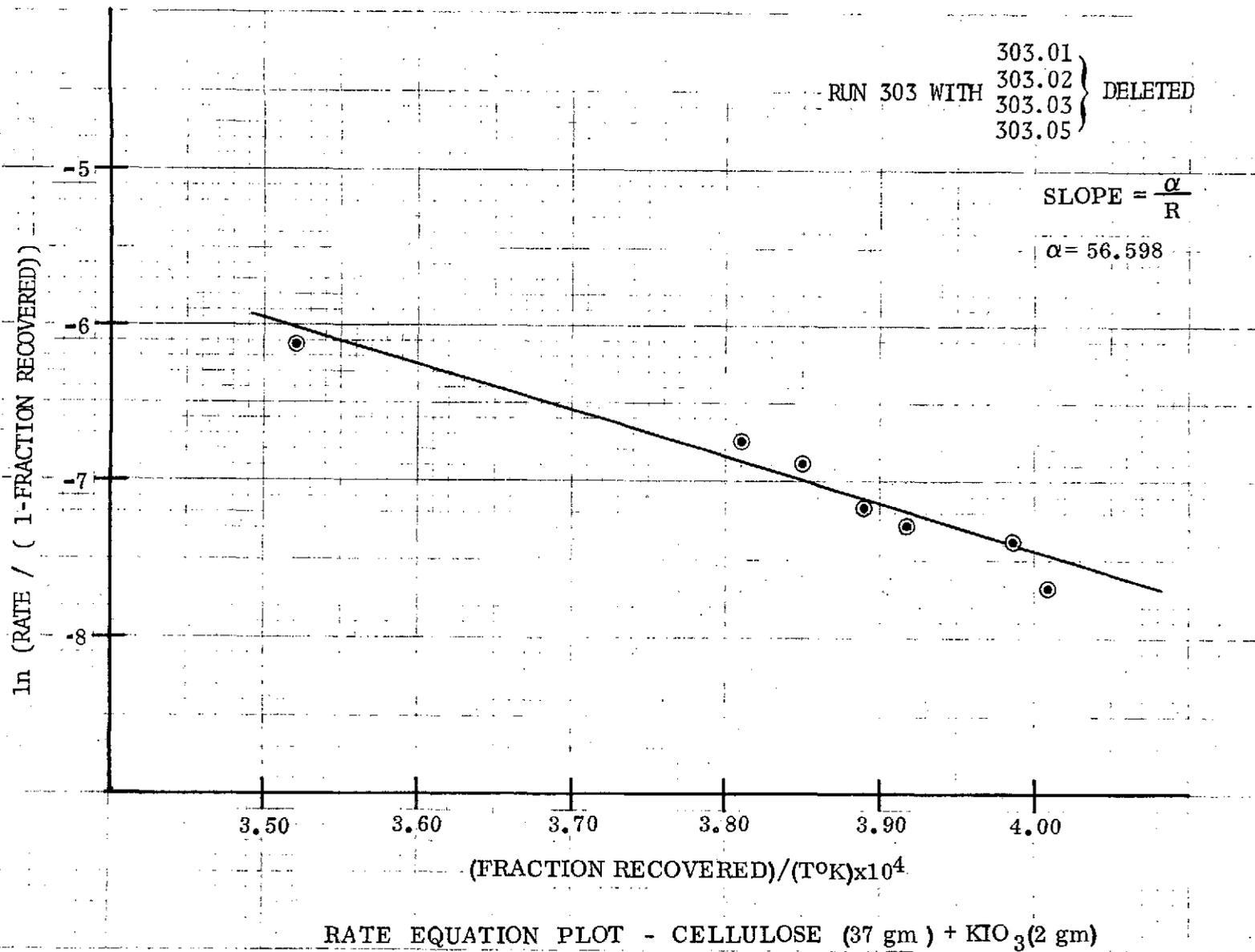
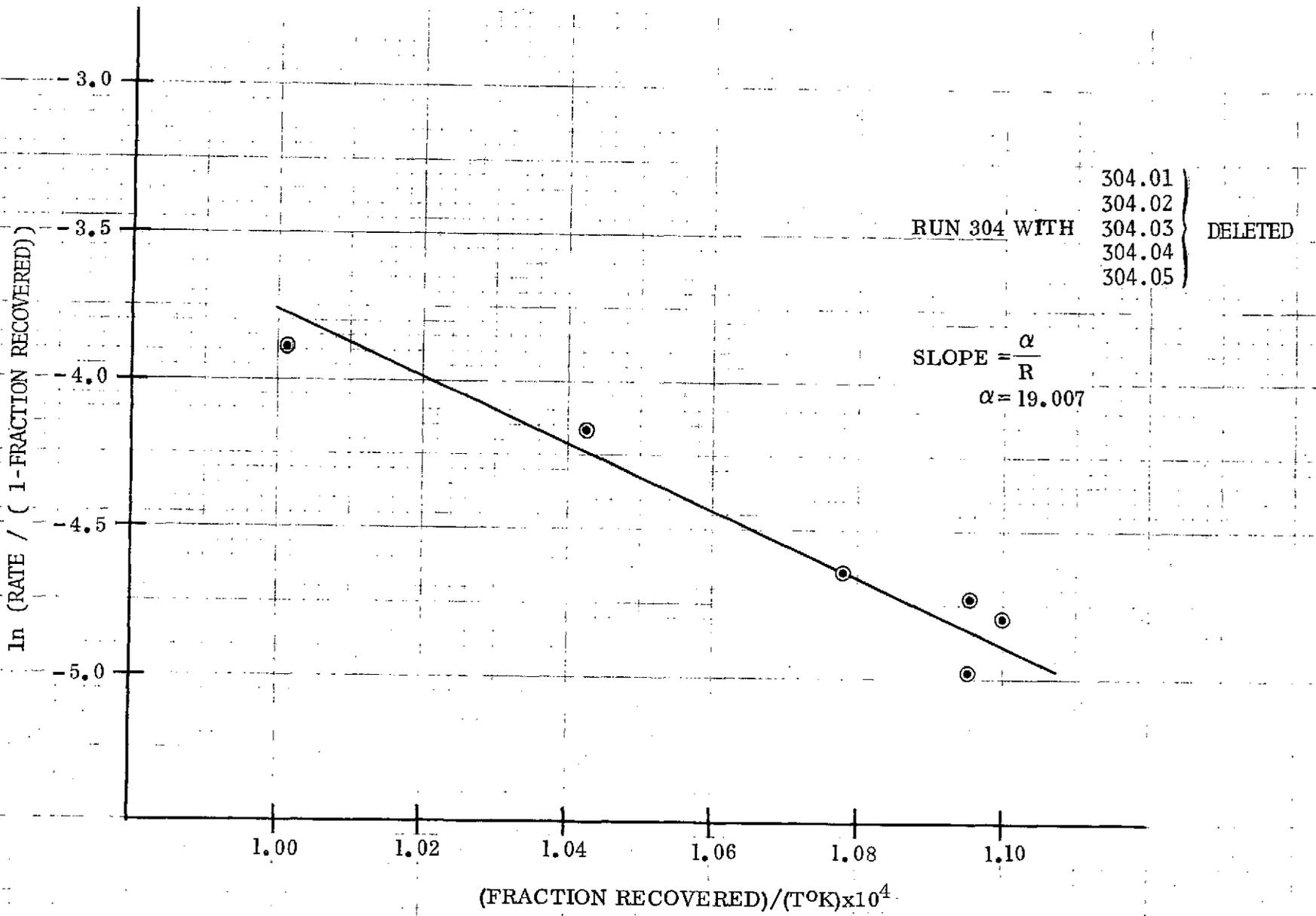


FIGURE 24



(FRACTION RECOVERED)/(T°K)x10⁴

RATE EQUATION PLOT-POLYETHYLENE

FIGURE 25

PRELIMINARY SYSTEM DEFINITION

The requirements of the Shuttle EC/LSS and the Space Station Prototype (SSP) ETC/LSS were reviewed in order to generate a preliminary definition of a Steam Reformation Central Waste processor.

SHUTTLE APPLICATION

The results of this review indicate that the Shuttle mission is not an attractive application for the steam reformation concept. The present Shuttle Waste Management system consists of:

- Urine - collected and treated with silver nitrate and stored in tanks for return to earth.
- Feces - vacuum dried with the moisture being vented overboard.
- Wash Water - collected and stored with the urine.
- Atmosphere Condensate - collected and stored with the urine.
- Wet Waste Materials - vacuum dried and stored with the feces.
- Dry Waste Materials - stored in separate dry waste containers for return to earth.

The ground rules for the disposal of wastes in the Shuttle program are:

- Solid wastes shall not be dumped overboard.
- Gases and vapors may be vented overboard.
- Liquid wastes may not be vented overboard except in a fail safe operation (following storage system failure).

The present Shuttle mission lasts 7 days with an additional 4 days contingency. Power is provided by fuel cells which presently produce approximately 244.9 kg lbs of excess potable water, which must be stored or vented overboard.

A major feature of steam reformation is the production of potable water through the condensation of steam and through the reaction of hydrogen and carbon dioxide in a Sabatier reactor. Although a steam reformation system could eliminate the waste storage subsystem, there is no need for the steam reformation products aboard Shuttle. In order to dispose of the steam and non condensible vapors produced, the Shuttle ground rules would require these to be discharged as gases or vapors and the thermal energy present in the steam would have only a limited recoverability. The effect of this inefficiency would be to increase the power associated weight penalty required for this application with a consequent increase in fuel cell size, weight, and water production.

SPACE STATION PROTOTYPE CONCEPT

Based on results from the batch test program a simplified concept of a steam reformation unit for space waste disposal was prepared for Space Station Prototype (SSP) application.

Requirements

The waste model was taken from the SSP program. The predicted quantities of wastes to be handled are shown in Table XIX.

TABLE XIX
PREDICTED WASTE QUANTITIES FOR SPACE STATION APPLICATION

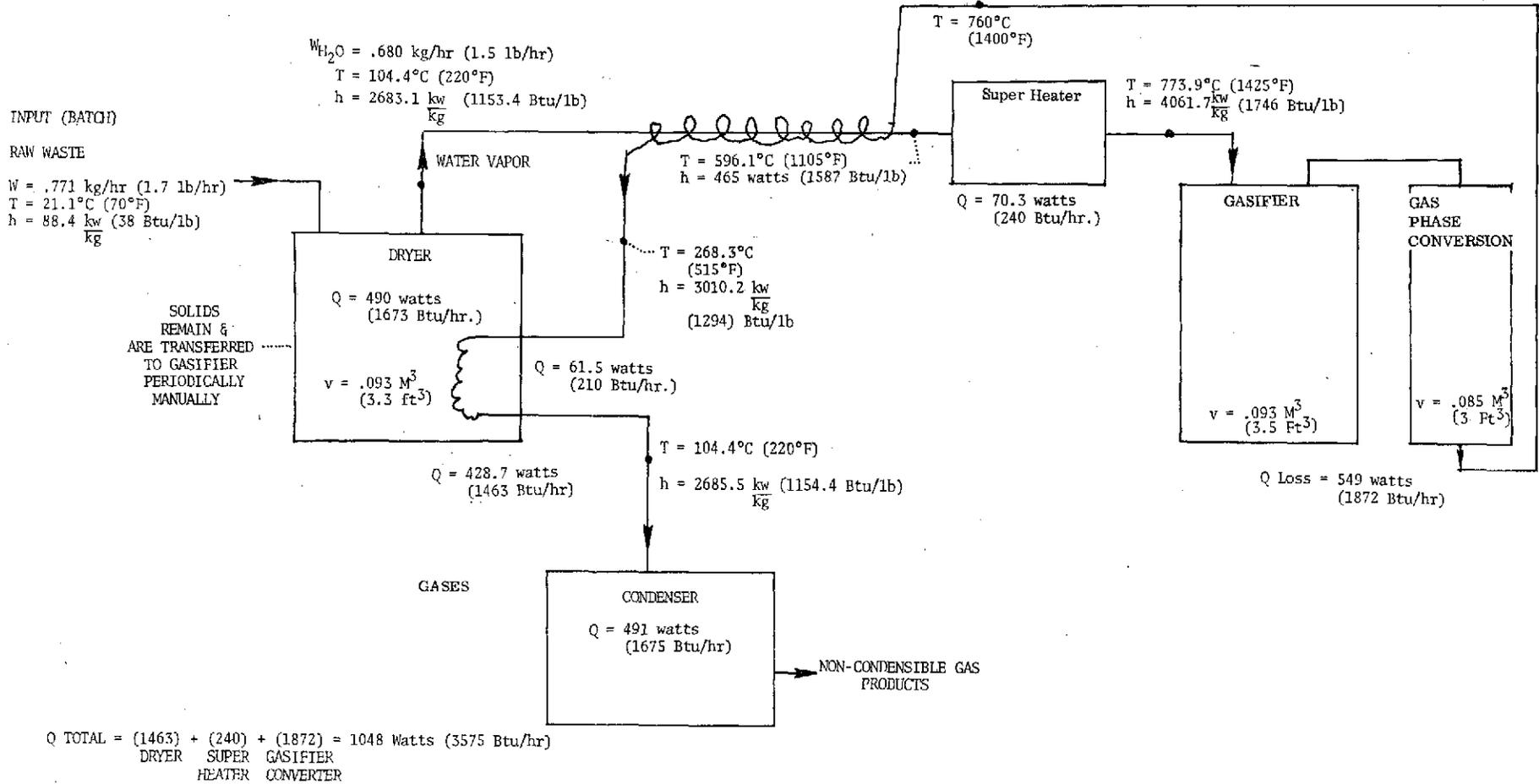
	Solids		Water	
	kg/Man Day	(Lbs/Man Day)	kg/Man Day	(Lbs/Man Day)
Urine	0.0590	0.13	1.5011	3.31
Feces	0.0316	0.07	0.0907	0.20
Sweat	0.0181	0.04	-	-
Food	0.2132	0.47	0.2086	0.46
Flush Water	-	-	0.4535	1.00
TOTALS	0.341	0.71	2.2539	4.97

For Six Men Total Solids = 0.71 x 6 = (4.26 lbs/day) 1.932 kg/day

Total H₂O = 4.97 x 6 = (29.82 lbs/day) 13.524 kg/day

Concept Description

A schematic of a potential system is shown in Figure 26. The wet wastes are placed in the dryer on a batch basis. External heat as well as recovered heat are used to dry the wastes. The steam from the dryer is superheated to 773.9°C (1425°F) and then goes to the gasifier. Previously dried wastes are contained in the gasifier and are partially reformed. The effluent from the gasifier then goes to the gas phase converter where conversion is completed.



BLOCK SYSTEM CONCEPT DIAGRAM

FIGURE 26

The hot reformed gases then pass through a heat exchanger which provides the major portion of the heat required for superheating the steam from the dryer. A condenser is then used to separate the non condensible gases from the water.

After each batch is processed the positions of the dryer and gasifier are reversed and the system is ready to accept a new batch of waste.

System Performance

The schematic shown in Figure 26 gives the flows, temperatures and heat loads for the system. A 20 hour/day duty cycle is assumed. External heat inputs are required to provide heat of vaporization in the dryer and to make up heat lost to ambient. The total input energy required is 1048 watts.

