

BIOCHEMICAL OXYGEN DEMAND REDUCTION  
OF SEMI-CHEMICAL NEUTRAL SULFITE  
WASTE BY HEAT HYDROLYSIS

by

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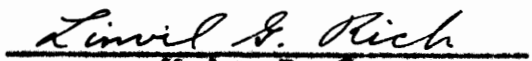
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## TABLE OF CONTENTS

	Page
I. Introduction . . . . .	8
II. Literature Review. . . . .	10
Disposal of S.N.W. . . . .	10
Pulping Processes . . . . .	11
III. Object of Investigation . . . . .	14
IV. Experimentation. . . . .	15
Plan of Investigation . . . . .	15
Biochemical Oxygen Demand Test. . . . .	15
Gas Analysis. . . . .	18
Preliminary Investigation . . . . .	18
Principle Investigation . . . . .	19
Materials . . . . .	21
Apparatus . . . . .	22
Data and Results. . . . .	23
Sample Calculations . . . . .	41
V. Discussion of Results. . . . .	44
Reduction of B.O.D. Obtained by	
Heat Hydrolysis . . . . .	44
Limitations . . . . .	54
VI. Conclusions. . . . .	59
VII. Summary . . . . .	61
VIII. Acknowledgements . . . . .	63
IX. Bibliography . . . . .	64

	Page
X. Vita . . . . .	66
XI. Appendix . . . . .	67
Data Obtained During Cook of Sample . .	68

## TABLES

	Page
Table 1. Data Obtained From Determination of B.O.D. Reduction of S.N.W. Liquor by Heat Hydrolysis . . . . .	26
Table 2. Data Obtained From Determination of B.O.D. Reduction of S.N.W. Liquor by Heat Hydrolysis . . . . .	28
Table 3. Data Obtained From Determination of B.O.D. Reduction of S.N.W. Liquor by Heat Hydrolysis . . . . .	30
Table 4. Summary of Analytical Results and Calculated Values Based Thereon for Semi-Chemical Soda Pulping Waste . . . . .	48
Table 5. Percent of Oxygen Present in a Gas Produced During a Cook of Semi- Chemical Neutral Sulfite Waste to Which Oxygen had Been Added. . . . .	55
Table 6. Percent of Oxygen Present in a Gas in Contact with Semi-Chemical Neutral Sulfite Waste Liquor to Which Oxygen had Been Added. . . . .	56

## FIGURES

	Page
Figure 1. Equipment Used in The Experimental Part of Investigation of Semi-Chemical Neutral Waste B.O.D. Reduction by Heat Hydrolysis . . . . .	24
Figure 2. Relationship of Gas Produced to B.O.D. Reduction in Heat Hydrolysis of Semi- Chemical Neutral Sulfite Waste . . . . .	32
Figure 3. Relationship of O <sub>2</sub> Produced to B.O.D. Reduction in Heat Hydrolysis of Semi- Chemical Neutral Sulfite Waste . . . . .	33
Figure 4. Relationship of Carbon Monoxide Produced to B.O.D. Reduction in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste .	34
Figure 5. Relationship of CO <sub>2</sub> Produced to B.O.D. Reduction in Heat Hydrolysis of Semi- Chemical Neutral Sulfite Waste . . . . .	35
Figure 6. Relationship of Time of Cook to B.O.D. Reduction in Heat Hydrolysis of Semi- Chemical Neutral Sulfite Waste . . . . .	36
Figure 7. Relationship of Highest Temperature Obtained to B.O.D. Reduction in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	37

Figure 8.	Relationship of Change in Solids to B.O.D. Reduction in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	38
Figure 9.	Relationship of Change in pH to B.O.D. Reduction in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	39
Figure 10.	Relationship of Highest Pressure Obtained to B.O.D. Reduction in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	40
Figure 11.	Relationship of Degree of Reaction Rate to Temperature in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	50
Figure 12.	Relationship of Degree of Reaction Rate to Temperature in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	51
Figure 13.	Relationship of Degree of Reaction Rate to Temperature in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	53

Figure 14.	Relationship of Degree of Reaction Rate to Temperature in Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . . . . .	53
Figure 15.	Effect of Addition of Oxygen on The Concentration of Oxygen in The Gas Produced During Heat Hydrolysis of Semi-Chemical Neutral Sulfite Waste . .	57



## I INTRODUCTION

The discharge of deleterious materials into lakes, rivers and tide waters has been the practice of municipalities and industry for many years. Little consideration has been given to the ill effects created by this discharge until recently. Now, at the insistence of the public, the Federal, State and many Local governments have legislation to control the discharge of such wastes. The methods for the treatment of domestic and some types of industrial wastes are well known, but various industries produce wastes high in biochemical oxygen demand (B.O.D.) which are not reduced by conventional methods.

The domestic Pulp and Paper industries use a tremendous quantity of water in their manufacturing processes and produce a waste that exerts a high oxygen demand on receiving waters. The oxygen requirements of the waste from a pulp plant discharging one half million gallons per day is equivalent to that of the waste from a city having a population of 167,000.

Realizing that research projects on disposal of their waste was necessary, the Pulp and Paper manufactureries organized the National Council for Stream Improvement, Inc. The council assigned a project to V.P.I. in 1947 on disposal of Semi-Chemical Neutral Sulfite Waste (S.N.W.). Although many methods of disposal have been tried to date, all have either proven

unsatisfactory or unecomonical. A similar project was established at Oregon State College for research on Full Acid Sulfite waste liquor. This research determined the B.O.D. content of the Full Acid Sulfite liquor could be reduced as much as 70 to 80 per cent by a process, termed "Heat Hydrolysis", in which the liquor is heated in a closed container to various temperatures and pressures.

Since there is a great similarity between S.N.W. waste and Acid Sulfite Waste (they differ only in the concentration of chemicals added and the length of cooking time), the Council assigned to the V.P.I. project the problem of ascertaining if Heat Hydrolysis would reduce the high B.O.D. content of the S.N.W. liquor.

## II LITERATURE REVIEW

All research on the disposal of S.N.W. has been conducted at V.P.I.; therefore, a short summary of this work and the results obtained therefrom will be presented. Included, also, is a description of the Sulfite and S.N.W. process of making pulp and a comparison of the two processes.

Disposal of S.N.W.

The following summary of work conducted at V.P.I. on S.N.W. was obtained from an unpublished Bulletin by R. E. Opferkuch, Jr., Resident Engineer for the National Council. The types of treatment investigated to date are: Combined Treatment (treatment with domestic sewage), Anaerobic Digestion and Foam Phase Aeration.

Combined Treatment. Combined treatment was employed to study the effect of treating S.N.W. along with domestic sewage. Results showed that when S.N.W. was added to sewage the removal of suspended solids and the decrease in removal of B.O.D. by settling was proportional to the amount of S.N.W. added and that the chlorine demand of the mixed waste increased two fold for a 10% increase of the S.N.W. liquor.

Anaerobic Digestion. Anaerobic digestion, one of the most efficient tools of the sanitary engineer for the degradation of organic matter by bacteria, was tried to determine the digester loading that would give the highest reduction in

B.O.D. It was found that a B.O.D. loading of 0.0061b/cu.ft./day gave a 70 to 75% reduction in B.O.D., whereas a loading of 0.071b/cu.ft./day gave a reduction of 30 to 40%.

Foam Phase Aeration. The property of the waste liquor to foam when agitated prompted the study of foam phase aeration and experiments were conducted at an eastern laboratory on sulfite waste liquor. A 70% reduction in B.O.D. was obtained by bubbling air through the liquor causing a slime suitable for bacterial growth to form on the side of the container.

The same method of treatment was tried on S.N.W. but the results obtained showed only a 35% reduction in 24 hours. It is interesting to note that when this treatment was tried on the Acid Sulfite waste by the researchers at Oregon State, the results obtained were similar to those obtained on S.N.W. at V.P.I.

### Pulping Processes

Sulfite Pulping. "The sulfite process," according to Caulfield(5), "is the elignification of wood by excess sulfurous acid and bisulfite. The alkaline bisulfites, commonly called "base" are largely of calcium with a few instances of magnesium and ammonia. The process is essentially the sulfonation of lignin which is then rendered by combination with the base."

The wood digestion process is as follows: "Chips are

charged to the digester and the cooking acid pumped in. As soon as the desired amount of each are charged, the cover is bolted on; pressure relief connections made; automatic instruments set; and the steam is turned into the digester. The temperature of cook is brought up to 230°F for not less than two hours. The temperature is then increased at a steady rate and at the end of a total of 5 or 6 hours, a temperature of 290°F is reached.

"When the blow-down time is decided, the sulfur dioxide and steam pressure within the digester are released until about 40 pounds remains. The remaining 40 pounds is used to blow the content through the blow line at the bottom of the digester to the blow pit."

Neutral Sulfite Semichemical Pulping. The neutral sulfite process consists of cooking wood or other cellulose materials with sodium sulfate buffered with sodium bicarbonate and carbonate.

Perry(14), explains the digestion of chips as follows:

"The chips are charged into the digester and steamed at atmospheric pressure for half an hour after which the liquor is added. The temperature is raised to 120°C and the chips thoroughly impregnated. The time required is about one hour for most woods between 120 and 130°C. It is during this period and within this range that most of the pyroligneous acids are formed and an excess of alkali is present to

neutralize it. At the end of this period the excess liquor is blown back to be refortified and reused in succeeding cooks. Cooking then proceeds with only the liquor contained in the chips plus a slight excess required for basting purposes. The temperature is raised rapidly to 170°C for the actual cooking period. The chips are then blown from the digester into the blow pit."

## III OBJECT OF INVESTIGATION

The object of the investigation was to determine whether or not the B.O.D. in Semi-Chemical Neutral Sulfite Pulp waste can be reduced by heating the liquor in a closed container to various temperatures and pressures; and, if such a reduction is possible, to determine the extent to which pH, dilution, and the addition of oxygen to the container are factors that limit B.O.D. reduction. Such information would help to establish the practicality of Heat Hydrolysis as an economical method of treating the S.N.W. waste.

## IV EXPERIMENTATION

Plan of Investigation

The preliminary investigation consisted of a review of literature, the construction of a test stand, and the construction and calibration of a thermocouple. The principle investigation was conducted to determine if dilutions, pH, and the addition of oxygen has any effect on the reduction of B.O.D., and to determine what cooking conditions produced the highest reduction in B.O.D.

Biochemical Oxygen Demand Test

The biochemical oxygen demand (frequently referred to as B.O.D.) of sewage, sewage effluents, polluted waters of industrial wastes is the oxygen (in parts per million) required to stabilize the decomposable organic matter by aerobic bacterial action(2). Rogers(18) further modifies this definition by stating, "it, (B.O.D.) as the amount of dissolved oxygen required by the mixed population of organisms in a sample during incubation for five days at the temperature of 20°C and at a 50% oxygen depletion." Phelps(16) defines B.O.D. differently by stating, "it, (B.O.D.) is not at all related to the complete oxygen requirements in chemical combustion but is determined wholly by the availability of the material as a bacterial food and by the amount of oxygen utilized by the bacteria during oxidation." Several



different procedures for conducting this test are found in Standard Methods for the Examination of Water and Sewage(2).

Limitations of B.O.D. Test. The B.O.D. test, if used correctly, can be used successfully for forecasting the effect of the effluent on the deoxygenation of the stream; however, this is only true within certain inherent limitations which arise from the low solubility of oxygen in water and the consequent extreme dilutions necessary to maintain dissolved oxygen(8). Ruchhoft(19) says that the factors which may be expected to effect the B.O.D. test include:

1. The number of complexity of microflora and microfauna.
2. The concentration and complexity of the organic constituents in the substrate.
3. The presence of necessary mineral accessory nutrients.
4. The pH and the buffering capacity of the substrate.
5. The presence of toxic or enzyme de-activating compounds.
6. The temperature.
7. The presence of dissolved oxygen or other hydrogen acceptors.

Coburn(6) modifies this by saying "the above items are critical because even if the faults are corrected for, it may not be the same as is found in the stream the waste is to be discharged into."

Lyons(10) notes that the B.O.D. test is not applicable to the evaluation of many organic wastes. He found 6 out of 17

organic compounds tested gave results that were not acceptable.

Heukelekian(9) states, "the deoxidation rate of sewage is independent of the dilution or concentration, even with the use of mineralized diluting water, fortified with nitrogen and phosphorus, the correction of the pH value and proper seeding consistent results are not obtained."

Effect of Dilution on B.O.D. The B.O.D. test by dilution is indicative of the relative intensity of the pollution and is not a test of high precision(13). Heukelekian(8) found that certain industrial wastes gave decreased B.O.D. with increase in dilutions. Phelps(16) found that increases in dilutions depresses the B.O.D. near the 8/10 power of the concentration. "B.O.D. by dilution," according to Burtle(4), "is distinctly an imperial determination which measures only the oxygen taken up from a diluting water in a certain length of time under a particular set of conditions for incubation."

Coburn(7) best summarizes the B.O.D. test by saying "the B.O.D. determination is only a measuring stick used in determining the effect of sewage or waste on a stream in a study of a pollution or abatement problem. It, the B.O.D. test, is a tool, not a precision instrument."

Diluting Water. The diluting water was made according to procedure outlined in Standard Methods for the Examination of Water and Sewage(3).

Standardization of Sodium Thiosulfate. A sodium

thiosulfate factor used in B.O.D. calculations was determined by the method recommended by Moggio and Burland(11).

#### Gas Analysis

In the gas analysis the following equipment was used.

Orsat Gas Apparatus. The use of the Orsat apparatus was conducted as set forth in Technical Service Department Bulletin No. 10-605, Fisher Scientific Company, Pittsburgh, Pennsylvania.

Tutweiler Gas Apparatus. Tutweiler gas apparatus was used as set forth in Bulletin No. 10-988, Technical Service Department, Fisher Scientific Company, Pittsburgh, Pa.

#### Preliminary Investigation

At the beginning of the experimental work, the only information available from the work done at Oregon State was that the liquor was heated for a period of time, therefore, the preliminary cooks were conducted by holding the temperature constant and letting the pressure vary. The first cook by this method was on raw liquor, but the next cooks were with the pH of the raw liquor adjusted to a value of three. This was done to determine if the hydrogen ion concentration was a factor in the reduction of the B.O.D.

Analysis of liquor similar to S.N.W. liquor Table 4, page 48, shows that over 50% of the calculated B.O.D. of the waste was exerted by sugars, xylose and sodium acetate

reported as acetic acid. R. E. Opferkuch, Jr., Resident Engineer for the National Council, suggested that enough oxygen be added to the container before the cooks so that oxidation of xylose and sodium acetate might occur. To determine whether or not the addition of oxygen was a factor in the reduction of the B.O.D., oxygen at 100 psi gage pressure was forced into the autoclave containing raw liquor.

On the first series of tests, the gas created during the cook was released immediately after the completion of the cook, but on later series the cooked liquor was left in the presence of the gas to determine the effect of the gas on reduction of the B.O.D.

#### Principle Investigation

Soon after the start of the preliminary investigation, additional information was received from the project at Oregon State disclosing that their method of cooking was to hold the temperature constant at a selected value until the pressure became constant at which time the cook was considered complete. They, Oregon State Project, stated that the reaction involved in the destruction of the B.O.D. was instantaneous when the pressure became constant; therefore, the cook was considered complete when this occurred.

Adjusting the Raw Liquor. When dilutional series cooks were conducted, the raw liquor was diluted with distilled water. The pH of the raw liquor was adjusted for different

cooks by the addition of sulfuric acid and the oxygen adjusted by the addition of oxygen at 100 psi gage pressure when necessary.

Cooking of Raw Liquor. After the raw liquor was treated and placed in an autoclave, the autoclave was placed in a test stand and a thermocouple attached. Pressure and temperature readings were recorded every ten minutes, the pressure being read directly from the pressure gage attached to the autoclave and the temperature determined by measuring the current through the thermocouple and reading temperature from the calibrated temperature-current curve for the thermocouple.

Analysis of The Raw Liquor, Cooked Liquor and Gas Created.

When the cook was completed, the gas in the autoclave was bled off and analyzed by the use of the Orsat Gas Analyzer and Tutweiler Gas Analyzer. The cooked liquor was then removed from the autoclave and analyzed for total solids and B.O.D.

The total solids were determined by first weighing two tared evaporating dishes and then adding 10 ml. of the raw liquor to one dish and 10 ml. of cooked liquor to the other. The dishes were then placed on an electric heater and the liquor evaporated to near dryness and then placed in a 103°C oven for two hours and weighed. The difference between the weight of the two dishes times 100 gave the increase or decrease in total solids in grams per liter.

The B.O.D. of the raw and cooked liquor was determined by

setting a series of B.O.D. bottles for the raw and cooked liquor. A series consisted of five bottles, two containing diluting water, the remaining containing dilutions of 1/15000, 1/10,000 and 1/6,000 of liquor. The bottles were then placed in a 20°C incubator. After five days, the bottles were removed and the B.O.D. determined by the Sodium Azide modification of the Winkler process as reported in Standard Methods for the Examination of Water and Sewage(1).

The percent change in B.O.D. was obtained by subtracting the B.O.D. of the cook liquor from the B.O.D. of the raw liquor and dividing this figure by the B.O.D. of the raw liquor.

#### Materials

Materials used in the experimental part of this investigation to determine the B.O.D. reaction of Semichemical Neutral Sulfite waste by Heat Hydrolysis are listed in the following section.

Alkaline Potassium Iodide. Used in B.O.D. determination.

Bacteria Seed. Sewage effluent, obtained from manhole on campus of V.P.I. used as seeding agent in diluting water.

Calcium Chloride. Used as nutrient salt in diluting water.

Diluting Water. Distilled water with nutrients added.

Used in B.O.D. determination test.

Ferric Chloride. Used as nutrient salt in diluting water.

Gasket Material. Palmetto Super Heat No. 2900,

manufactured by The Greene Tweed & Company, North Wales, Pennsylvania.

Iodine Solution. 0.0236 Normal solution used in determination of  $H_2S$  content in gas created by cooking.

Sodium Hydroxide. 2 Normal used in preparation of Phosphate buffer stock solution which is used as nutrient salt in dilution water.

Liquor. Semichemical Neutral Sulfite Mixed Hardwoods waste, obtained from the Mead Corporation, Lynchburg, Virginia.

Manganese Sulfate. Used in B.O.D. determination test.

Phosphate Buffer Solution. Used as a nutrient salt in diluting water.

Potassium Acid Phosphate. Used as a nutrient salt in diluting water.

Sodium Azide. Used in B.O.D. determination.

Sodium Thiosulfate. Used in B.O.D. determination.

Sulfuric Acid, Concentrated. Used in B.O.D. determination and in adjusting pH of raw liquor when desired.

Starch Indicator. Used in Tutweiler Gas Analyzer and in determination of B.O.D.

#### Apparatus

The apparatus used in the experimental part of this investigation to determine the B.O.D. reduction of Semichemical Neutral Sulfite waste by Heat Hydrolysis are listed in the

following section. (Schematicly shown in Figure 1, page 24).

Autoclave. 1183 ml. volume, built by the Mead Corporation, Lynchburg, Virginia.

Batteries. Ever Ready, No. 6. Used to operate potentiometer.

Galvanometer. Catalog number 570-201. Made by General Motors Laboratories. Used in recording heat of autoclave.

Incubator. 20°C converted refrigerator. Used in determination of B.O.D.

Oven. Drying 103°C. Used in total solid determination.

Oxygen. Airco Oxygen, 99.5% pure. Used when oxygen was added to autoclave.

Pressure Gage. Zero to eight hundred psi gaged manufactured by Champion Gage Company. The gage was attached to autoclave for indicating pressure of gas when cook was being made.

Potentiometer. Type S, manufactured by Fisher Scientific Company. Used in recording heat of autoclave.

pH Meter. Beckman Model M. 115 volts, 50-60 cycle, glass electrodes, manufactured by National Technical Laboratories, South Pasadena, California.

Thermometer. 10°C to 110°C, Nitrogen filled. Made by Fisher Scientific Company, Pittsburgh, Pennsylvania.