System Sizing

Assuming the density of compacted wet trash is 480.6 kg/m^3 (30 lbs/ft^3) and allowing for 25% utilization of the volume the evaporator volume required is 0.0991 m^3 (3.5 ft^3). The gasifier and evaporator are interchangeable. The reformer consists of 60.1 meters of 25.4 mm I. D. tubing (200 feet of 1 inch).

The estimated weight of the system is 204.08 kg (450 lbs) for the functions identified in the block diagram.

REFERENCES

- (1) NASA Contractor Report CK-1205 (111) "Compendium of Human Responses to the Aerospace Environment", November 1968.
- (2) Paper Titled "Catalysis of Coal Gasification at Elevated Pressures", by Haynes, W. P., et al, presented at the 165th National Meeting of the American Chemical Society, Dallas, Texas, April 1973. Volume 18, No. 2.*
- (3) Bureau of Mines Report, No. 7560, "Converting Organic Waste to Oil", Appell, H. R. et al. (1971)
- (4) Paper Titled "Gasification of Organic Waste", by Cox, J. L., et al, presented at the 165th National Meeting of the American Chemical Society, Dallas, Texas, April 1973. Volume 18, No. 1.*
- (5) Paper Titled "Catalyzed Hydrogasification of Coal Chars by Garner, H. presented at the 165th National Meeting of the American Chemical Society, Dallas, Texas, April 1973.
- (6) Paper Titled "Kinetics of Bituminous Coal Char Gasification with Steam and Hydrogen", by Johnson J., presented at the 165th National Meeting of the American Chemical Society, Dallas, Texas, April 1973. Volume 18, No. 1.*

*Available at nominal cost from:

Division of Fuel Chemistry
Office of Secretary Treasurer, Dr. H. R. Appell
Bureau of Mines
4800 Forbes Avenue
Pittsburgh, Pennsylvania 15213

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APPENDIX A

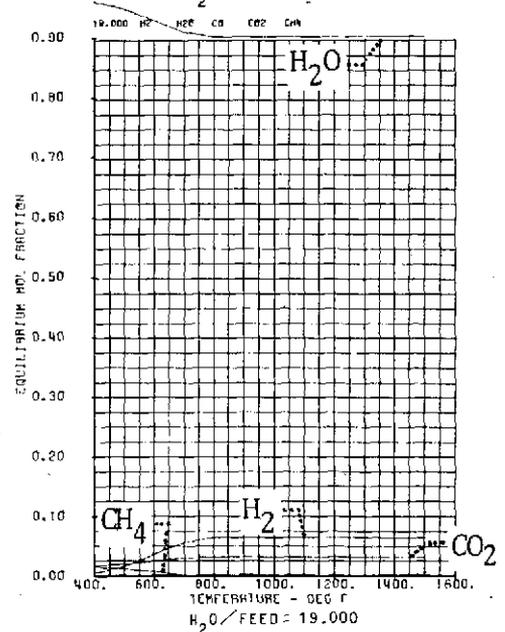
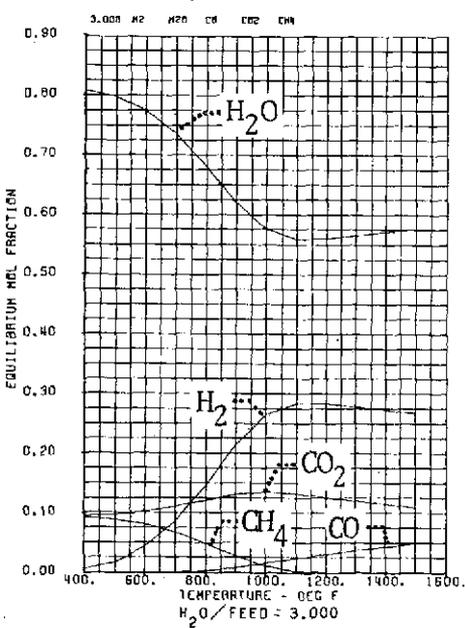
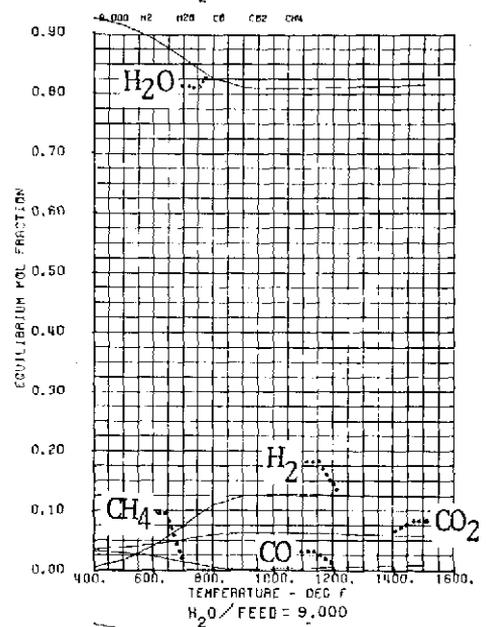
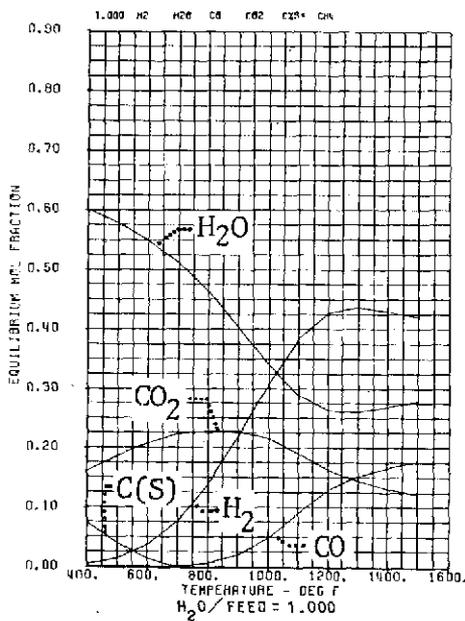
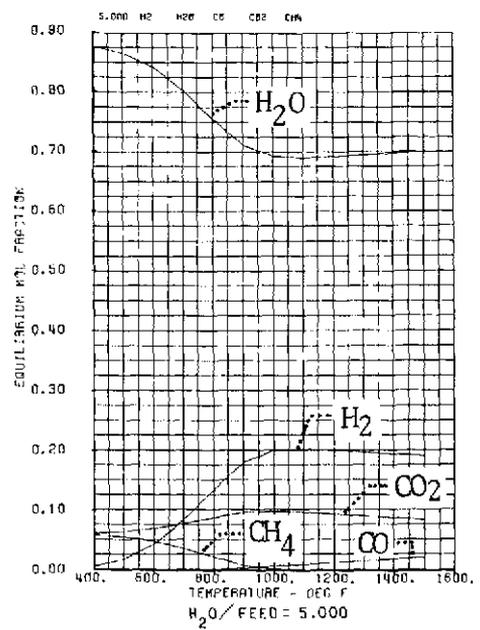
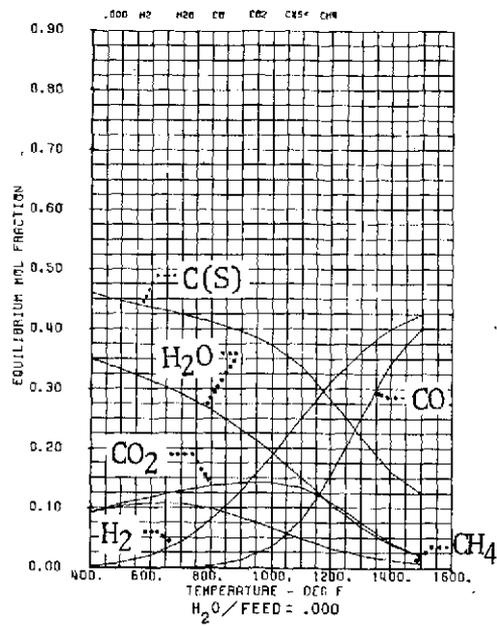
EQUILIBRIUM MOLE FRACTION CURVES

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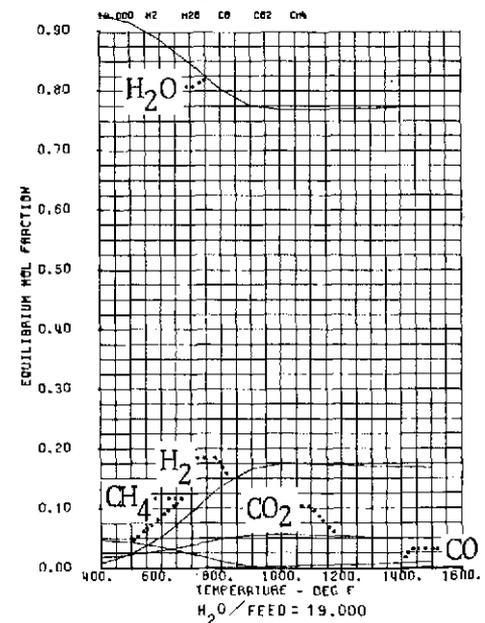
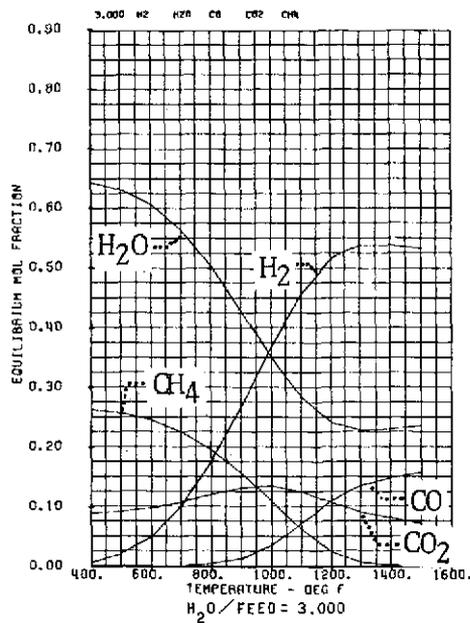
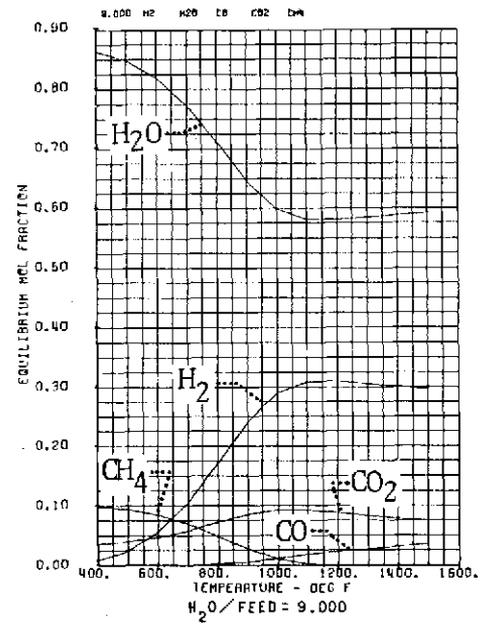
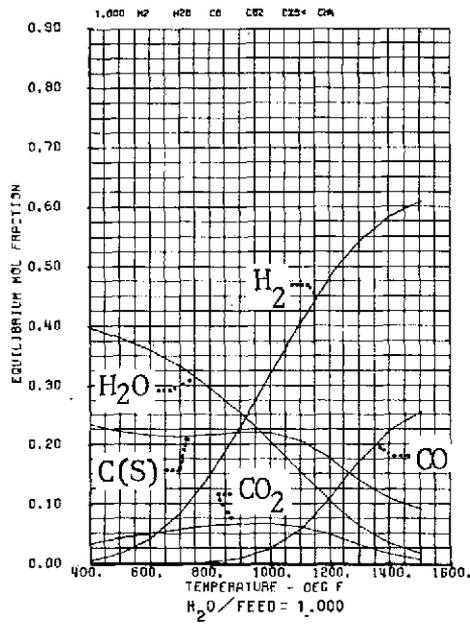
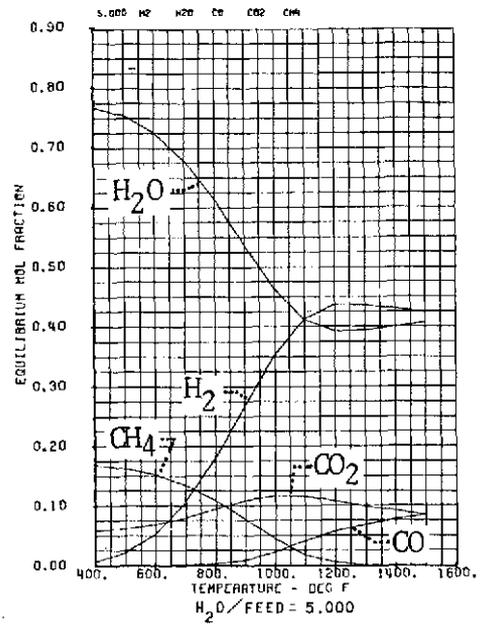
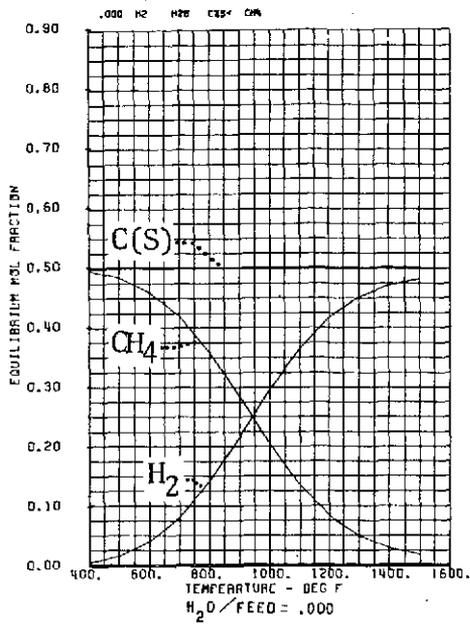
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CELLULOSE