#### Data and Results

The data obtained in the experimental part of this



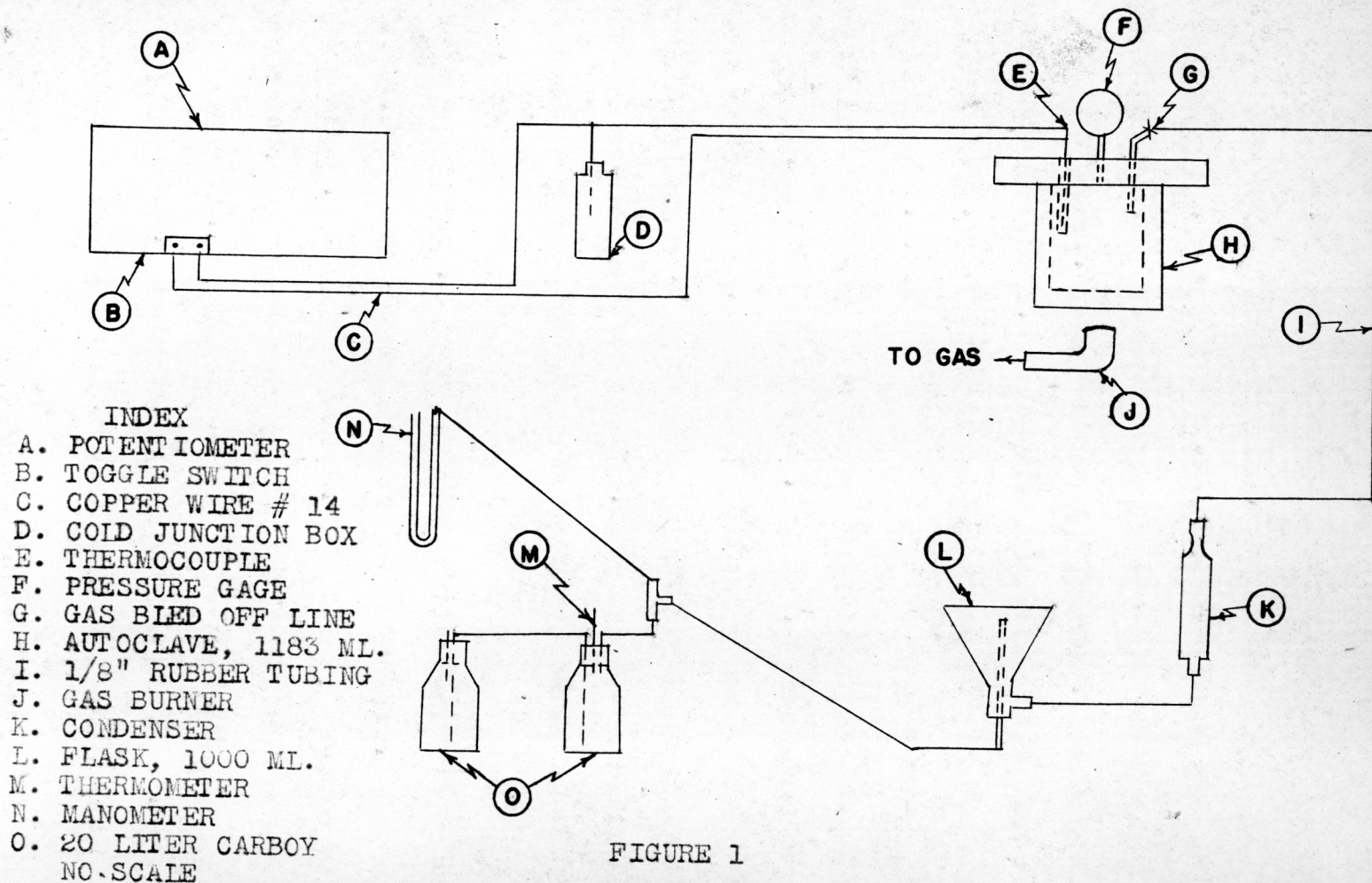


FIGURE 1

EQUIPMENT USED IN THE EXPERIMENTAL PART OF INVESTIGATION  
 OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE B.O.D. REDUCTION  
 BY HEAT HYDROLYSIS

investigation is presented in Tables 1, 2 and 3, pages 26, 27, 28, 29, 30, and 31. Column I, shows the four series of cooks conducted, and the amount of sample used in each cook. The pH of the raw and cooked liquor is shown in columns II and III, but it should be noted that the pH of the raw liquor in the Acid series is after sulfuric acid was added. Column IV indicates the time of the cook in minutes, while column V and VI show the highest pressure (in psi) and the highest temperature (in °F) during the cook. The amount of oxygen added to the autoclave before a cook was conducted is listed in column VII. Column VIII and IX give the 5-day 20°C B.O.D. of the raw and cooked liquor and the percent change in the B.O.D. is shown in column X. The amount of total solids reported in grams per liter for the raw liquor is shown in column XI, for the cooked liquor, column XII, and the change in total solids in column XIII. The gas volume created during the cook and the amount of CO<sub>2</sub>, CO, O<sub>2</sub> and H<sub>2</sub>S in the gas is presented in columns XIV, XV, XVI, XVII and XVIII respectively.

TABLE 1

DATA OBTAINED FROM DETERMINATIONS OF B.O.D. REDUCTION OF  
S.N.W. LIQUOR BY HEAT HYDROLYSIS

I Sample	II pH Raw	III pH Cook	IV Length of Cook (min)	V Highest P (psi)	VI Highest T (°F)	VII Oxygen Added (gms)
<b>Neutral</b>						
(1) 700	8.3	8.6	167	359	340	0
(2) 700	8.4	8.15	210	369	450	0
(3) 700	8.5	7.4	210	359	470	0
(4) 700	8.3	8.6	215	385	410	0
(5) 700	7.9	8.45	85	411	640	0
(6) 465	8.0	8.8	100	398	580	0
Dilution 1:1						
(7) 465	7.65	8.3	170	372	570	0
Dilution 2:1						
(8) 465	7.98	6.4	165	382	580	0
<b>Acid</b>						
(9) 700	3.15	5.4	220	409	440	0
(10) 700	3.05	5.5	220	420	424	0
(11) 700	3.15	5.35	235	382	410	0
(12) 700	3.15	5.01	205	352	430	0
(13) 700	3.1	4.7	200	439	540	0
(14) 700	3.15	5.05	205	384	440	0
(15) 700	3.15	4.7	60	385	400	0
(16) 465	2.98	5.1	180	405	590	0
Dilution 1:1						
(17) 465	3.0	4.7	145	392	580	0
Dilution 2:1						
(18) 465	2.8	4.5	135	373	595	0
<b>Neutral-Oxygen Added</b>						
(19) 700	8.5	8.3	60	341	410	5.1

TABLE 1 (continued)

DATA OBTAINED FROM DETERMINATIONS OF B.O.D. REDUCTION OF  
S.N.W. LIQUOR BY HEAT HYDROLYSIS

I Sample	II pH Raw	III pH Cook	IV Length of Cook(min.)	V Highest P (psi)	VI Highest T (°F)	VII Oxygen Added (gms)
<b>Neutral-Oxygen Added</b>						
(20) 700	8.5	6.45	28	416	400	5.1
(21) 700	8.35	8.05	42	364	400	5.1
(22) 465	8.2	7.25	130	400	490	7.4
Dilution 1:1						
(23) 465	7.65	7.8	115	375	610	7.4
Dilution 2:1						
(24) 465	7.9	5.19	105	428	570	7.4
<b>Acid-Oxygen Added</b>						
(25) 700	3.15	4.45	44	447	420	5.1
(26) 700	3.1	4.2	35	440	400	5.1
(27) 700	3.0	4.3	30	451	440	5.1
(28) 465	3.1	4.91	150	420	560	7.4
Dilution 1:1						
(29) 465	2.95	4.4	60	456	600	7.4
Dilution 2:1						
(30) 465	2.98	4.05	90	392	425	7.4

TABLE 2

DATA OBTAINED FROM DETERMINATIONS OF B.O.D. REDUCTIONS  
OF S.N.W. LIQUOR BY HEAT HYDROLYSIS

I Sample	VIII Raw-5Day 20°C B.O.D.	IX Cook-5Day 20°C B.O.D.	X % Change in B.O.D.	XI Raw- Total Solids (g/ltr)	XII Cook-Total Solids (g/ltr)	XIII %Change in Total Solids (g/ltr)
Neutral						
(1) 700	52,939	28,653	45.87	220.08	185.97	34.11
(2) 700	29,750	17,268	41.96	-	-	-
(3) 700	27,917	21,806	39.80	157.33	185.97	I 28.64
(4) 700	33,875	28,280	16.52	219.09	208.77	10.32
(5) 700	38,125	25,250	33.77	222.46	147.05	74.8
(6) 465	64,750	48,433	25.23	220.43	206.29	14.14
Dilution 1:1						
(7) 464	25,454	21,951	13.76	-	-	-
Dilution 2:1						
(8) 465	14,789	13,260	10.34	68.86	65.17	3.69
Acid						
(9) 700	42,037	26,667	36.56	-	-	-
(10) 700	37,333	30,333	18.75	-	-	-
(11) 700	35,562	23,196	34.77	183.67	126.64	57.03
(12) 700	43,750	27,361	37.46	133.87	116.36	17.51
(13) 700	34,700	27,400	21.04	139.00	101.57	37.43
(14) 700	41,700	27,000	35.25	157.41	117.55	39.86
(15) 700	29,722	17,268	41.90	-	-	-
(16) 465	64,590	42,200	34.66	235.58	136.17	99.41
Dilution 1:1						
(17) 464	20,303	16,873	16.89	103.31	85.90	17.41
Dilution 2:1						
(18) 465	16,993	10,310	39.33	69.89	68.60	1.29

TABLE 2 (continued)

DATA OBTAINED FROM DETERMINATIONS OF B.O.D. REDUCTIONS  
OF S.N.W. LIQUOR BY HEAT HYDROLYSIS

I Sample	VIII Raw-5Day 20°C B.O.D.	IX Cook-5Day 20°C B.O.D.	X % Change in B.O.D.	XI Raw-Total Solids (g/ltr)	XII Cook-Total Solids (g/ltr)	XIII % Change in Total Solids (g/ltr)
Neutral-Oxygen Added						
(19) 700	30,015	36,773	I 18.38	149.3	184.5	I 35.20
(20) 700	27,712	31,755	I 12.73	182.19	143.21	38.98
(21) 700	27,869	31,282	I 10.91	154.12	141.91	12.20
(22) 465	58,718	43,691	25.59	251.48	167.92	83.56
Dilution 1:1						
(23) 464	25,360	22,900	9.70	102.59	85.14	17.45
Dilution 2:1						
(24) 465	15,850	8,710	45.05	71.02	69.75	1.27
Acid-Oxygen Added						
(25) 700	30,000	21,364	28.79	154.67	128.98	25.69
(26) 700	37,236	26,636	28.47	147.42	122.07	25.35
(27) 700	40,000	30,970	22.57	270.49	196.66	73.83
(28) 465	39,825	25,025	37.16	210.05	121.92	90.13
Dilution 1:1						
(29) 464	23,059	15,448	33.01	104.5	104.6	I 0.1
Dilution 2:1						
(30) 465	13,824	5,506	60.17	72.36	51.50	20.86