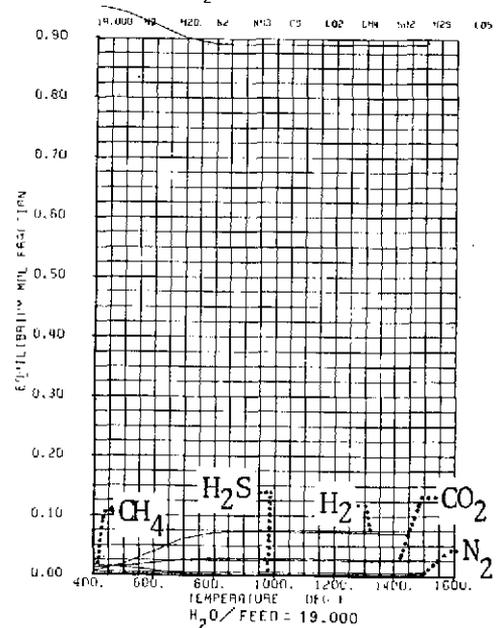
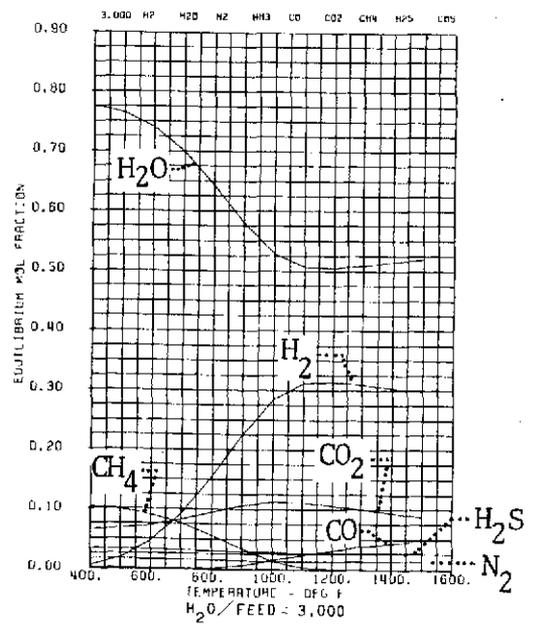
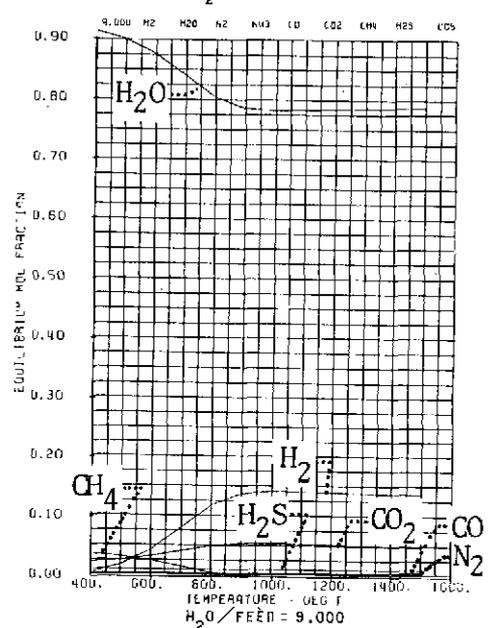
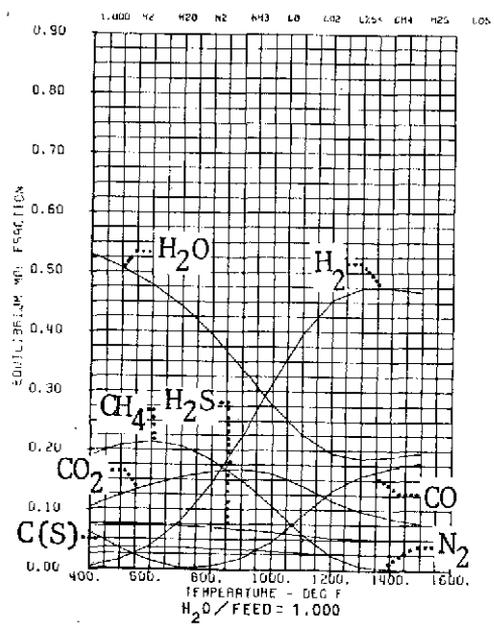
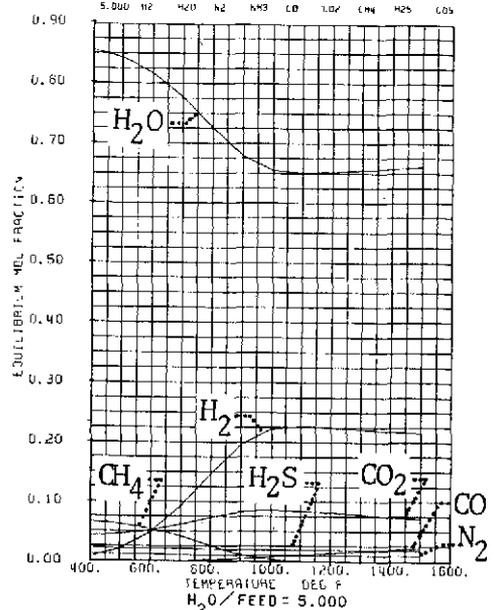
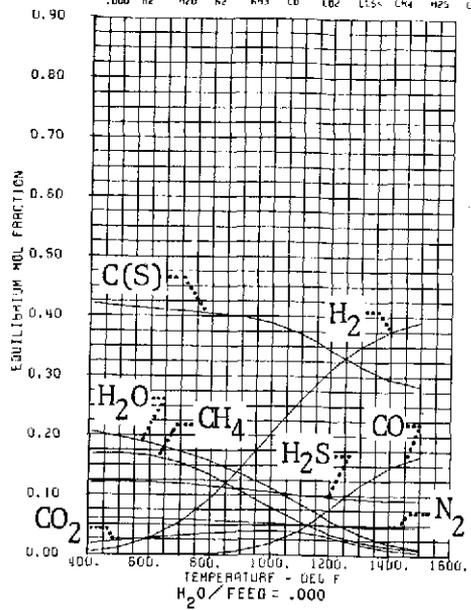
CELLULOSE EQUILIBRIUM COMPOSITION

POLYETHYLENE



POLYETHYLENE EQUILIBRIUM COMPOSITION

PURE METHIONINE



PURE METHIONINE EQUILIBRIUM COMPOSITION

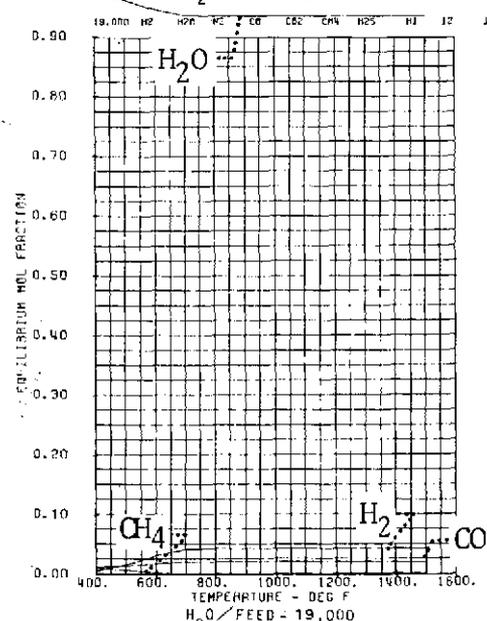
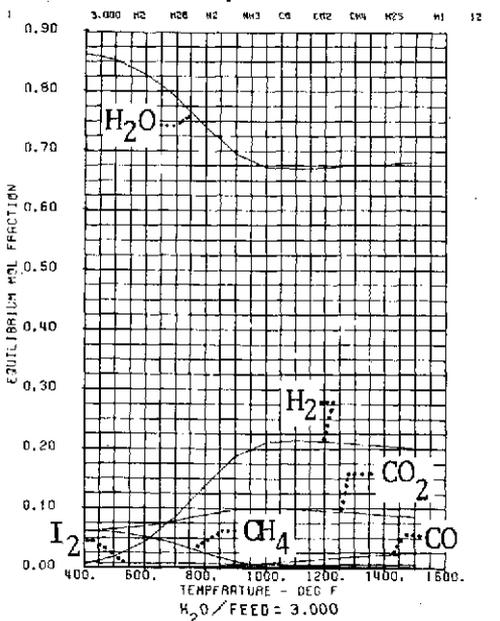
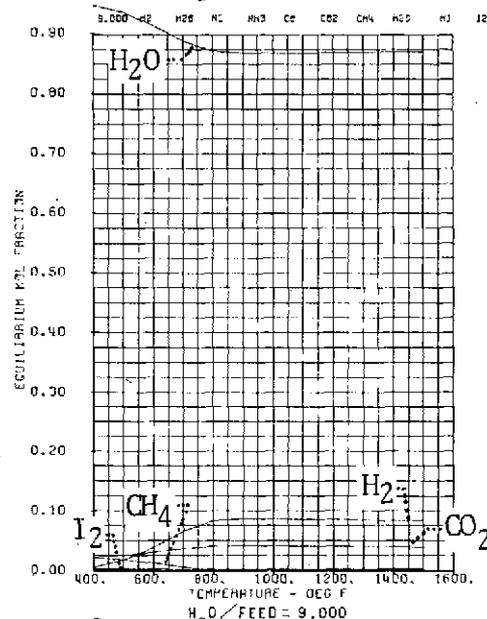
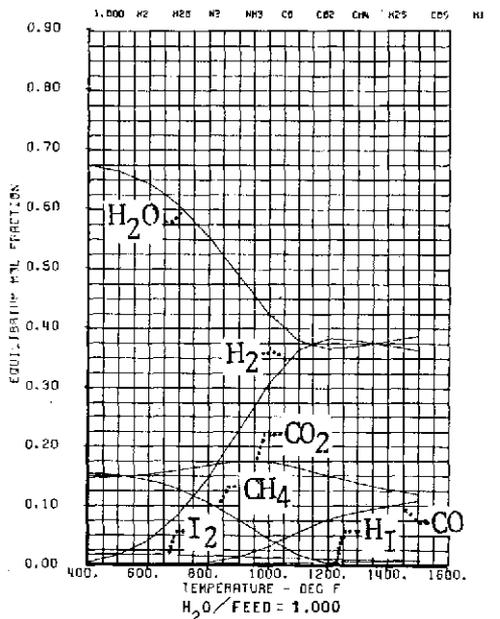
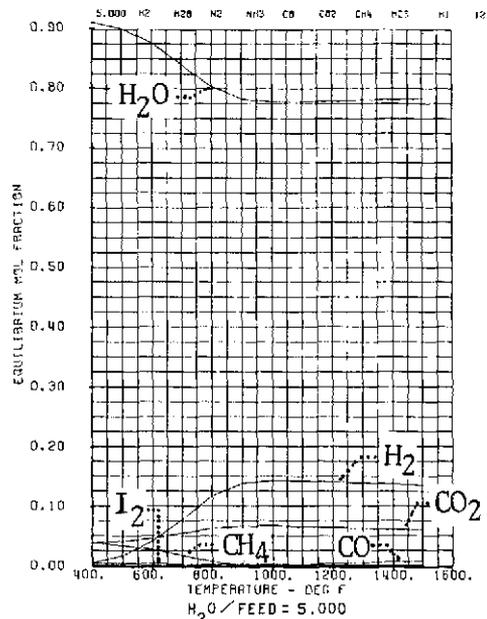
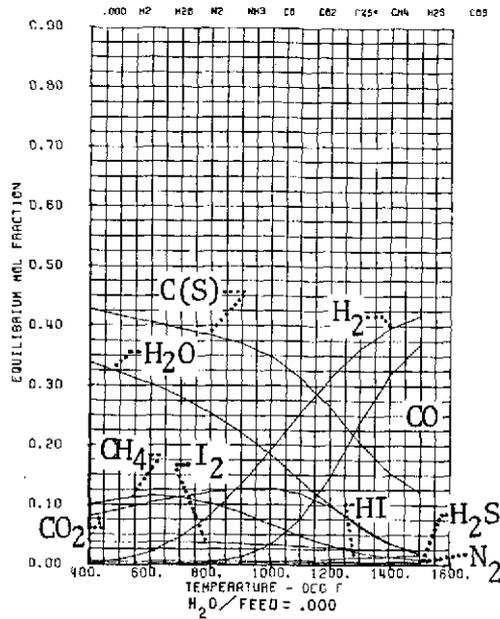
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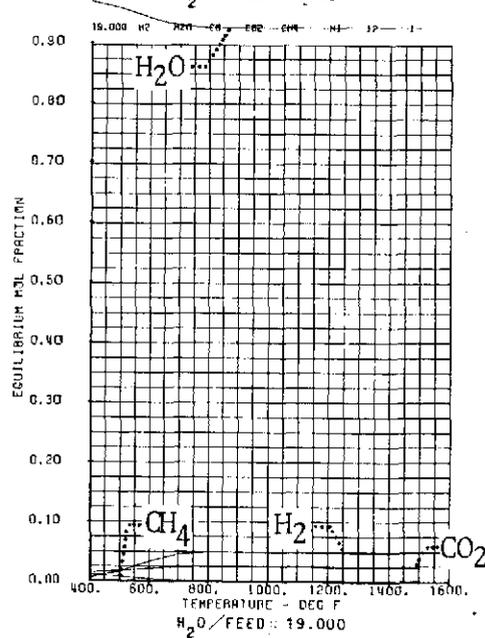
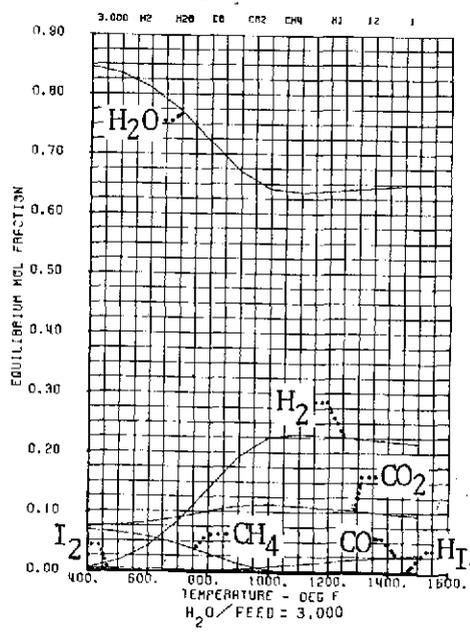
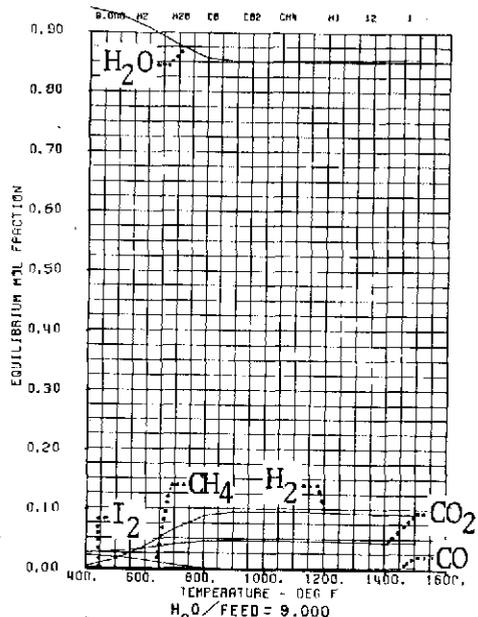
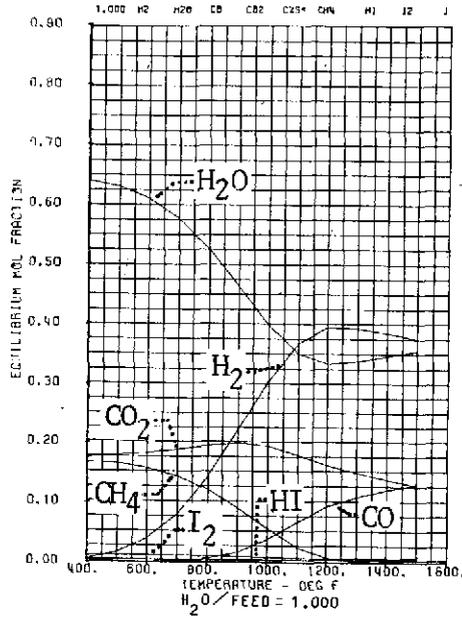
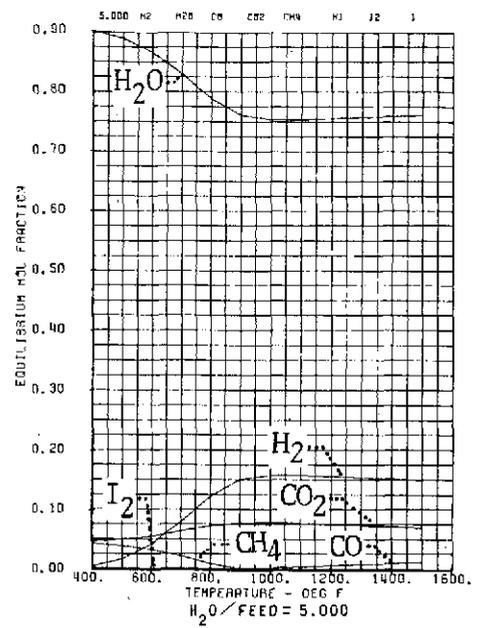
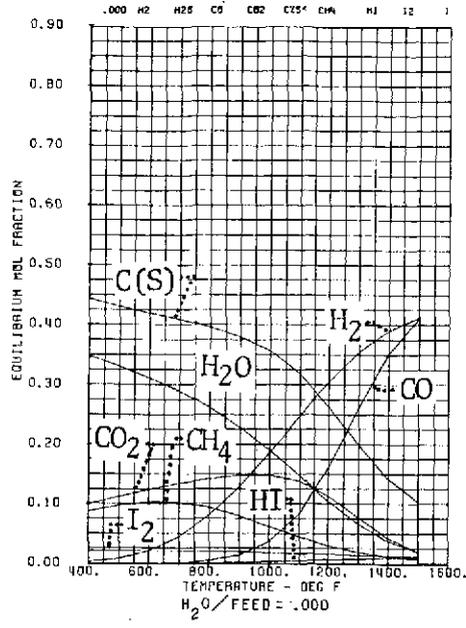
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C₁₀₀H₁₈₀O₄₈ S, N, I
EQUILIBRIUM MOLE FRACTION



SPACECRAFT MIXTURE (AS $C_{100}H_{180}O_{48}S_1N_1I_1$) EQUILIBRIUM COMPOSITION

95% CELLULOSE + 5% IODATE



CELLULOSE AND 5 PERCENT IODATE EQUILIBRIUM COMPOSITION

APPENDIX B

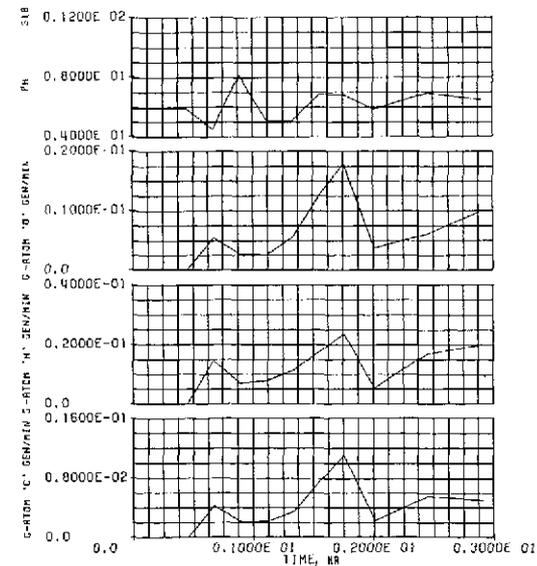
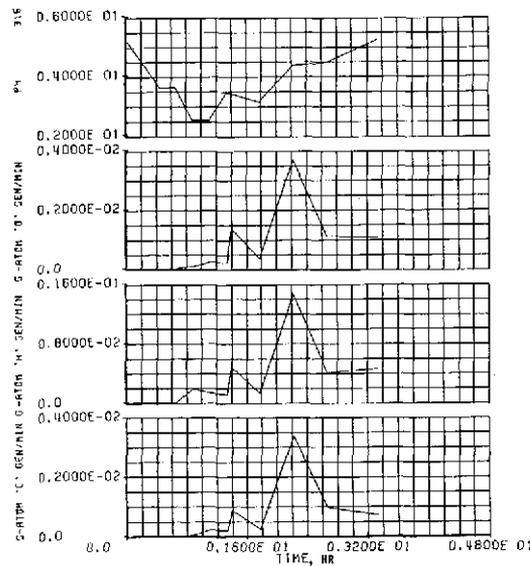
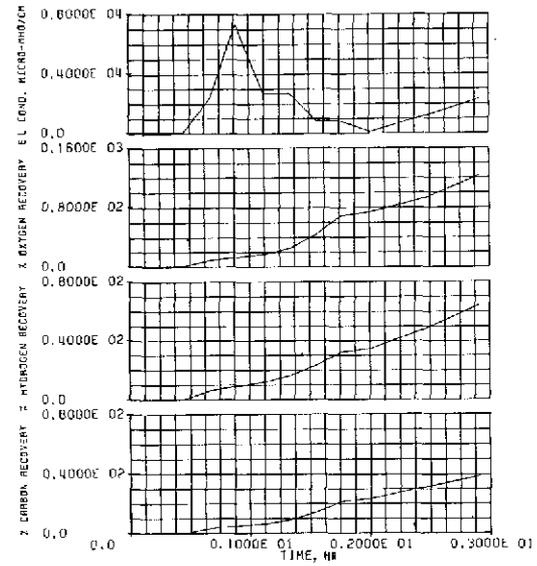
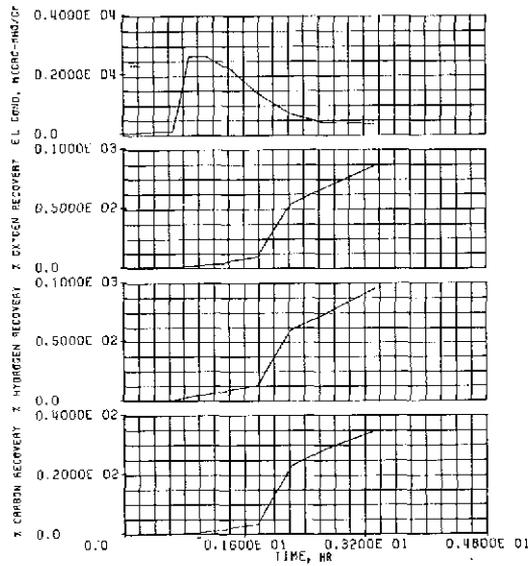
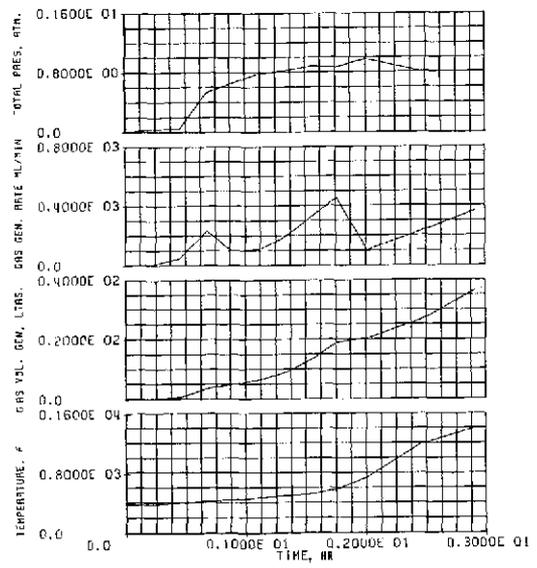
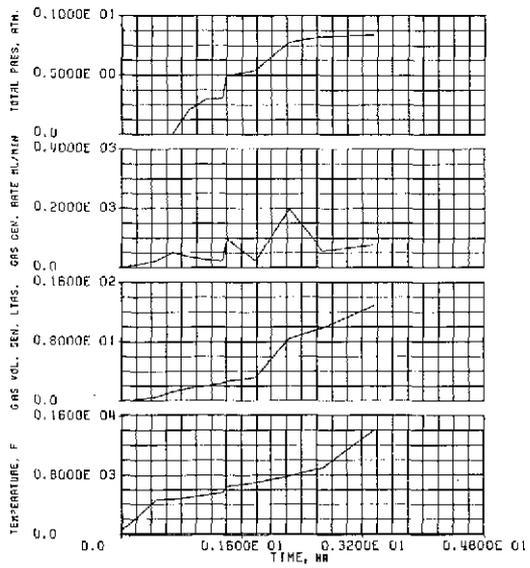
SAMPLE OF DATA REDUCTION COMPUTER
PRINTOUT FOR LARGE SCALE/BATCH TESTS

SAMPLE NO.	RUN TIME, MIN.	REACTOR TEMPERATURE, F	SYRINGE VOLUME, ML.	VOLUME COLLECTED, ML.	GAS GENERATION RATE, ML/MIN	REACTOR VOLUME, ML.	DENSITY OF STEAM, G/ML	NUMBER OF COMPONENTS
26-02	15.8333	1196.00	38.0000	3.90000	39.0000	227.000	0.23160E-03	5.
SET CONDITIONS			MEASURED CONDITIONS					
LIQUID FEED RATE, ML/MIN	0.330000		LIQUID FEED RATE	0.315789				
GAS FEED RATE, ML/MIN	0.0		LIQUID COLLECT. RATE	0.240000				
SPACE VELOCITY, 1/HR	376.617		SPACE VELOCITY, 1/HR	360.399				
CONTACT TIME, SEC	9.55877		CONTACT TIME, SEC	9.98892				
PSIG	ATM		TOTAL	H2	CO	CH4	CO2	H2O
REACTOR PRESSURES								AIR
15.3500	1.04450							6 MIN CH3OH 12.5 MIN.
CHROMATOGRAPH PEAKS, CM			2.5500	10.0800	1.5400	12.2200	0.0	
PARTIAL PRESSURES, ATM		1.0074	0.4720	0.2948	0.0542	0.1864	0.0	
EXPERIMENTAL MOLE FRACTIONS			0.4685	0.2926	0.0538	0.1850	0.0	
PREDICTED MOLE FRACTIONS (H2O FREE)			0.1196	0.0041	0.0	0.0578	0.8185	
			0.6592	0.0225	0.0	0.3183		
PREDICTED PARTIAL PRESSURES (H2O FREE)			0.1249	0.0043	0.0	0.0603	0.8550	
			0.6592	0.0225	0.0	0.3183		
(EXPERIMENTAL/PREDICTED) MOLE FRACTION (H2O FREE)			3.917	71.548*****	3.204	0.0		
			0.7108	12.9830*****	0.5813			
(EXPERIMENTAL/PREDICTED) PARTIAL PRESSURES (H2O FREE)			3.778	69.007*****	3.090	0.0		
			0.7161	13.0793*****	0.5856			
RATE OF CARBON GENERATION, GM-ATOM/MIN			0.88008E-03					
CARBON FEED RATE, GM-ATOM/MIN			0.11084E-02					
(GENERATION/FEED) RATE RATIO			79.399					
MOLAR GENERATION RATE, MOLE/MIN			CO	CH4	CO2	CH3OH		
			0.0005	0.0001	0.0003	0.0		
RATE OF HYDROGEN GENERATION, GM-ATOM/MIN			0.19081E-02					
HYDROGEN FEED RATE, GM-ATOM/MIN			0.20242E-02					
(GENERATION/FEED) RATE RATIO			94.264					
MOLAR GENERATION RATE, MOLE/MIN			H2	CH4	H2O	CH3OH		
			0.0008	0.0001	0.0	0.0		
RATE OF OXYGEN GENERATION, GM-ATOM/MIN			0.10974E-02					
OXYGEN FEED RATE, GM-ATOM/MIN			0.10137E-02					
(GENERATION/FEED) RATE RATIO			108.26					
MOLAR GENERATION RATE, MOLE/MIN			CO	CO2	H2O	CH3OH		
			0.0005	0.0003	0.0	0.0		

B-1/A

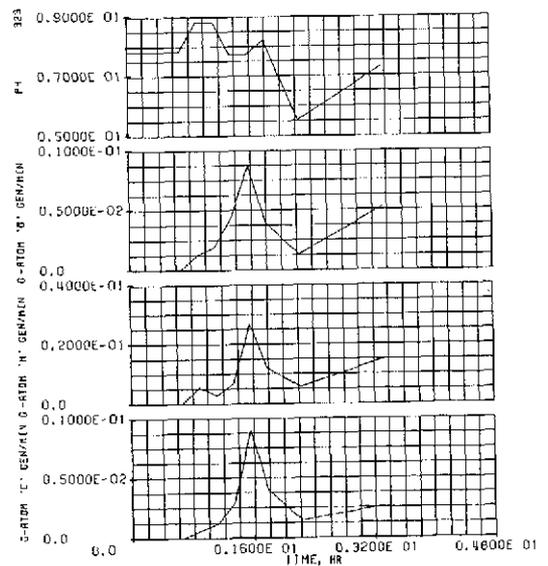
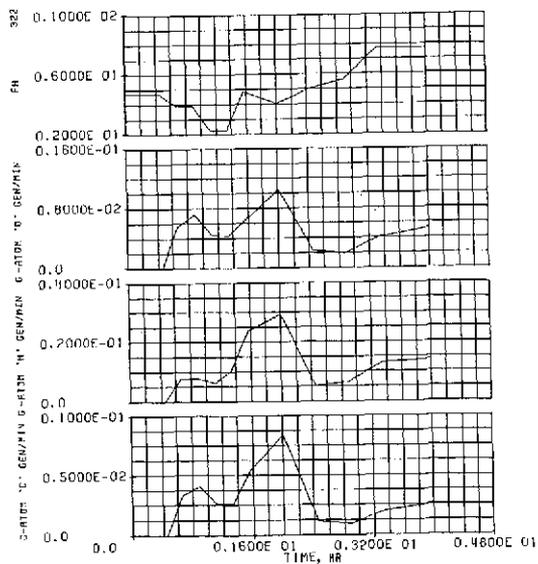
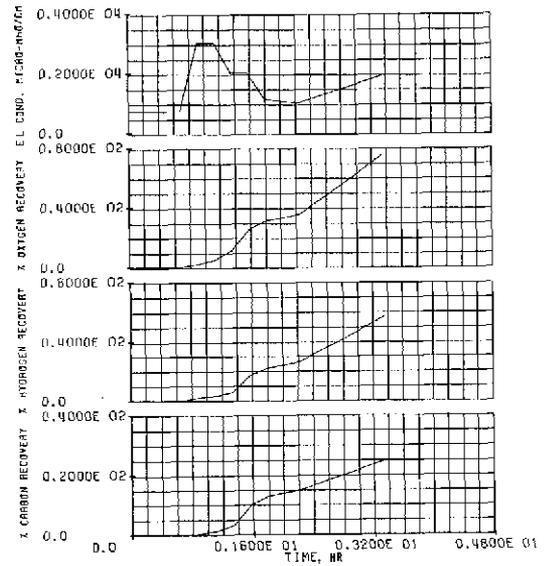
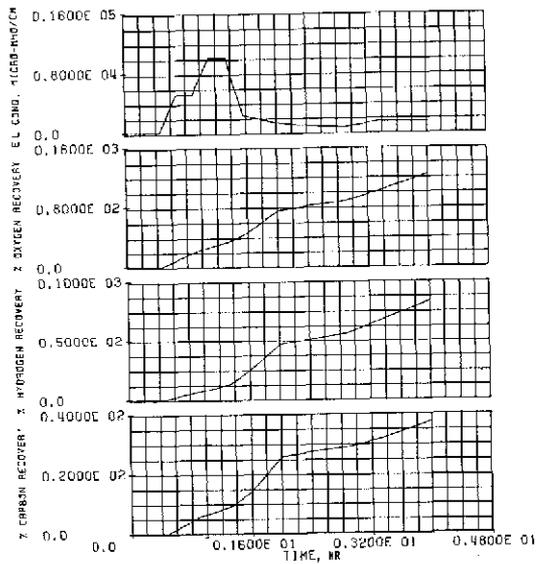
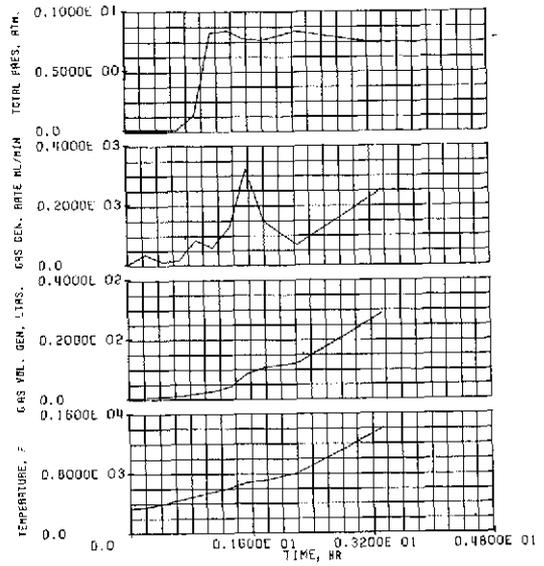
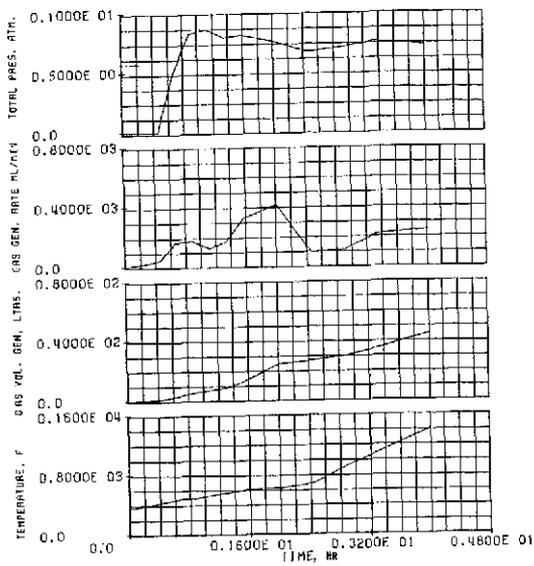
APPENDIX C