TABLE 3

DATA OBTAINED FROM DETERMINATIONS OF B.O.D. REDUCTIONS  
OF S.N.W. LIQUOR BY HEAT HYDROLYSIS

I Sample	XIV Volume Gas Created at S.T.P.(ltr)	XV % CO <sub>2</sub> In Gas	XVI % CO In Gas	XVII % O <sub>2</sub> In Gas	XVIII % H <sub>2</sub> S In Gas
Neutral					
(1) 700	-	-	-	-	-
(2) 700	-	-	-	-	-
(3) 700	-	-	-	-	-
(4) 700	-	-	-	-	-
(5) 700	-	-	-	-	-
(6) 465	-	48.6	7.2	0	1.23 x 10 <sup>-4</sup>
Dilution 1:1					
(7) 464	4.6	35.4	2.5	0.6	0.35 x 10 <sup>-4</sup>
Dilution 2:1					
(8) 465	4.5	38.8	1.73	1	1.56 x 10 <sup>-4</sup>
Acid					
(9) 700	-	-	-	-	-
(10) 700	-	-	-	-	-
(11) 700	-	-	-	-	-
(12) 700	-	-	-	-	-
(13) 700	-	-	-	-	-
(14) 700	-	-	-	-	-
(15) 700	-	-	-	-	-
(16) 465	7.8	51.8	2.2	0	0.3 x 10 <sup>-4</sup>
Dilution 1:1					
(17) 464	2.5	36.3	2.0	0.2	0.91 x 10 <sup>-4</sup>
Dilution 2:1					
(18) 465	6.9	43.9	2.2	0	3.14 x 10 <sup>-4</sup>

TABLE 3 (continued)

DATA OBTAINED FROM DETERMINATIONS OF B.O.D. REDUCTIONS  
OF S.N.W. LIQUOR BY HEAT HYDROLYSIS

I Sample	XIV Volume Gas Created at S.T.P.(ltr)	XV % CO <sub>2</sub> In Gas	XVI % CO In Gas	XVII % O <sub>2</sub> In Gas	XVIII % H <sub>2</sub> S In Gas
Neutral-Oxygen Added					
(19) 700	-	-	-	-	-
(20) 700	-	32.1	21.1	2.07	-
(21) 700	-	69	21	6	-
(22) 465	2.3	39.4	1.0	2.0	0.15 x 10 <sup>-4</sup>
Dilution 1:1					
(23) 464	2.3	38.8	4.0	0.6	0.91 x 10 <sup>-4</sup>
Dilution 2:1					
(24) 465	2.5	35.8	0	1.0	3.22 x 10 <sup>-4</sup>
Acid-Oxygen Added					
(25) 700	-	42.2	20.2	0	-
(26) 700	-	39	16.8	2	-
(27) 700	-	17	18	2	-
(28) 465	3.3	52	4.03	0	0.15 x 10 <sup>-4</sup>
Dilution 1:1					
(29) 464	5.2	45.7	5.8	3.0	1.29 x 10 <sup>-4</sup>
Dilution 2:1					
(30) 465	2.0	-	-	-	-



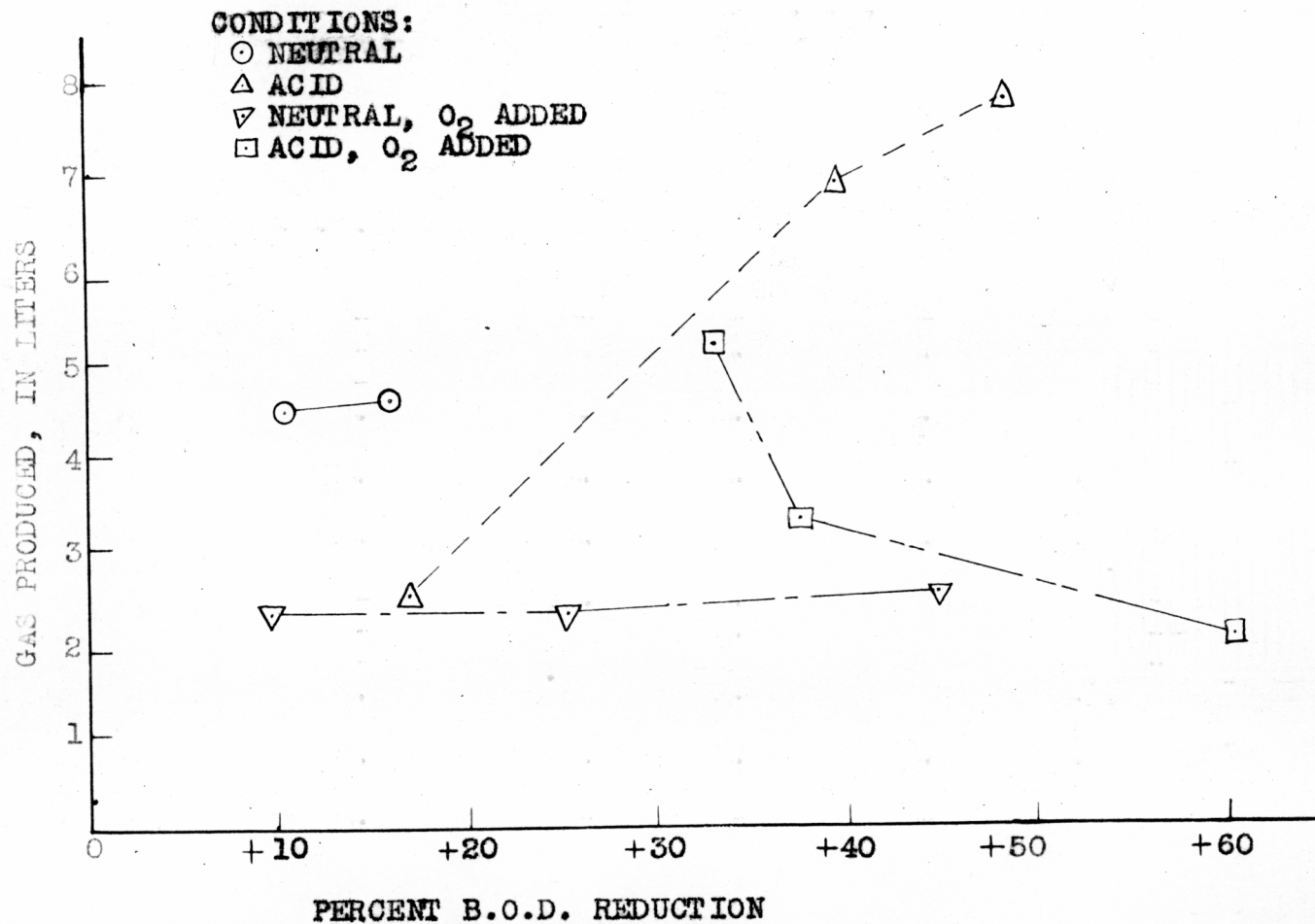


FIGURE 2. RELATIONSHIP OF GAS PRODUCED TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

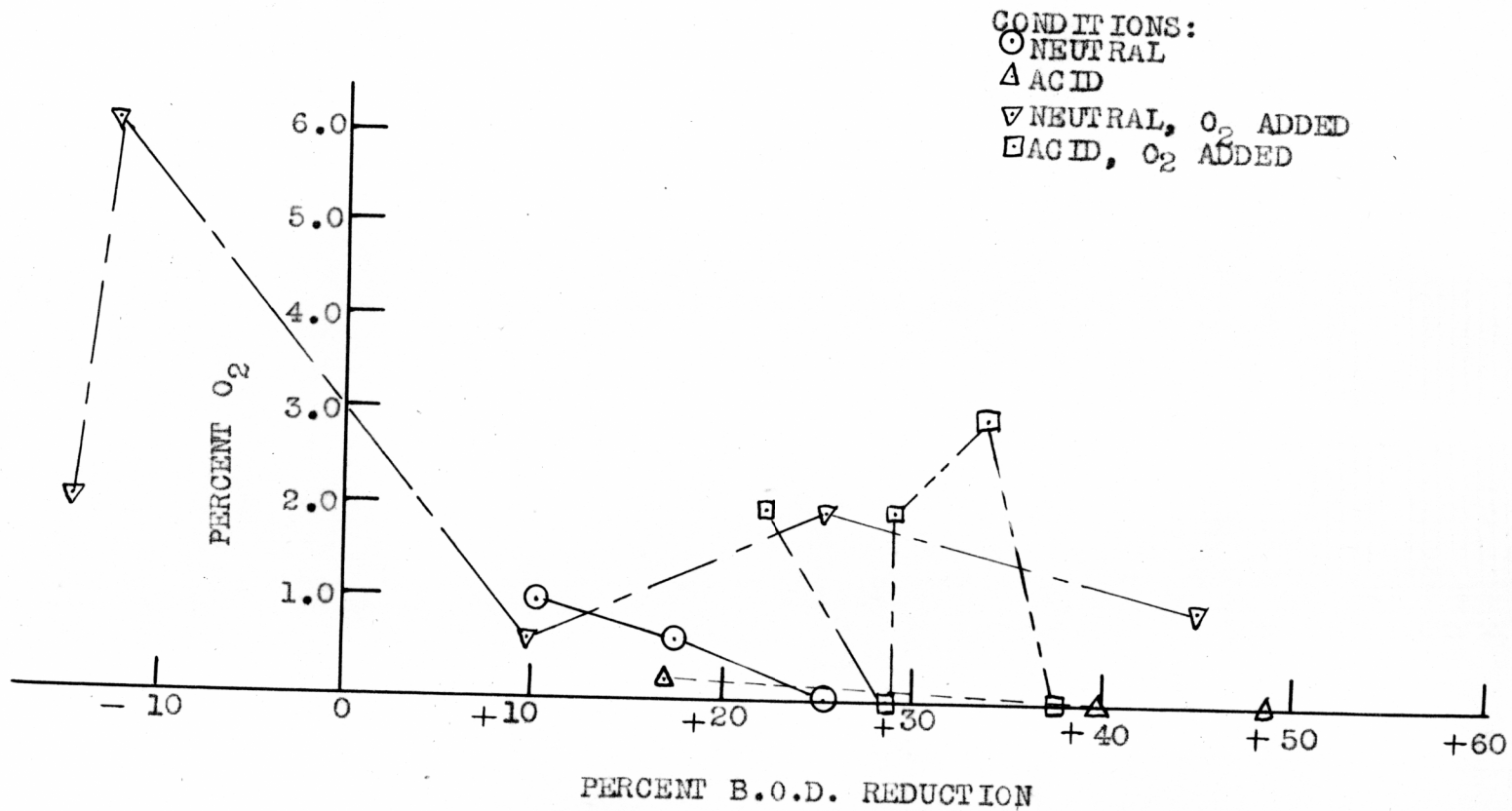


FIGURE 3. RELATIONSHIP OF O<sub>2</sub> PRODUCED TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

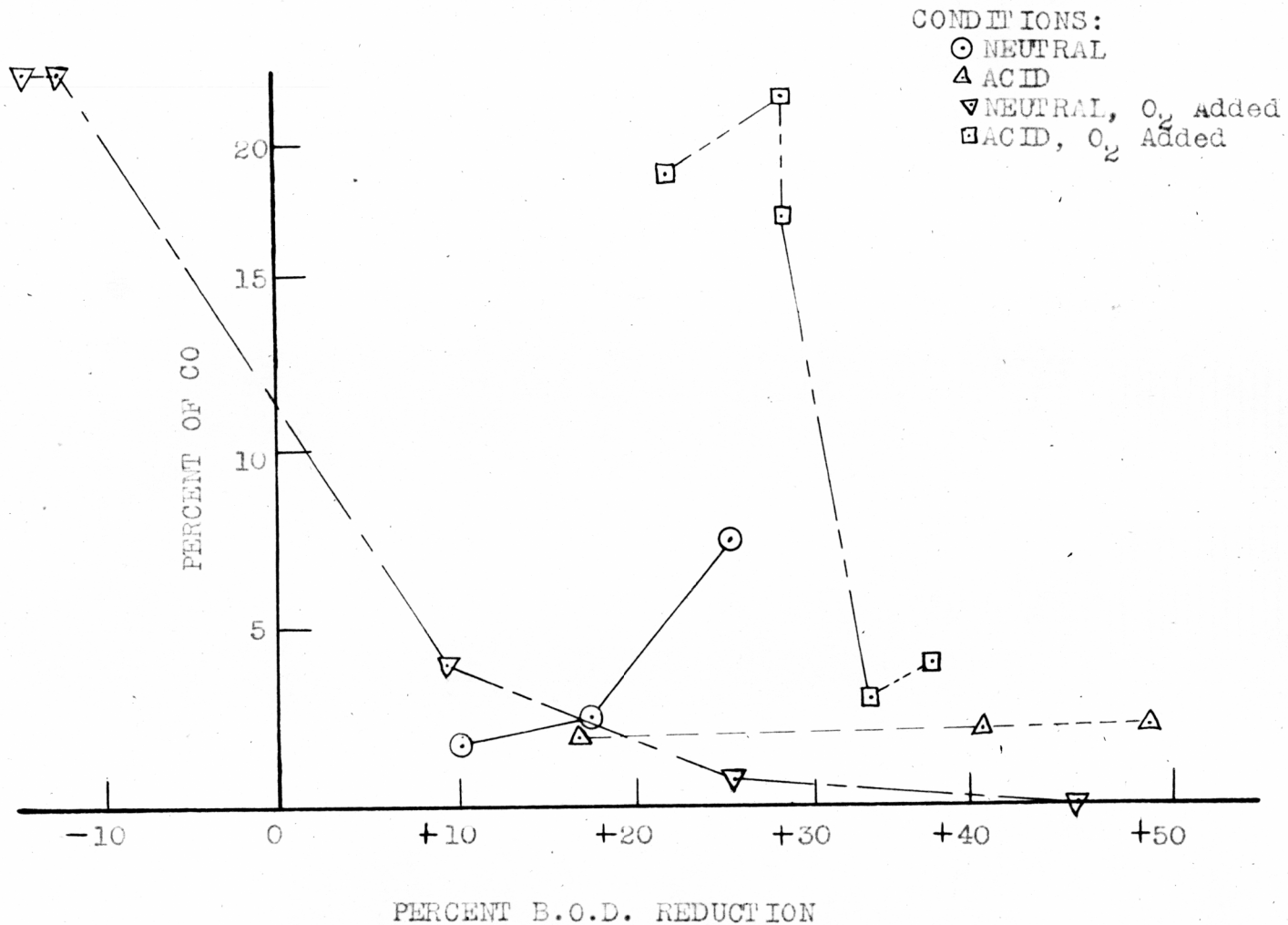


FIGURE 4. RELATIONSHIP OF CARBON MONOXIDE PRODUCED TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

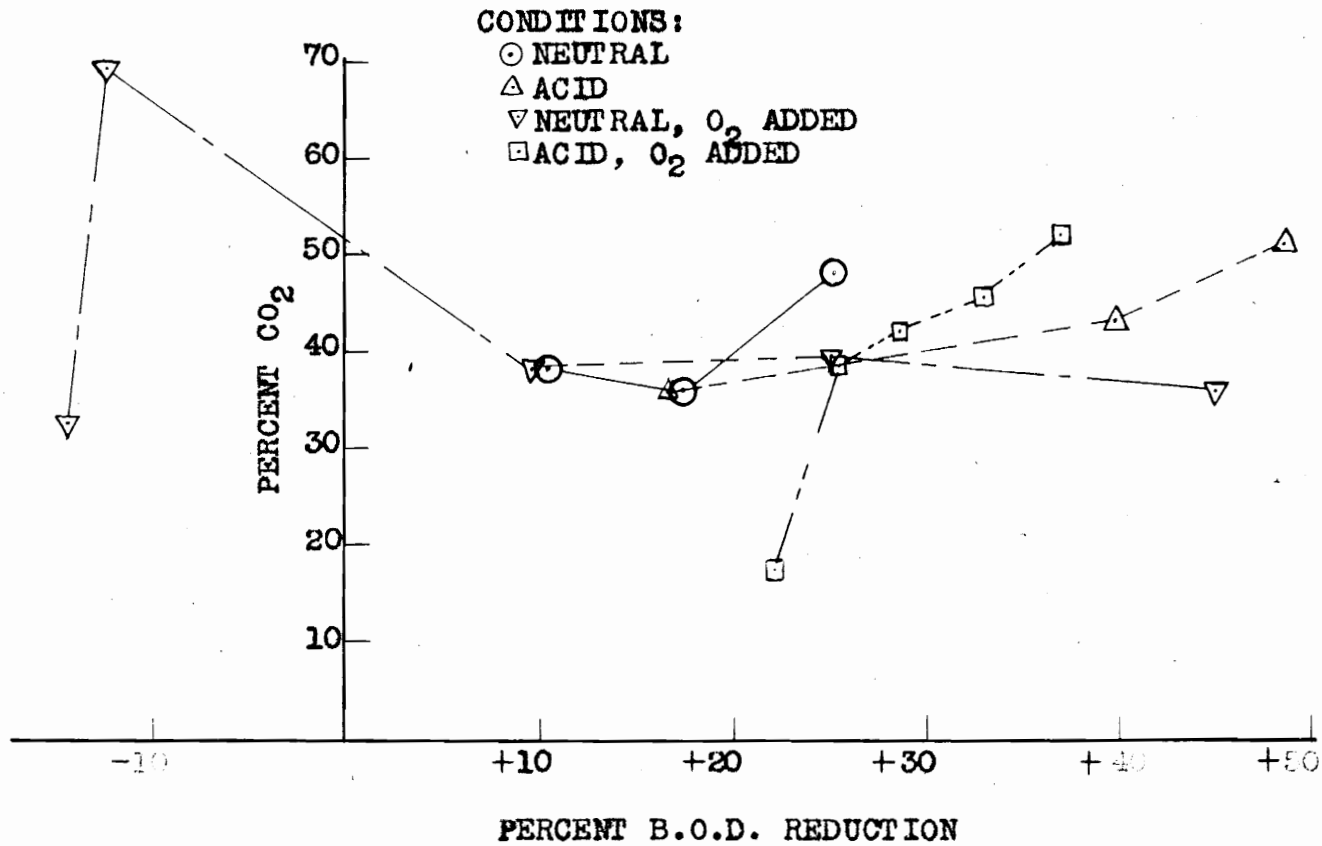


FIGURE 5. RELATIONSHIP OF CO<sub>2</sub> PRODUCED TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

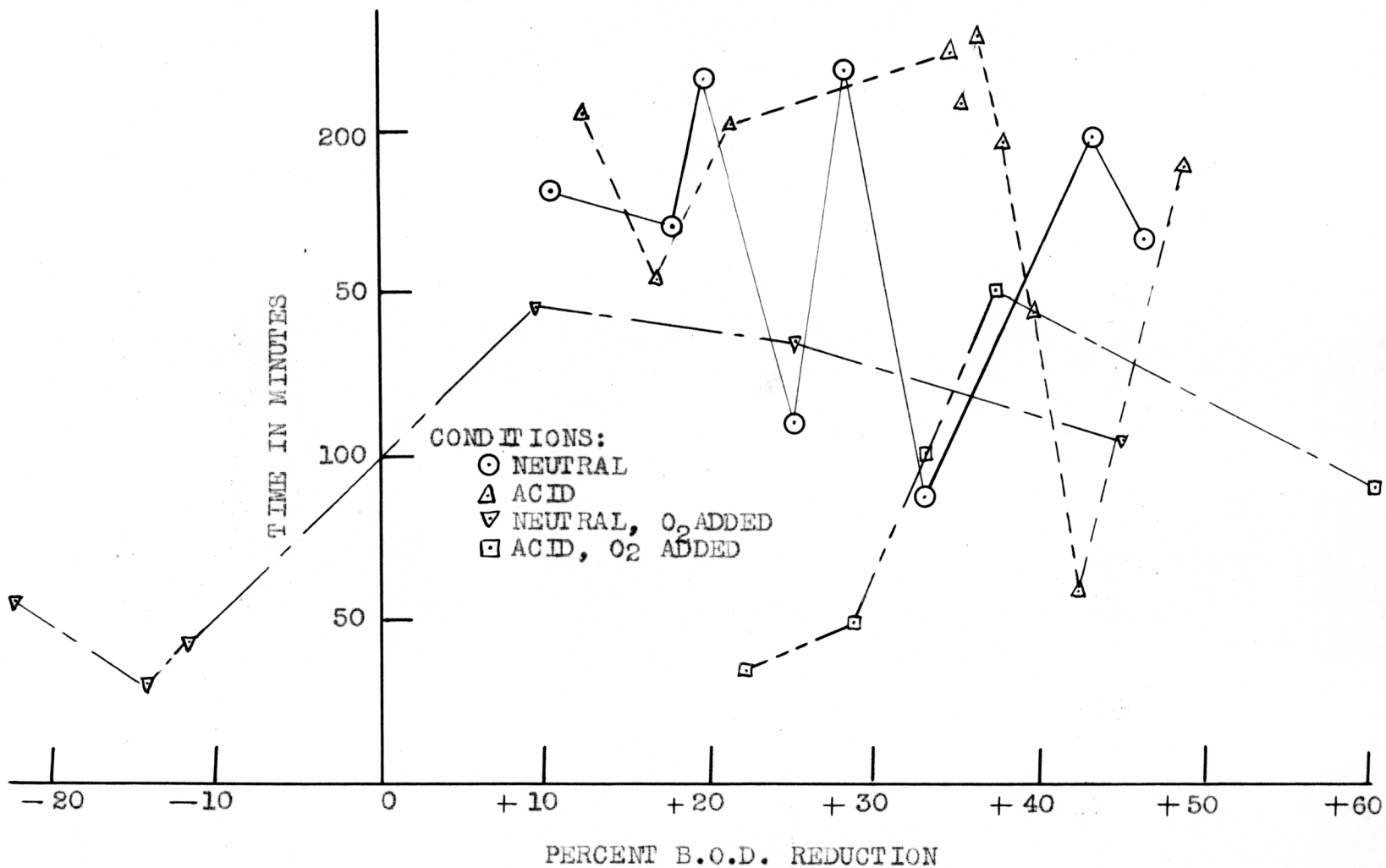


FIGURE 6. RELATIONSHIP OF TIME OF COOK TO B.O.D. REDUCTION IN HEAT HYDROLYSIS IN SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