COMPUTER SUMMARY PLOTS



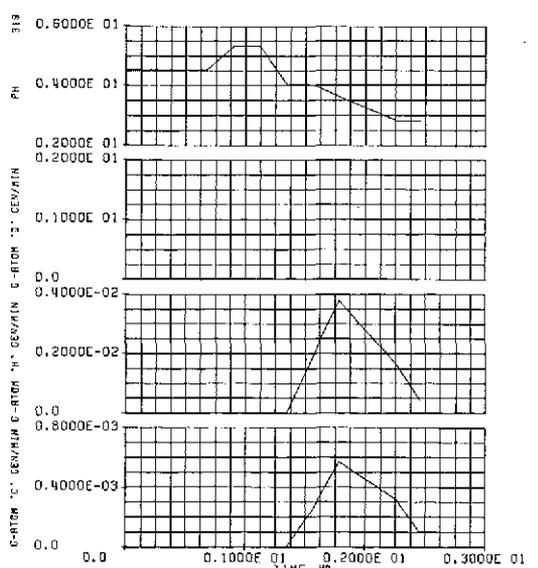
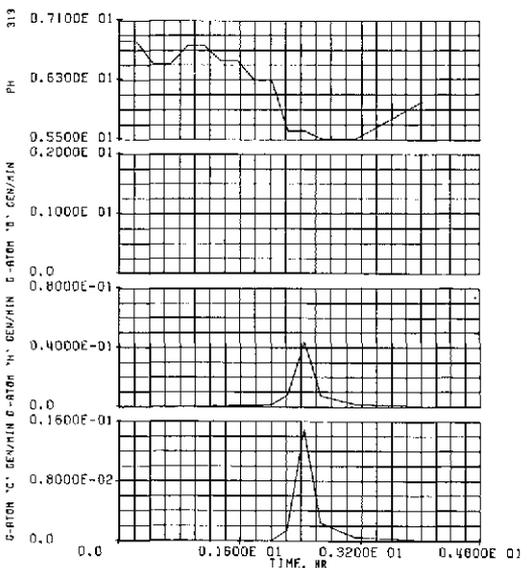
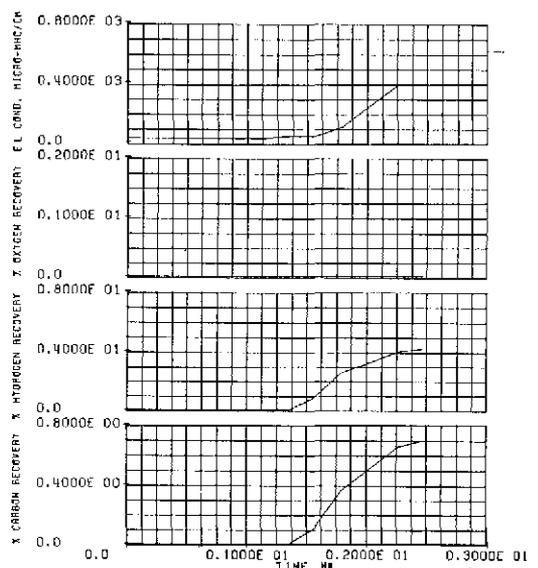
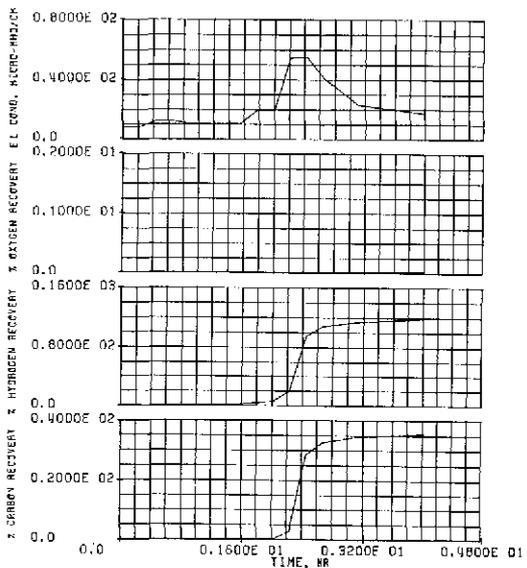
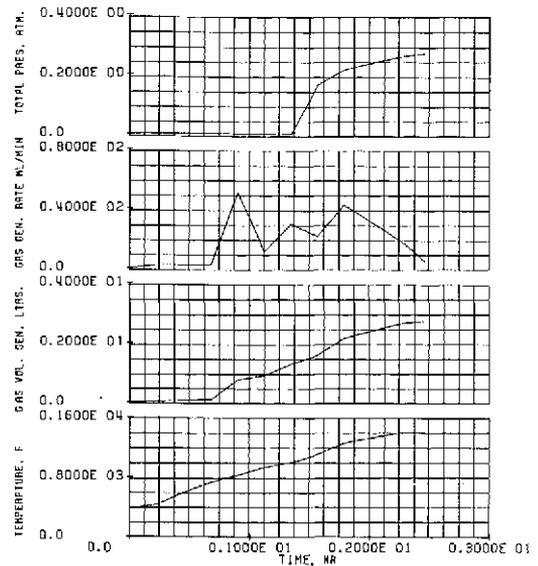
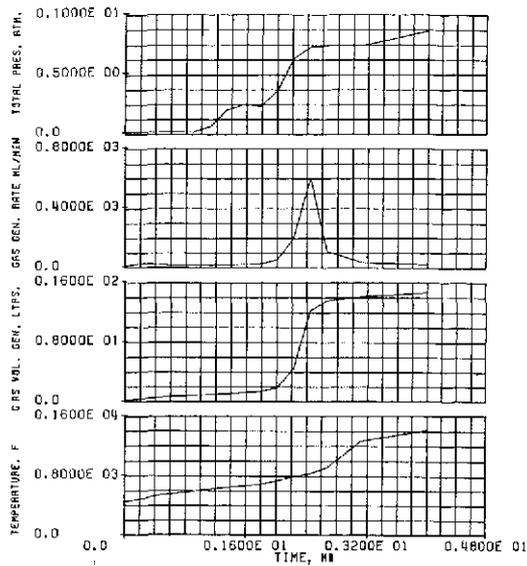
RUN 315 - SPACECRAFT MIXTURE

RUN 318 - SYNTHETIC MIXTURE



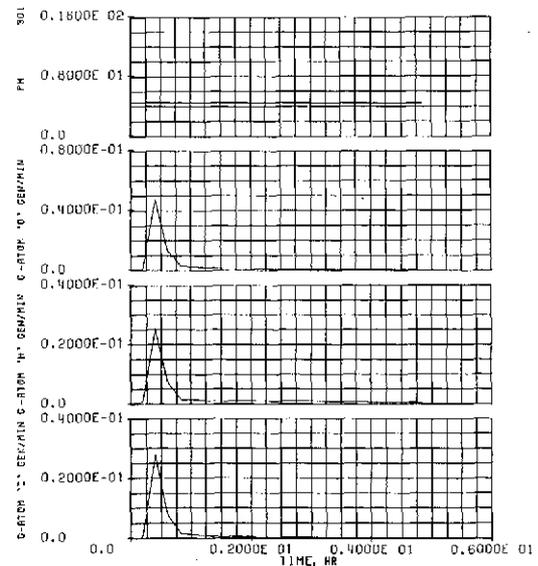
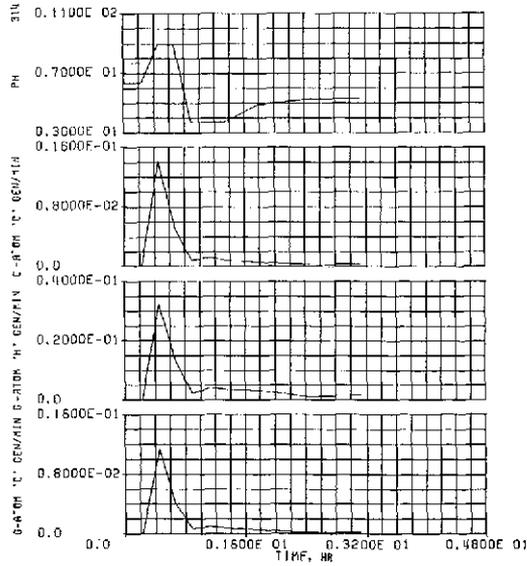
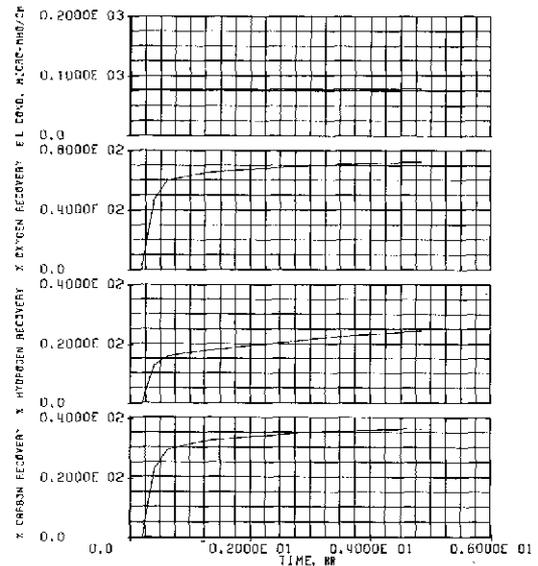
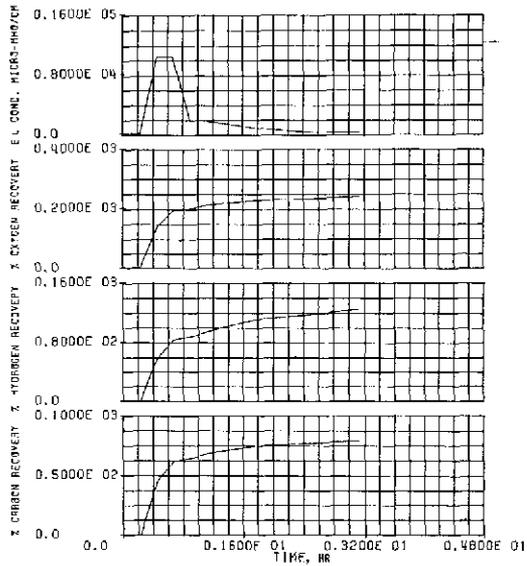
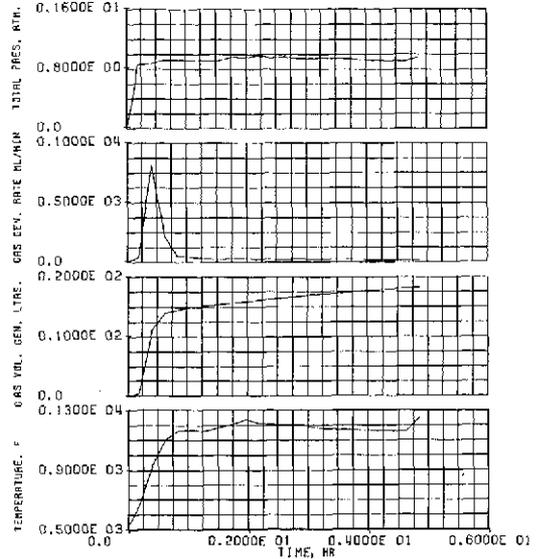
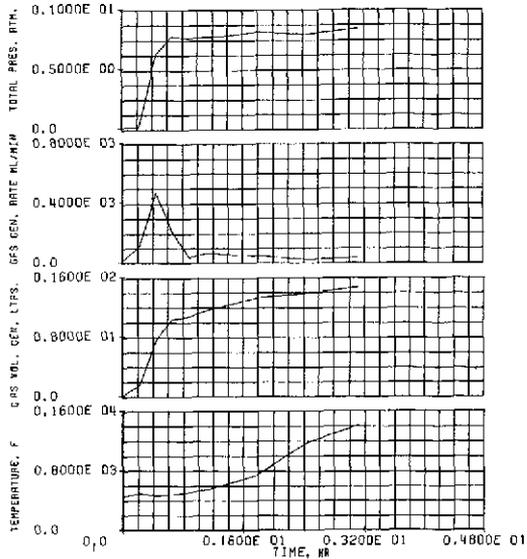
RUN 322 - SPACECRAFT MIXTURE