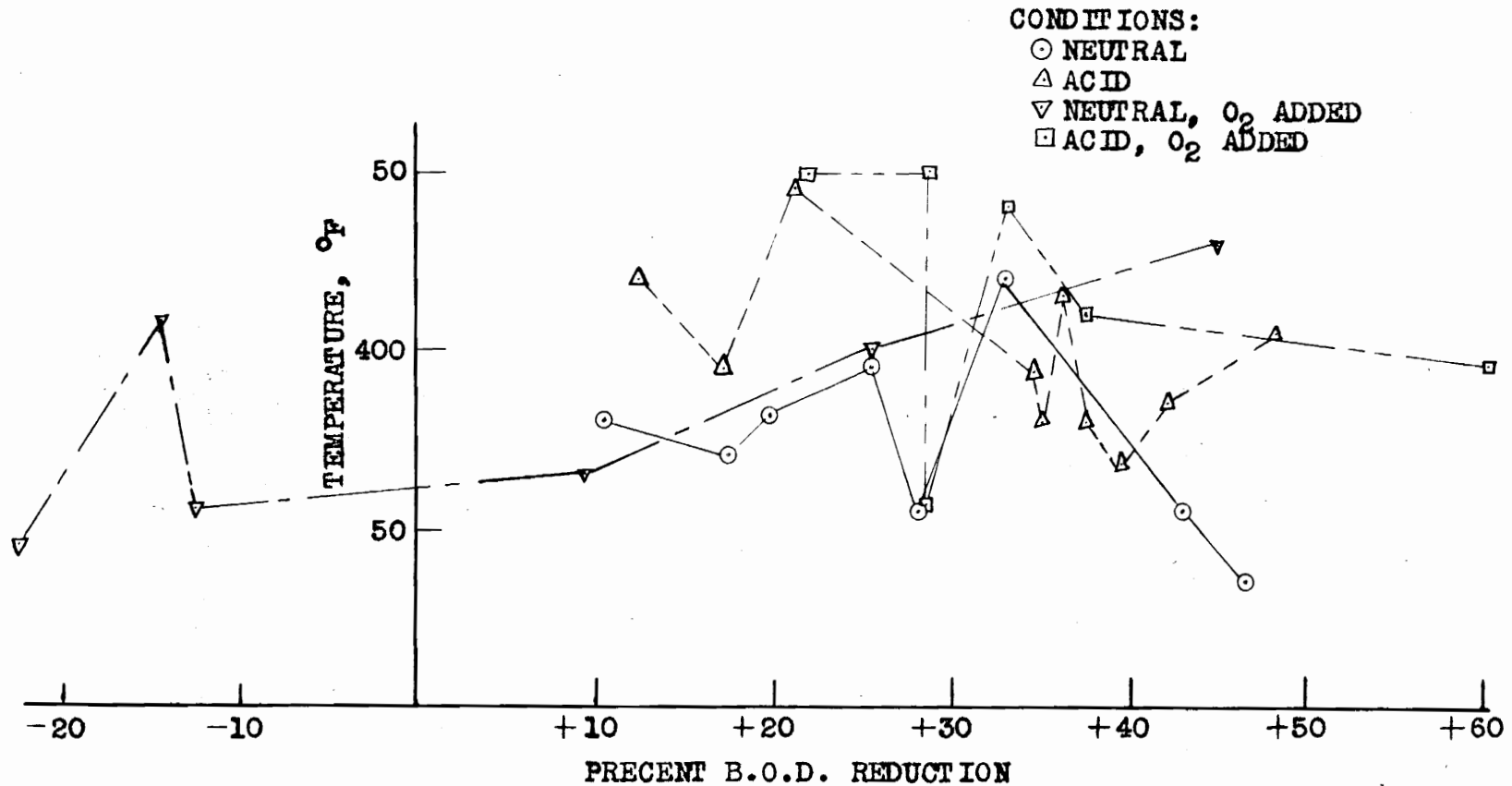


FIGURE 7. RELATIONSHIP OF HIGHEST TEMPERATURE OBTAINED TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

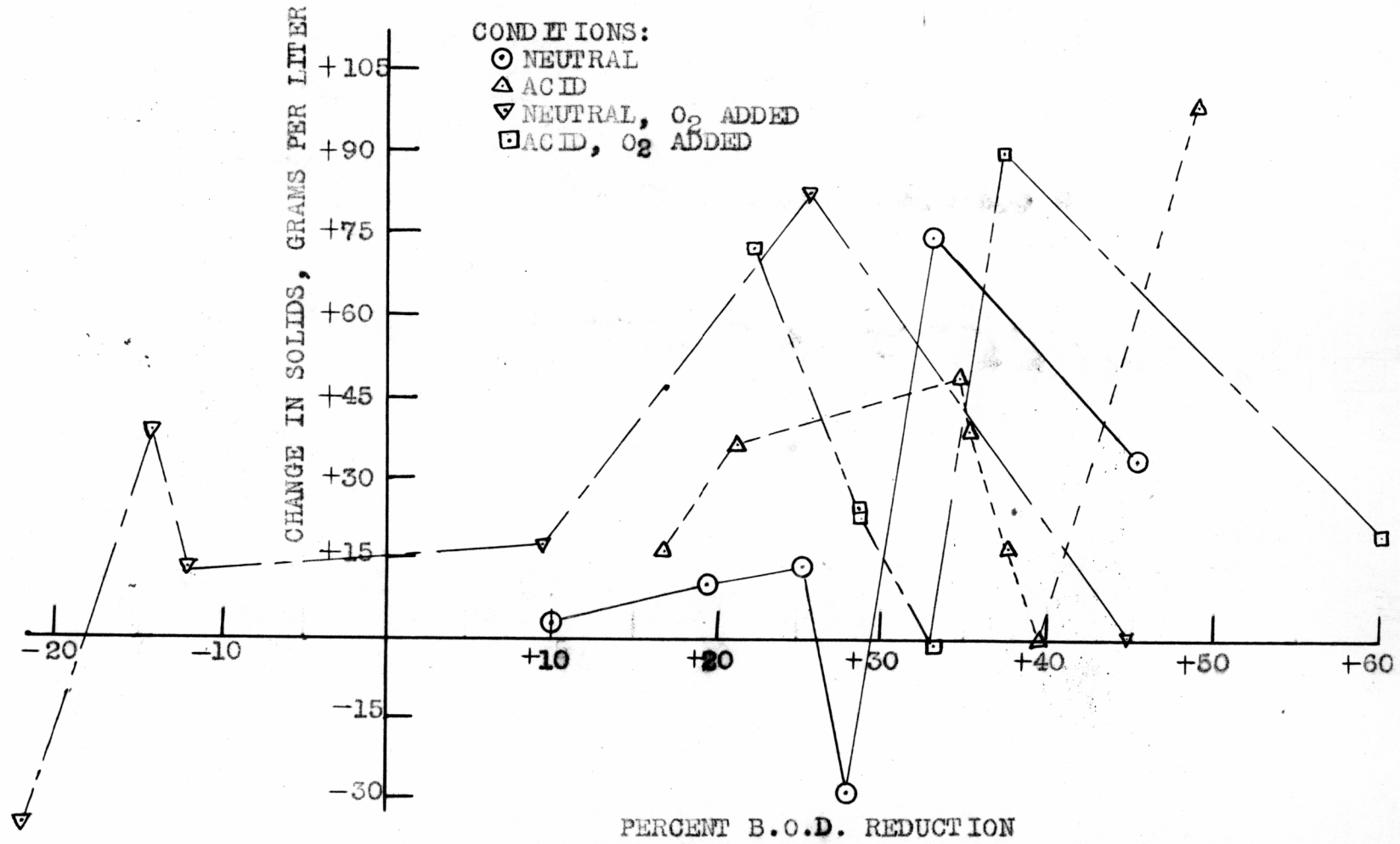


FIGURE 8. RELATIONSHIP OF CHANGE IN SOLIDS TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

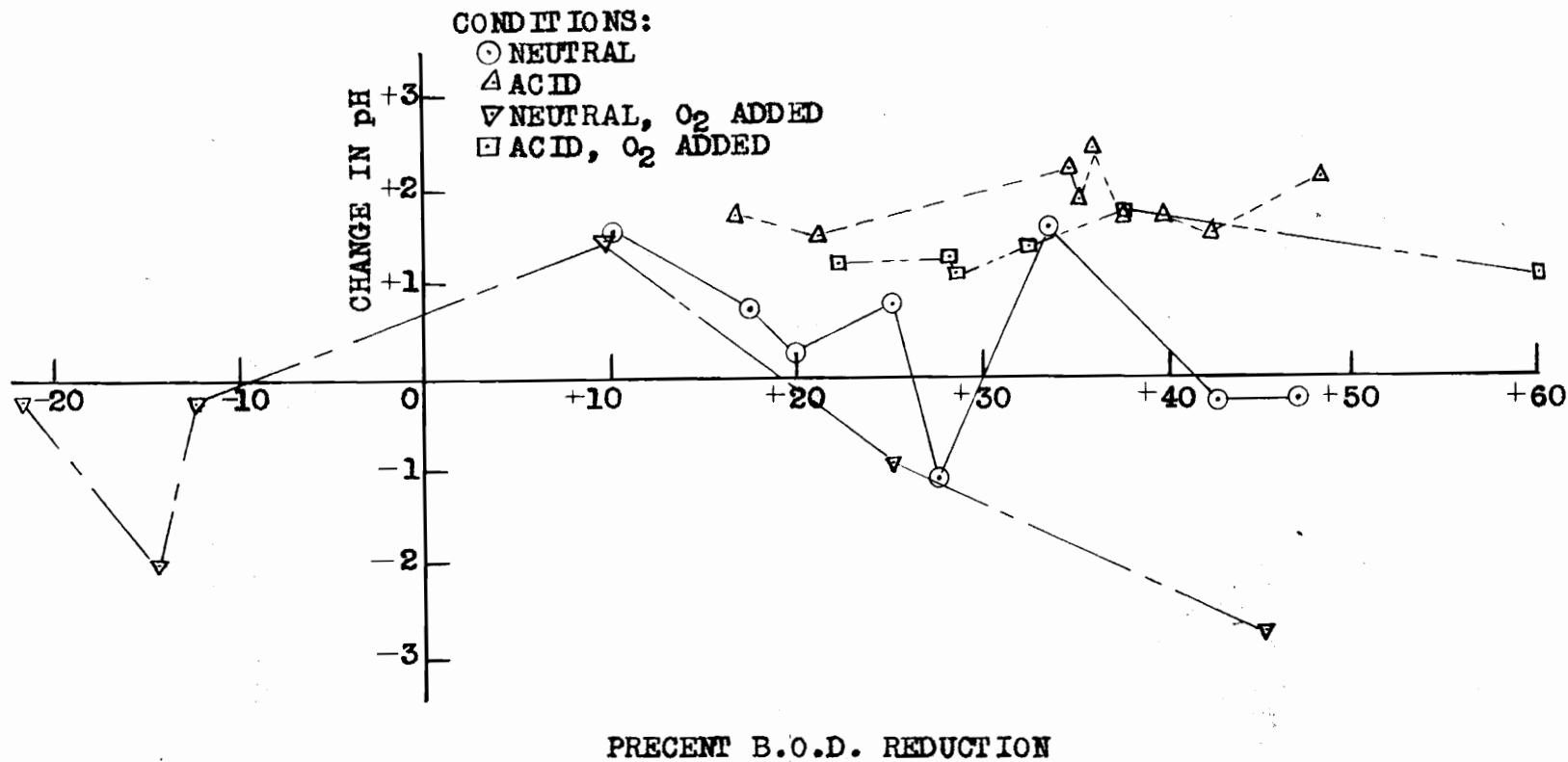


FIGURE 9. RELATIONSHIP OF CHANGE IN pH TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.



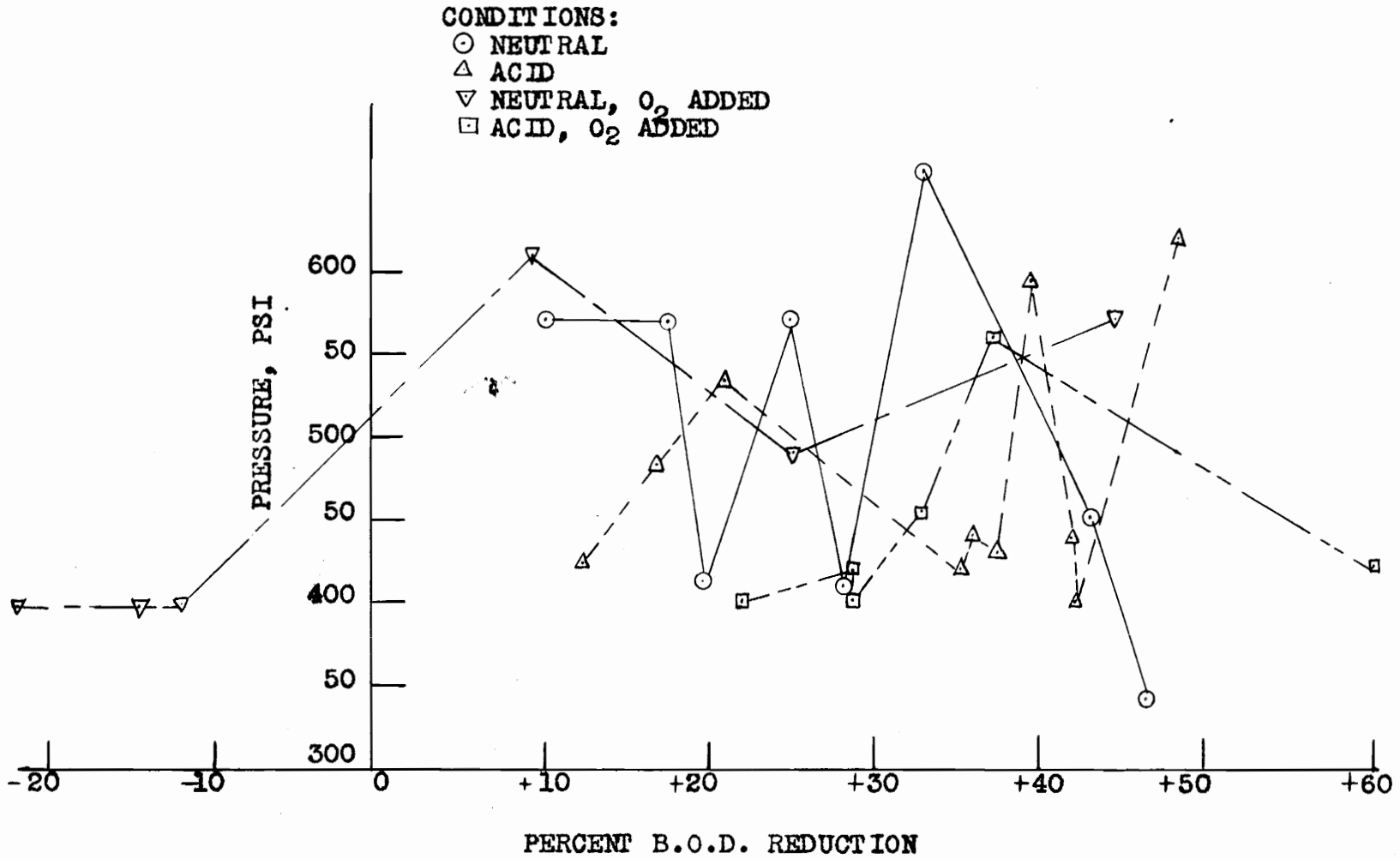


FIGURE 10. RELATIONSHIP OF HIGHEST PRESSURE OBTAINED TO B.O.D. REDUCTION IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

Sample Calculations

The methods used in calculating the gas volume and the grams of oxygen added are outlined in the following section.

Gas Volume. The gas volumes were calculated in the following manner:

$V$  = Volume of Autoclave.

$V_w$  = Volume of water in carboy displaced by gas.

$V_c$  = Volume of cooked liquor in suction flask  
after gas bled off.

$V_a$  = Volume of cooked liquor in autoclave after  
gas bled off.

$V_f$  = Volume of suction flask.

$T$  = Room temperature.

$T_s$  = Temperature of gas in autoclave when gas bled  
off.

$T_c$  = Temperature of gas in carboy.

$P_c$  = Pressure of gas in carboy.

$P$  = Room pressure.

$S$  = Amount of sample used.

$$\frac{(V-V_c) \times P_c}{T_s} = V_q = \text{Ml. of gas in autoclave at S.T.P.}$$

$$\frac{(V_f-V_c) + V_w \times P_c}{T_c} = V_r = \text{Ml. of gas in carboy and suction flask at S.T.P.}$$

$$\frac{(V-S) \times P}{T} = V_s = \text{Ml. of gas in autoclave at start of cook at S.T.P.}$$

Example:

$$V = 1183 \text{ ml.}$$

$$V_w = 7210 \text{ ml.}$$

$$V_c = 315 \text{ ml.}$$

$$V_a = 115 \text{ ml.}$$

$$V_f = 1000 \text{ ml.}$$

$$T = 297^\circ \text{ absol.}$$

$$T_s = 466.5^\circ \text{ absol.}$$

$$T_c = 292^\circ \text{ absol.}$$

$$P = 707.6 \text{ mm pressure.}$$

$$S = 465 \text{ ml.}$$

$$V_q = \frac{(1183 - 115) \times 1.005 \times 273}{466.5 \times 1} = 626 \text{ ml.}$$

$$V_r = \frac{(1000 - 315) + 7210 \times 1.005 \times 273}{297 \times 1} = 7360 \text{ ml.}$$

$$V_s = (1183 - 465) \times 707.6 \times 273 = 537$$

Total volume of gas at S.T.P. = 7449 ml.

Oxygen Added. The grams of oxygen added was calculated in the following way:

S = Volume of sample.

V = Volume of autoclave.

T = Room temperature.

P = Room pressure.

O = Gage pressure due to the addition of oxygen.

R = Mole of oxygen.

P<sub>a</sub> = Atmospheric pressure.

$$\frac{R \times P}{T} = O_w = \text{Grams per liter of oxygen at room conditions.}$$

$$\frac{O + P_a}{P_a} = A = \text{Pressure created due to the addition of oxygen (absolute).}$$

$$\frac{A \times (V-S) \times P_a}{P} = V_r = \text{Volume of oxygen at room conditions.}$$

$$V_r \times O_w = G = \text{Grams of oxygen added at room conditions.}$$

Calculation:

$$R = 1.43 \text{ grams per liter at S.T.P.}$$

$$O^0 = 100 \text{ pounds.}$$

$$T = 293^0 \text{ absolute.}$$

$$P = 13.6 \text{ pounds per inch square.}$$

$$P_a = 14.7 \text{ pounds per inch square.}$$

$$V = 1183 \text{ ml.}$$

$$S = 465 \text{ ml.}$$

$$O_w = 1.43$$

$$A = \frac{100 + 14.7}{14.7} = 7.8$$

$$V_r = \frac{7.8 \times (1183 - 465)}{13.6} \times 14.7 = 6.05 \text{ liters.}$$

$$G = 7.8 \times 6.05 = 7.4 \text{ grams of oxygen added at room conditions.}$$

## V DISCUSSION OF RESULTS

Reduction of B.O.D. Obtained by Heat Hydrolysis

The purpose of the investigation was to determine whether or not Heat Hydrolysis would reduce the B.O.D. content of the S.N.W. liquor to a point where the liquor could be discharged without further treatment.