RUN 323 - SPACECRAFT MIXTURE



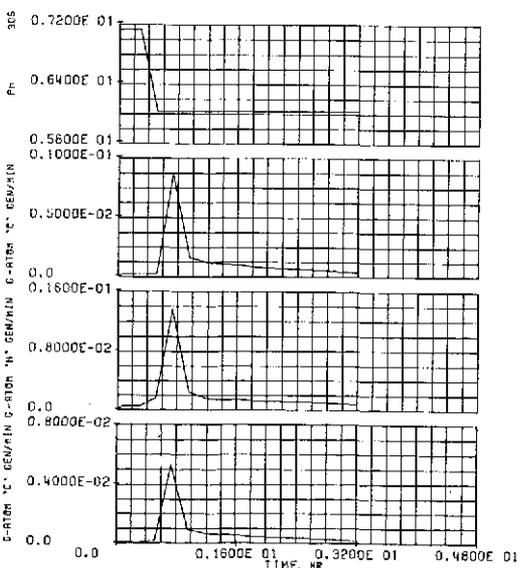
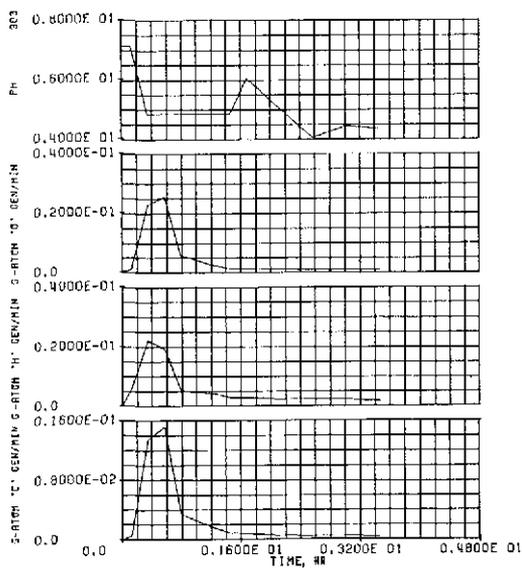
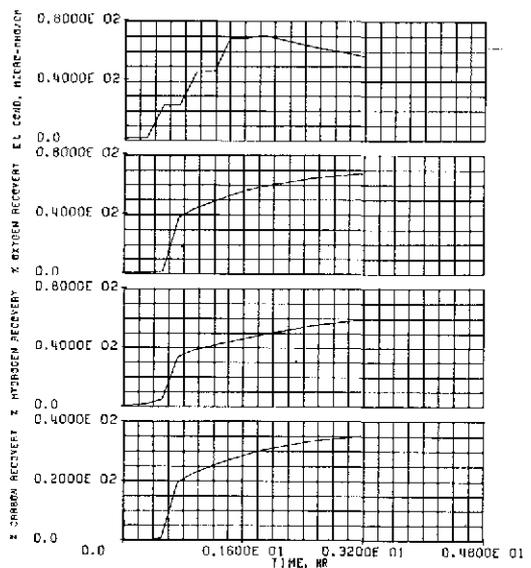
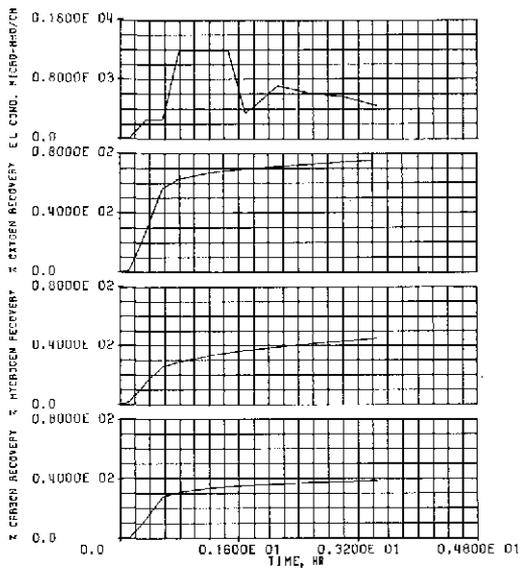
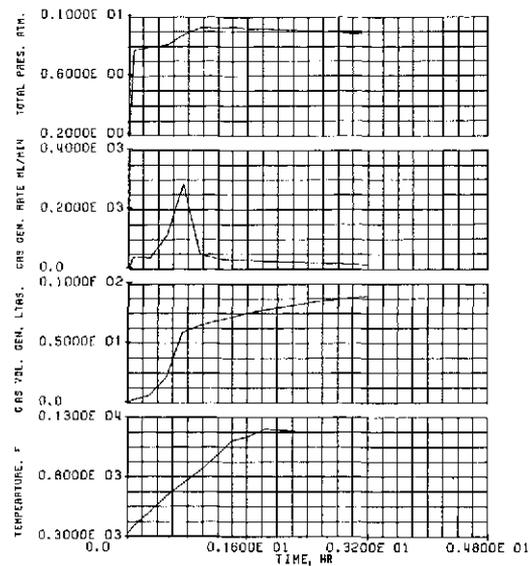
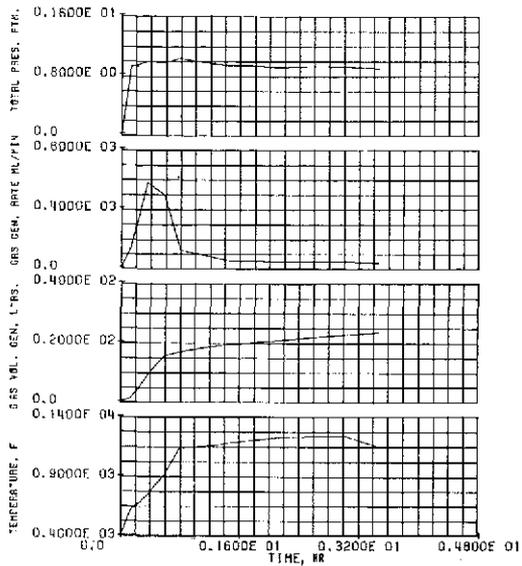
RUN 313 - POLYETHYLENE

RUN 319 - POLYETHYLENE



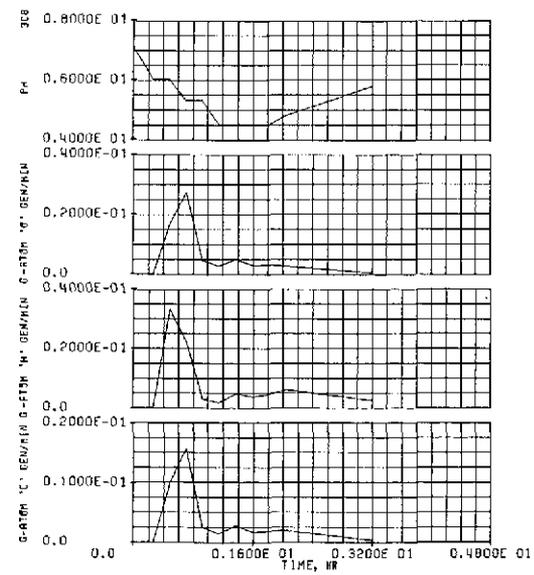
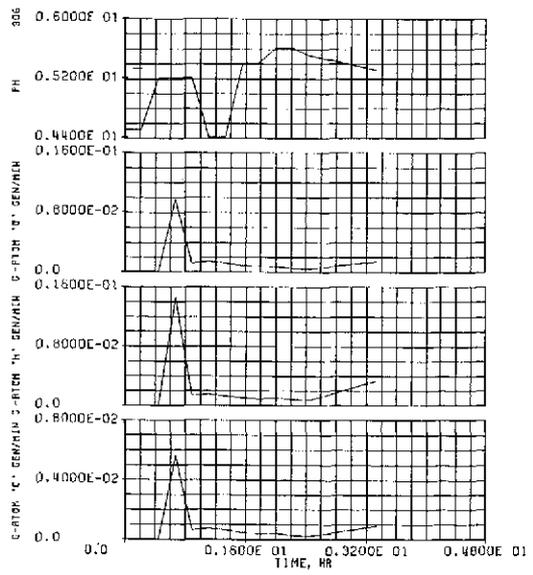
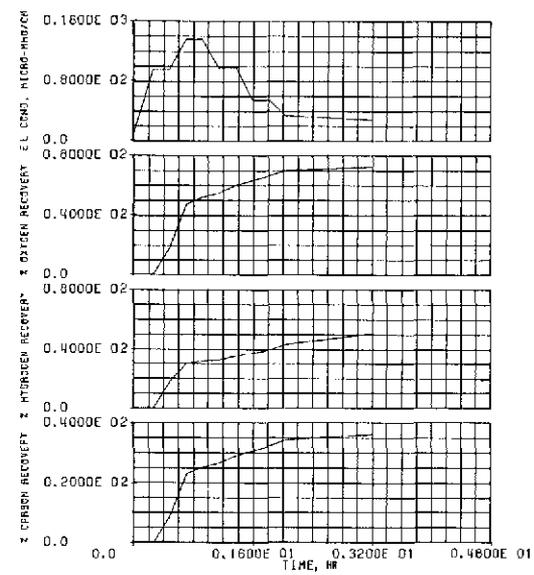
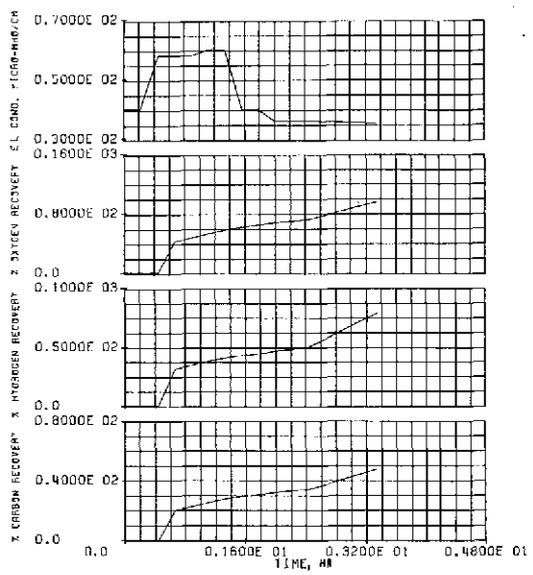
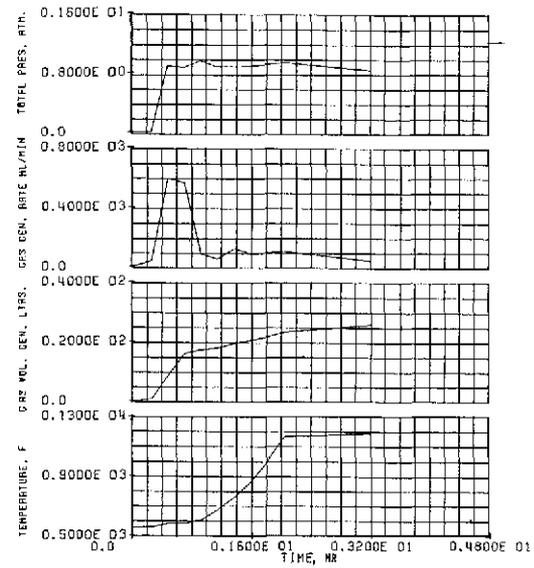
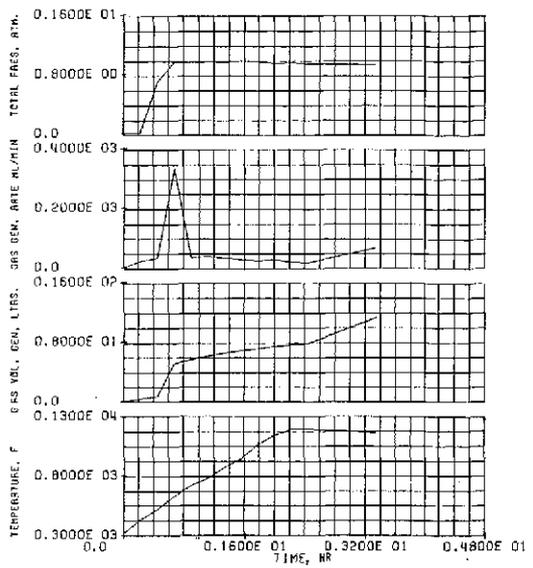
RUN 314 - METHIONINE

RUN 301 - CELLULOSE



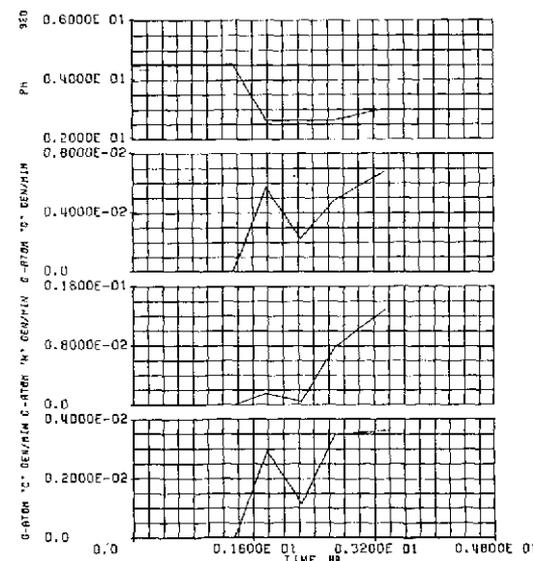
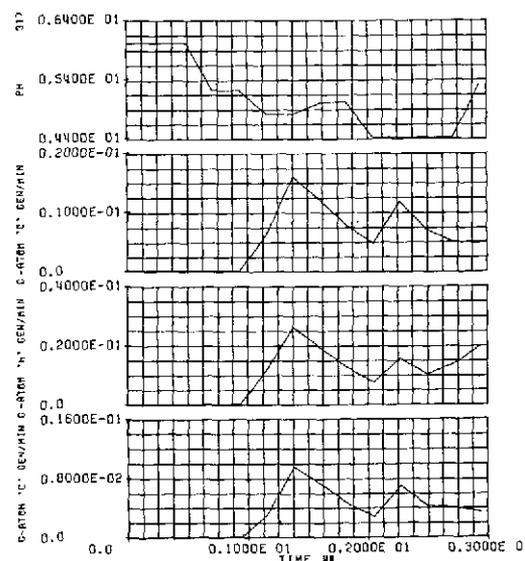
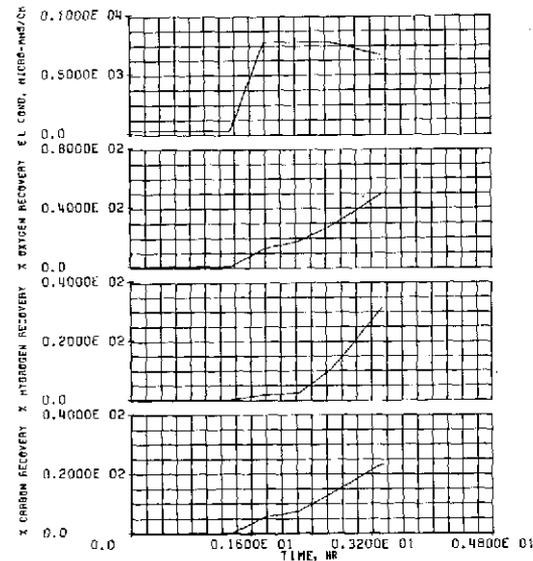
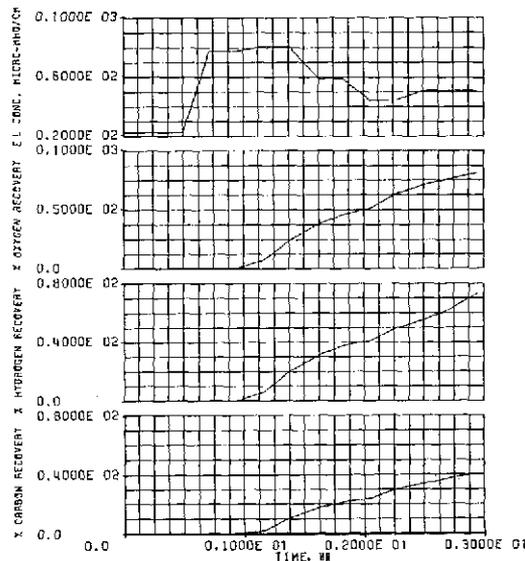
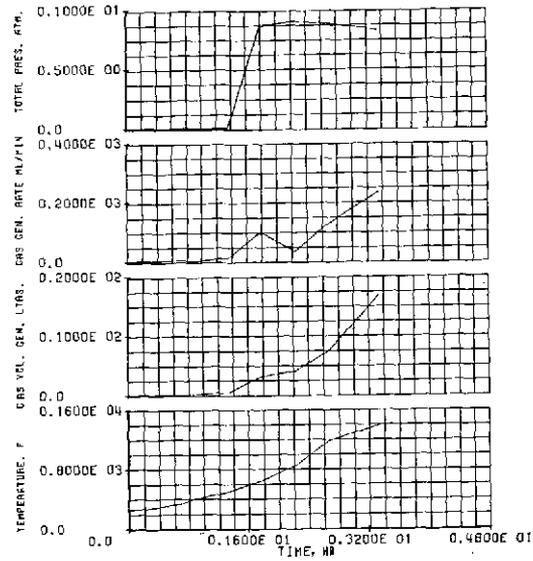
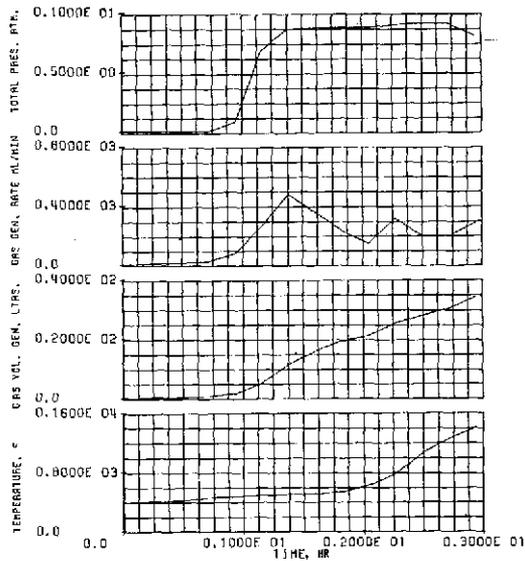
RUN 303 - CELLULOSE & KIO₃

RUN 305 - CELLULOSE



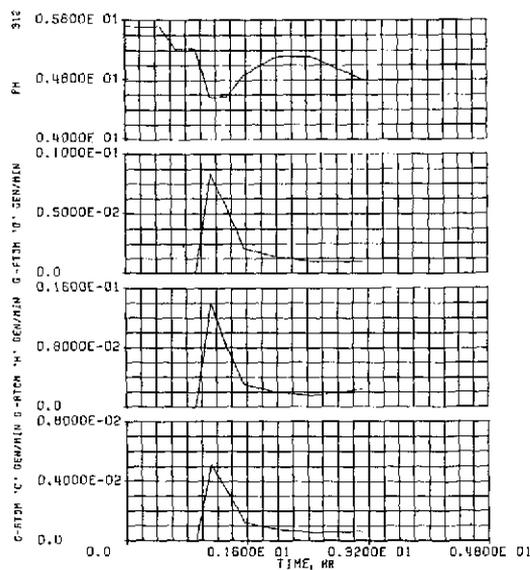
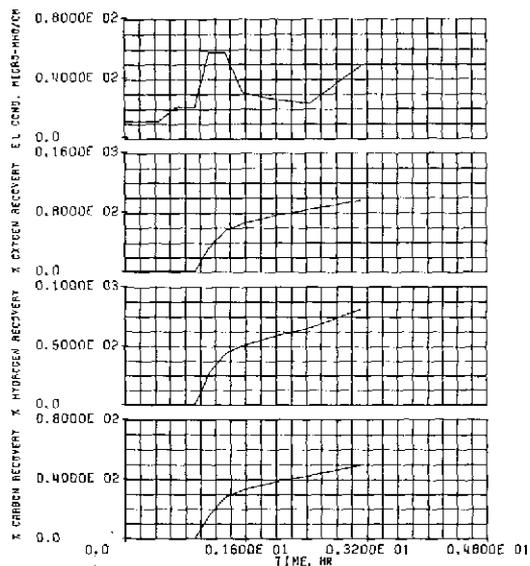
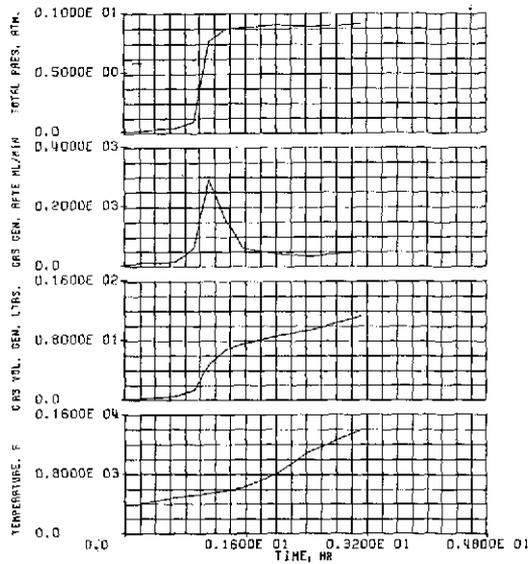
RUN 306 - CELLULOSE

RUN 308 - CELLULOSE

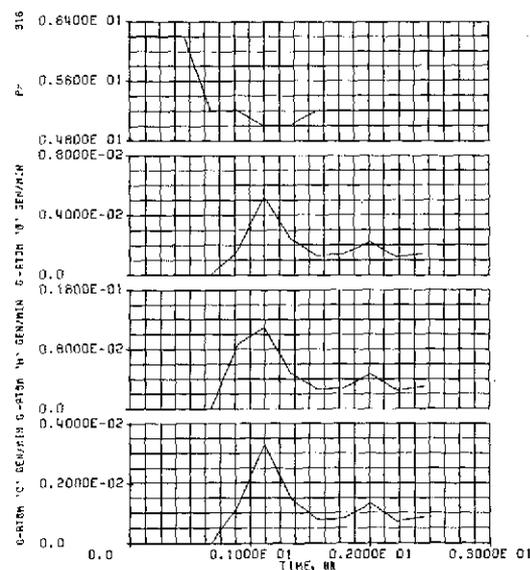
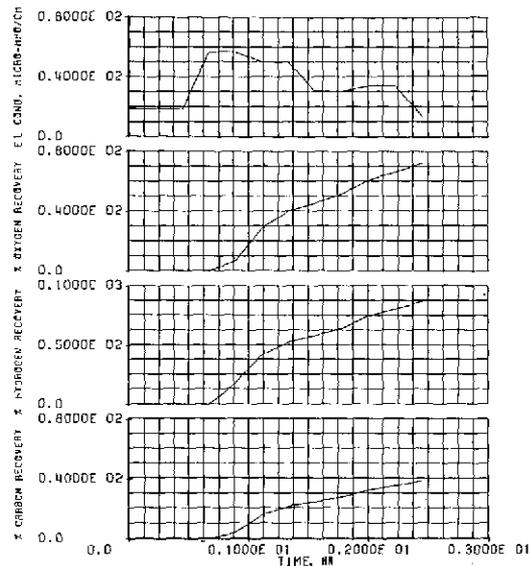
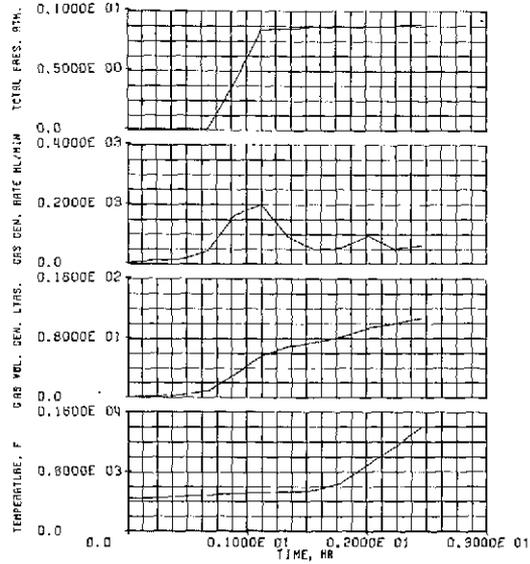


RUN 317 - CELLULOSE

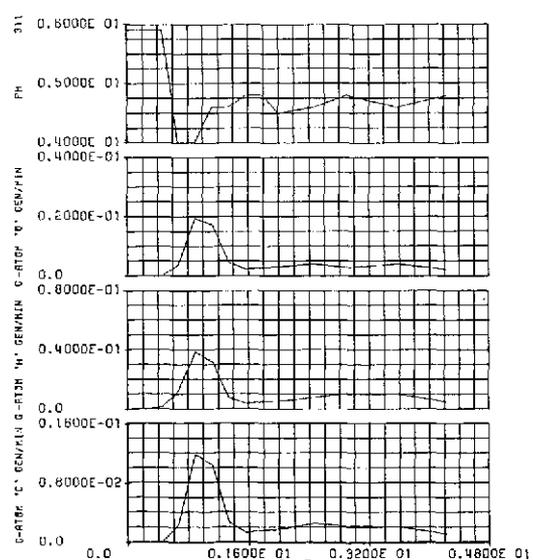
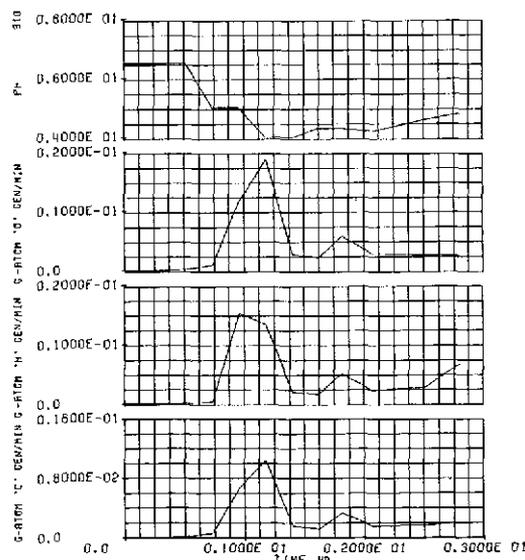
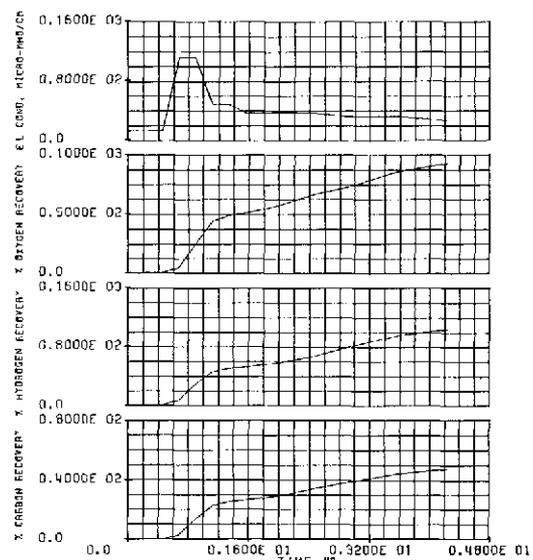
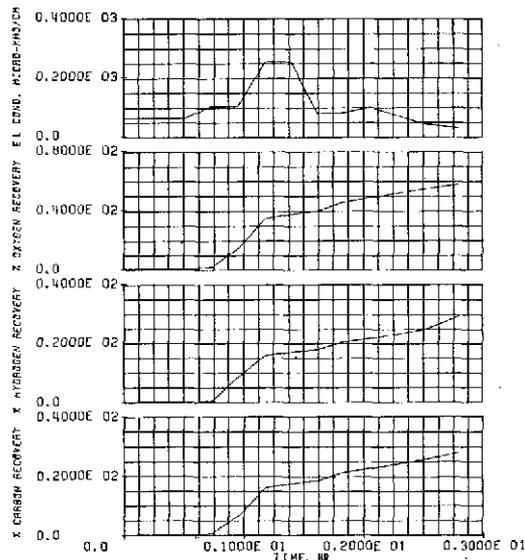
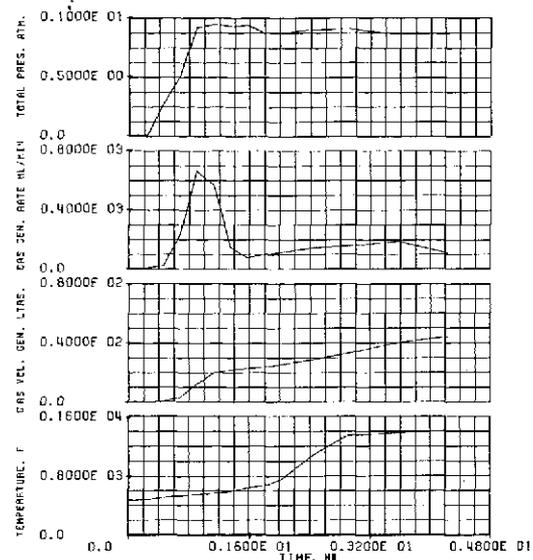
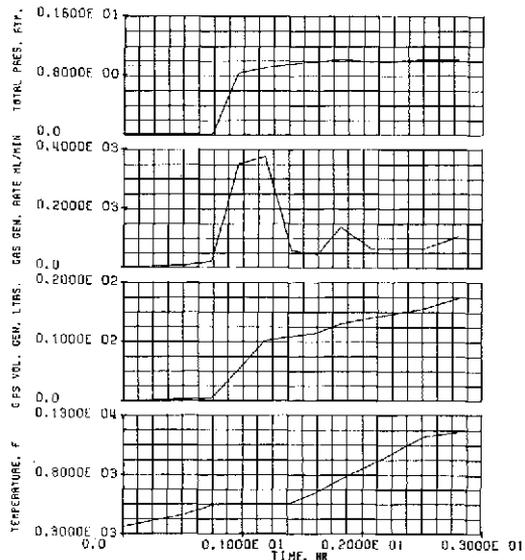
RUN 320 - CELLULOSE



RUN 312 - CELLULOSE

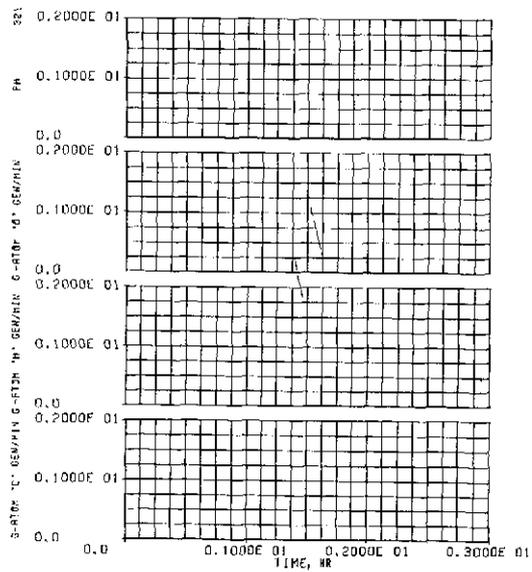
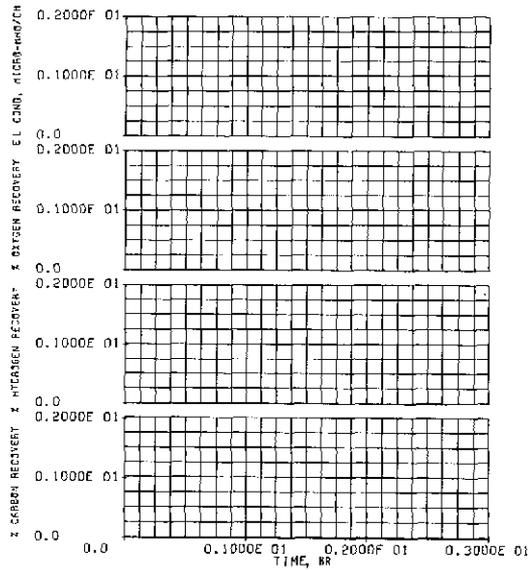
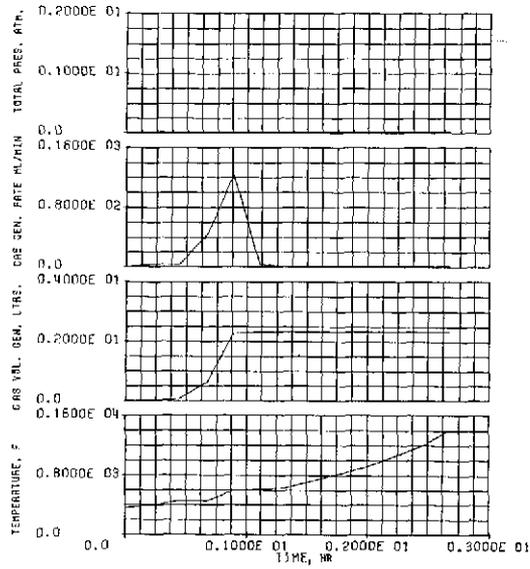


RUN 316 - CELLULOSE



RUN 310 - CELLULOSE

RUN 311 - CELLULOSE



RUN 321 - CELLULOSE

APPENDIX D

TEST PLAN

CENTRAL WASTE PROCESSING SYSTEM

MASTER TEST PLAN

PREPARED UNDER CONTRACT NAS 9-12730

by

HAMILTON STANDARD
DIVISION OF UNITED AIRCRAFT CORPORATION
WINDSOR LOCKS, CONNECTICUT

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MANNED SPACECRAFT CENTER
HOUSTON, TEXAS

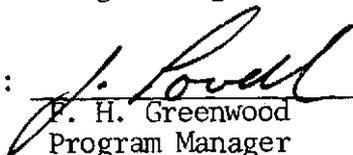
AUGUST 1972

Prepared by:



W. Coe
Program Engineer

Approved by:



F. H. Greenwood
Program Manager

for

1.0 GENERAL INFORMATION1.1 Scope

The purpose of this plan is to define the parametric tests to be performed on the full scale 1-g six man steam reformation laboratory test system, and to define the test method to be used. This testing will be conducted under NASA MSC contract NAS 9-12730 at the Hamilton Standard, Space Systems Department test facilities.

1.2 Applicable Documents

NASA MSC Contract NAS 9-12730
Hamilton Standard Program Operating Plan
for Contract NAS 9-12730
Computer Program for Chromatographic Data
Reduction HS 465
Computer Program for Calculation of Complex
Chemical Equilibrium Compositions HS 554
Space Station Prototype document A-8

1.3 Laboratory Test Systems Functional Description

The laboratory test system to be used for this test program is a Hamilton Standard rig constructed from commercial hardware which simulates a spacecraft steam reformation system. This facility permits the preparation and testing on a continuous basis over the full range and amounts of spacecraft waste products currently anticipated.

The facility consists of a "slurrifier" section for waste preparation, steam reformation section, and a reaction products collection section.

The waste preparation section consists of a mixing tank and material grinder with a pump to recirculate the waste mixture and maintain a homogeneous slurry of the components. Screens will be added to the system to limit the size of particles of undissolved solids based upon the results of this test program. A variable speed pump introduces the waste material into the steam reformation section.

The steam reformation section consists of a two foot long, one inch diameter waste flash boiler and an eighteen foot long, one inch diameter stainless steel reaction pipe. The boiler and reaction pipe are heated by means of an integrally wound nichrome wire furnace. The temperature and power available to the boiler and reformer section can be separately adjusted

by means of variable transformers and monitored by means of thermocouples and voltmeters and ammeters. The reformer section has gas sample ports located at three foot spacings along its length for obtaining samples for direct analysis by the process chromatograph or sample collection for use with other techniques of analysis.

The reaction products collection system separates the reformation products into insoluble ash, condensable liquids and non-condensable gasses. The ash will be collected in a drum equipped with fine stainless steel screens which will allow the gasses and vapors to pass through. The vapors will then be liquified in a water cooled condensor and collected while the non-condensable gasses will be discharged to the process chromatograph or a sample collection bottle.

1.4 Test Condition Tolerances

Unless otherwise specified the following tolerances will apply to the referenced test parameters:

Temperature	+ 15°F @ 1000°F
Pressure	+ .5 psi @ Ambient
Waste Flow Rate	+ 5% of Nominal
Waste Constituents	+ 5% of Nominal

1.5 Test Data Tolerances

Product Gas Composition	+ 5% of Major Components
Product Gas Volume	+ 2% of Sample Volume
Product Liquid Composition	+ 5% of Major Compounds
Product Liquid Volume	+ 2% of Sample Volume
Product Solid Composition	+ 5% of Major Components
Product Solid Weight	+ 2% of Sample Weight
Heater Voltage	+ 3% of Full Scale
Heater Current	+ 3% of Full Scale

2.0 TEST PLAN AND PROCEDURES

The test program to be conducted will consist of evaluation of pure chemical substances and typical spacecraft wastes, singly or in mixtures, as described in paragraphs 2-2, 2.3 and 2.4. The overall test schedule is shown in figure 2-1.

2.1 Laboratory Test System Schematic

The test rig schematic is shown in figure 2-2.

2.2 Pure Chemical Substances

Chemicals representative of the carbohydrate, protein, and hydrocarbon type wastes as well as typical halogens and sulfur compounds, will be evaluated on a continuous flow basis as pure substances. The processing temperature and reaction contact time will be varied in order to establish their effect upon the kinetics of the reaction. Each substance will be tested with a water to chemical ratio typical of its concentration in spacecraft waste mixtures.

The chemicals selected for each type of waste and the test conditions for each test are defined below.

Each test condition as defined below will be maintained for a period of four (4) hours in order to achieve steady state conditions. During the test period the gaseous mixture produced will be monitored by a process chromatograph in order to verify that equilibrium conditions have been established. An analysis of these gases will be made from the process chromatograph and if necessary, a sample will be taken for further analysis by other techniques such as infrared spectroscopy or mass spectroscopy. Samples of the non-gaseous residues will also be collected to be analyzed for minerals and carbonaceous constituents. The test apparatus will be cleaned between test conditions either mechanically or by means of a steam flush.

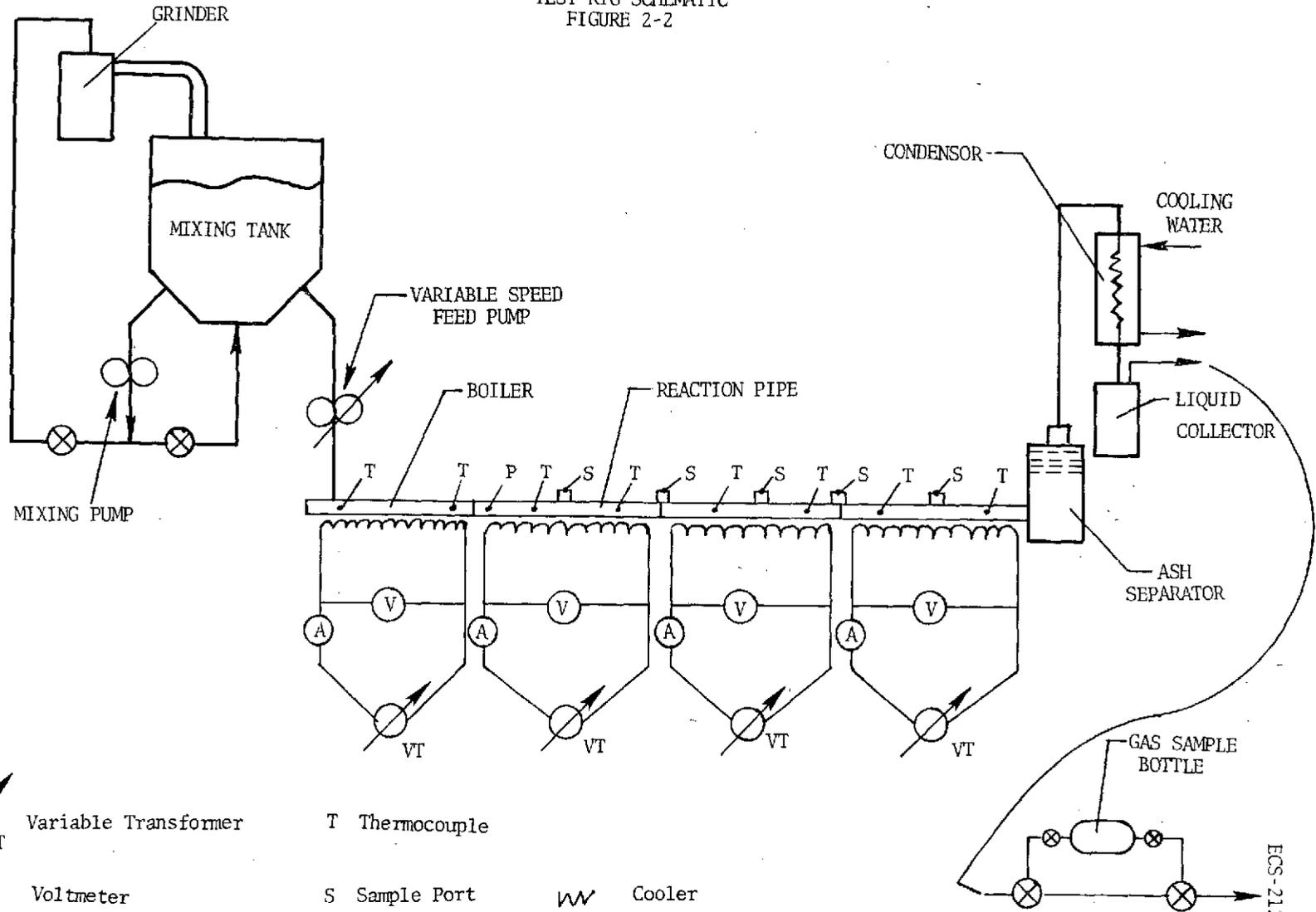
The values of the parameters selected below for evaluation are subject to change based upon the results obtained as the test program proceeds.

1972		1973		
NOV.	DEC.	JAN.	FEB.	
				PURE CHEMICALS EVALUATION TEST NUMBERS 1 - 21
				WASTE PARTICLE SIZE EVALUATION TEST NUMBERS 22 - 25
				SPACECRAFT WASTE MIXTURE EVALUATION TEST NUMBERS 26 - 29

A

TEST SCHEDULE
 FIGURE 2-1

TEST RIG SCHEMATIC
 FIGURE 2-2



- | | | | | | |
|--|----------------------|---|----------------|--|-----------------|
| | Variable Transformer | T | Thermocouple | | Cooler |
| | Voltmeter | S | Sample Port | | Pump |
| | Ammeter | | Ball Valve | | Pump - Variable |
| | Heater | P | Pressure Gauge | | |

ECS-2122-1-003

Test No.	Material Category	Chemical	Concentration Weight %	Reaction Temp. -°F	Reaction Time-Sec.
1	Carbohydrate	Cellulose	5%	1200	1
2		Cellulose	5%	1200	5
3		Cellulose	5%	1200	30
4		Cellulose	5%	1400	1
5		Cellulose	5%	1400	5
6	Protein/Sulfur	Methionine	1%	1200	1
7		Methionine	1%	1200	5
8		Methionine	1%	1200	30
9		Methionine	1%	1400	1
10		Methionine	1%	1400	5
11	Repeat test based upon conditions selected from tests 6-10, but with the addition of catalyst* to promote conversion of ammonia to nitrogen.				
12	Halogen	{ Cellulose & Pretreat Chemical**	{ Cellulose 4.5% Chemical** .5%	1200	1
13				1200	5
14				1200	30
15				1400	1
16				1400	5
17	Hydrocarbon	Polyethylene	0.5%	1200	1
18			0.5%	1200	5
19			0.5%	1200	30
20			0.5%	1400	1
21			0.5%	1400	5

*Catalyst - Ruthenium Tri Chloride
1% by weight application
**Pretreat Chemical - Containing Iodophor (IO₃) 3% by weight

2.3 Particle Size Evaluation

Tests will be conducted to evaluate the effect of particle size upon the steam reformation process. The material to be used will be cellulose and polyethelene and the particle sizes will be typical of ground materials. The tests are defined as follows:

Test No.	Chemical	Concentration Weight %	Particle Size Microns	Reaction Temp. -°F	Reaction Time-Sec.
22	Cellulose	5%	840	1400	5
23	Cellulose	5%	250	1400	5
24	Polyethylene	0.5%	840	1400	5
25	Polyethylene	0.5%	250	1400	5

The tests will be conducted in the same manner as described in section 2.2. Particular emphasis will be placed upon inspection of the boiler for non-volatilized feed material.

2.4 Spacecraft Waste Mixture

A typical spacecraft solid waste mixture will be prepared consisting of feces, urine, paper and plastic. This mixture will be slurried and tested as follows:

Test No.	Waste Type	Concentration Weight %	Reaction Temp. - °F	Reaction Time - Sec.
26	Spacecraft Mixture	5	1400	1
27		5	1400	5
28		5	1400	30
29		10	1400	5

The testing will be conducted in the manner described for section 2.2.

2.5 Test Schedule and Test Sequence

The overall schedule for the conducting of tests defined in sections 2.2, 2.3 and 2.4 is presented in figure 2-1. The test number is not necessarily the testing sequence.

The initial sequence of tests will be as follows:

<u>Test Sequence</u>	<u>Test Number</u>
1	3
2	2
3	1
4	8
5	7
6	6
7	4
8	5
9	9
10	10
11	11

This sequence and the parameters of tests may be altered depending upon the results obtained as the test program proceeds.

2.6 Data

The data from each test will be recorded on Hamilton Standard Log Sheets. This data will consist of the rig operational parameters as well as the results of chemical analyses performed. The performance data calculated from each test will be plotted and compared with the performance predicted from theoretical chemical equilibria.