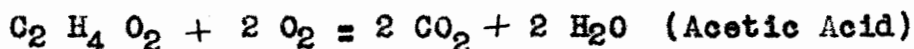
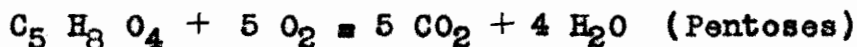
The largest B.O.D. reduction obtained during the investigation was 60.2%. This reduction was accomplished when the liquor was diluted with two parts of distilled water to one part of liquor, the pH value was at 3, and 7.4 grams of oxygen were added before the cook was made. The best reduction obtained in the other series were; 45.1%, Neutral series; 48.8%, Acid series; and 44.8%, Acid-Oxygen added series. Since no analytical determinations were made on the cooked liquor it is not known which of the constituents present, chemically degraded, resulted in the B.O.D. reduction obtained in the cooked liquor; however, if the author may be permitted to speculate the following explanation might be made: the B.O.D. reduction obtained was due to the chemical degradation of pentoses and sodium acetate in the S.N.W. liquor.

Two items should be considered in comparing the chemical degradation of pentoses and sodium acetate with B.O.D. reduction obtained, namely, the calculated B.O.D. for

pentoses (xylose) and sodium acetate, reported as acetic acid, and the average B.O.D. reduction obtained in all series of cooks.

The theoretical B.O.D. due to pentoses (xylose) and sodium acetate, reported as acetic acid, was calculated as follows:

It was assumed that complete metabolism is represented by the overall equations:



It was assumed that the normal B.O.D. reaction rate applied, so that the 5-day 20°C B.O.D. equals 70 percent of the ultimate B.O.D. as follows(15):

F = Fraction of compound consumed.

k = Reaction velocity constant.

t = Time in days.

$$\text{Log } F = kt. \quad k = 0.1 \text{ with times in days.}$$

$$\text{Log } F = 0.1 \quad F = 0.7$$

$$\frac{160 \times 0.7}{132} = 0.85 \text{ grams oxygen per gram pentoses.}$$

$$\frac{64 \times 0.7}{60} = 0.75 \text{ grams oxygen per gram acetic acid.}$$

Analysis of the raw waste showed it contained 20.12 grams of sodium acetate reported as acetic acid, 6.77 grams of pentoses reported as xylose, and 53 grams per liter volatile solids(12).

The percent of the 5-day 20°C B.O.D. contributed by these substances is then:

$$\frac{20.12 \times 0.75}{53} = 28.5\% \quad (\text{Acetic Acid})$$

$$\frac{6.7 \times 0.83}{53} = 10.5\% \quad (\text{Pentoses})$$

The average B.O.D. reduction obtained in all series of cooks was 30.4 percent. This value was obtained by averaging the percent B.O.D. reductions shown in Table 2, pages 28 and 29.

When the above items, theoretical B.O.D. and B.O.D. reduction, are considered, it can be assumed that the reduction in the B.O.D. of S.N.W. liquor was due to the chemically degradation of a large percentage of the sodium acetate and pentoses present.

Research is now being conducted by the V.P.I. project to determine what chemical reactions take place in the S.N.W. liquor when subjected to Heat Hydrolysis.

Effect of pH on Reduction of B.O.D. The pH of the liquor used in the experimental work at Oregon State College was approximately 3, therefore, it was decided to determine what effect lowering the pH would have on the reduction of B.O.D. By lowering the pH of the raw liquor to a value of 3, an average increase in B.O.D. reduction of 13.4% was obtained over the B.O.D. reduction of the Neutral series of cooks.

Effect of Dilution on B.O.D. Reduction. To determine the effect of dilution on the reduction of B.O.D., two dilutions were made, one part liquor to one part distilled water, and one part liquor to two parts distilled water, and the results obtained were compared with those of the undiluted waste. In the Acid, Neutral, and Neutral-Oxygen added series of cooks, a dilution of one to one was used, and a decrease in the reduction of B.O.D. was obtained while an increase in B.O.D. reduction was obtained in the Acid-Oxygen added series cook. A dilution of two to one produced an increase in the B.O.D. reduction in Acid, Neutral-Oxygen added, Acid-Oxygen added series of cooks but a decrease in the Neutral series cook.

Effect of Addition of Oxygen in B.O.D. Reduction. It was demonstrated on waste similar to S.N.W. that over 50 percent of the calculated B.O.D. was due to pentoses and sodium acetate in the liquor (Table 4). It was believed that the addition of oxygen to the autoclave before a cook was made would aid in the destruction of these materials and a greater reduction in the B.O.D. obtained. When, in the present investigation, oxygen was added to a 700 ml. sample as in cooks number 19, 20, and 21, a 16.5 percent increase over the B.O.D. of the raw liquor was obtained; when a 465 ml. sample was used, a decrease in the B.O.D. over the raw was observed, this being 5 percent less than the average



TABLE 4

SUMMARY OF ANALYTICAL RESULTS AND CALCULATED VALUES  
 BASED THEREON FOR SEMI-CHEMICAL SODA PULPING WASTE.(a)

Component	Percentage	
	Attributed to Each Component	
	Total Solids %	Total 5-Day B.O.D. %
Pentosans	5.7	18.9
Sodium Bicarbonate	11.5	0.0
Sodium Acetate	26.1	56.7
Sodium Formate	6.8	4.2
Lignin	14.9	*
Hydrolyzable fraction of suspended solids (other than pentosans)	5.4	*
<b>Total</b>	<b>70.4</b>	<b>79.8</b>
Undentified	29.6	20.2
* Doubtful 5-day B.O.D. value. Removal of suspended solids by filtration gave no B.O.D. removal.		

(a) Unpublished 1948 Annual Report on Semi-Chemical Waste  
 by Virginia Polytechnic Institute Engineering Research  
 Foundation Inc., Blacksburg, Virginia.

reduction obtained in the Neutral series. With the addition of oxygen to the Acid series of cooks, an 8.3 percent increase in the reduction of B.O.D. was noted.

Degree of Reaction. It was believed that the degree of reaction rate might be correlated with the B.O.D. reduction; no correlation could be found in any series of cooks, as shown in Figures number 11, 12, 13, and 14. The lack of correlation may be due to experimental error in the following way: (1) measurement of gas temperature in the autoclave, and (2) pressure created by the addition of oxygen during the cook.

The location of the thermocouple well used in measuring the temperature of the gas was very near the side of the autoclave, and it is possible that the temperature obtained was not that of the gas but of the gas and radiant heat from the autoclave. This seems logical since in several cooks the calculated water vapor pressure was greater than the indicated pressure in the autoclave.

The pressure exerted by the addition of oxygen at different times was determined by making a cook using a 465 ml. Neutral sample with 9.4 grams of oxygen added. At ten minute intervals, 50 ml. of the gas was withdrawn and analyzed using an Orsat Gas Analyzer. Another determination was conducted using a 465 ml. sample with 7.4 grams of oxygen, but no heat was applied to the autoclave. The results obtained in both

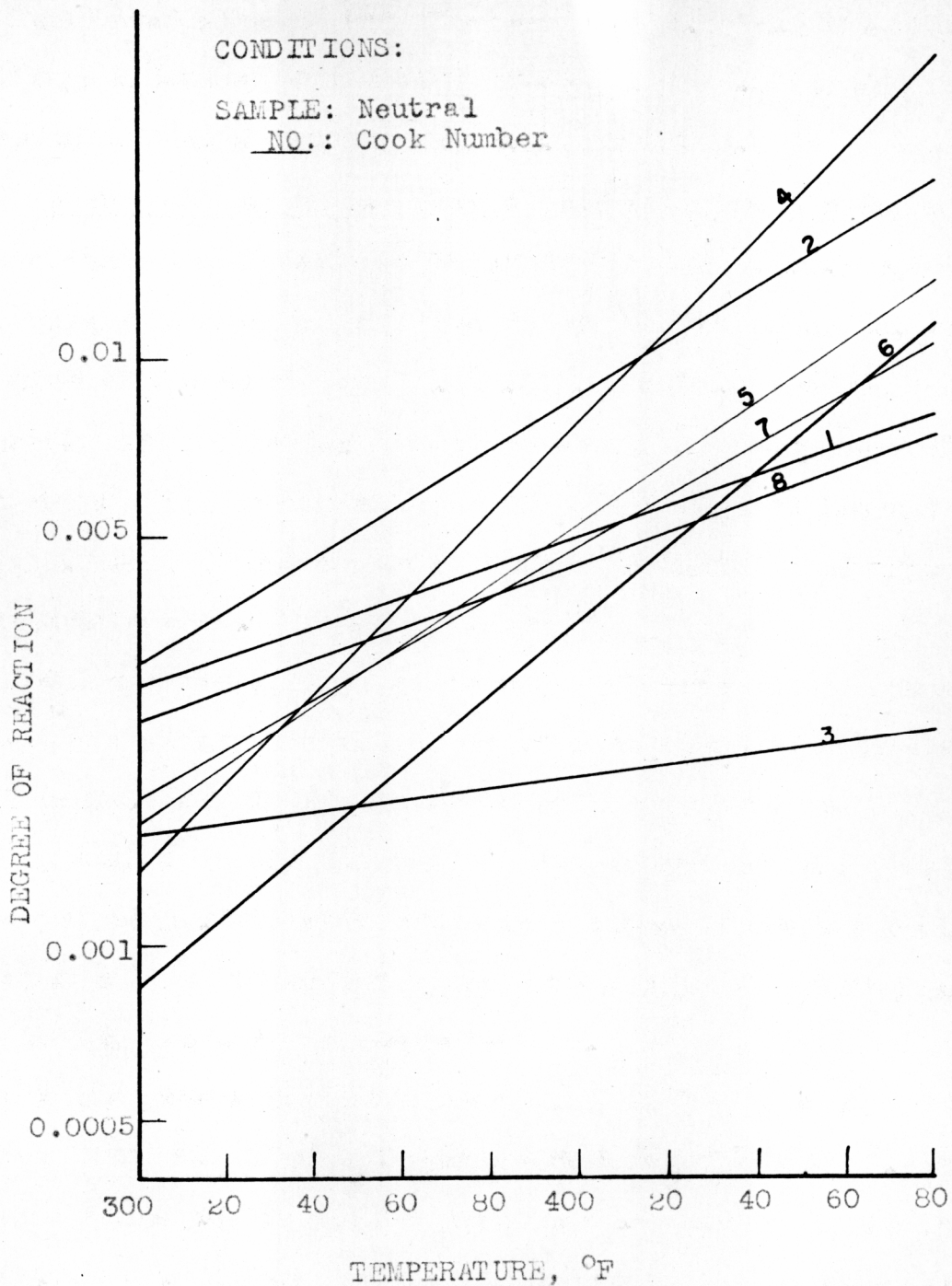


FIGURE 11. RELATIONSHIP OF DEGREE OF REACTION RATE TO TEMPERATURE IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

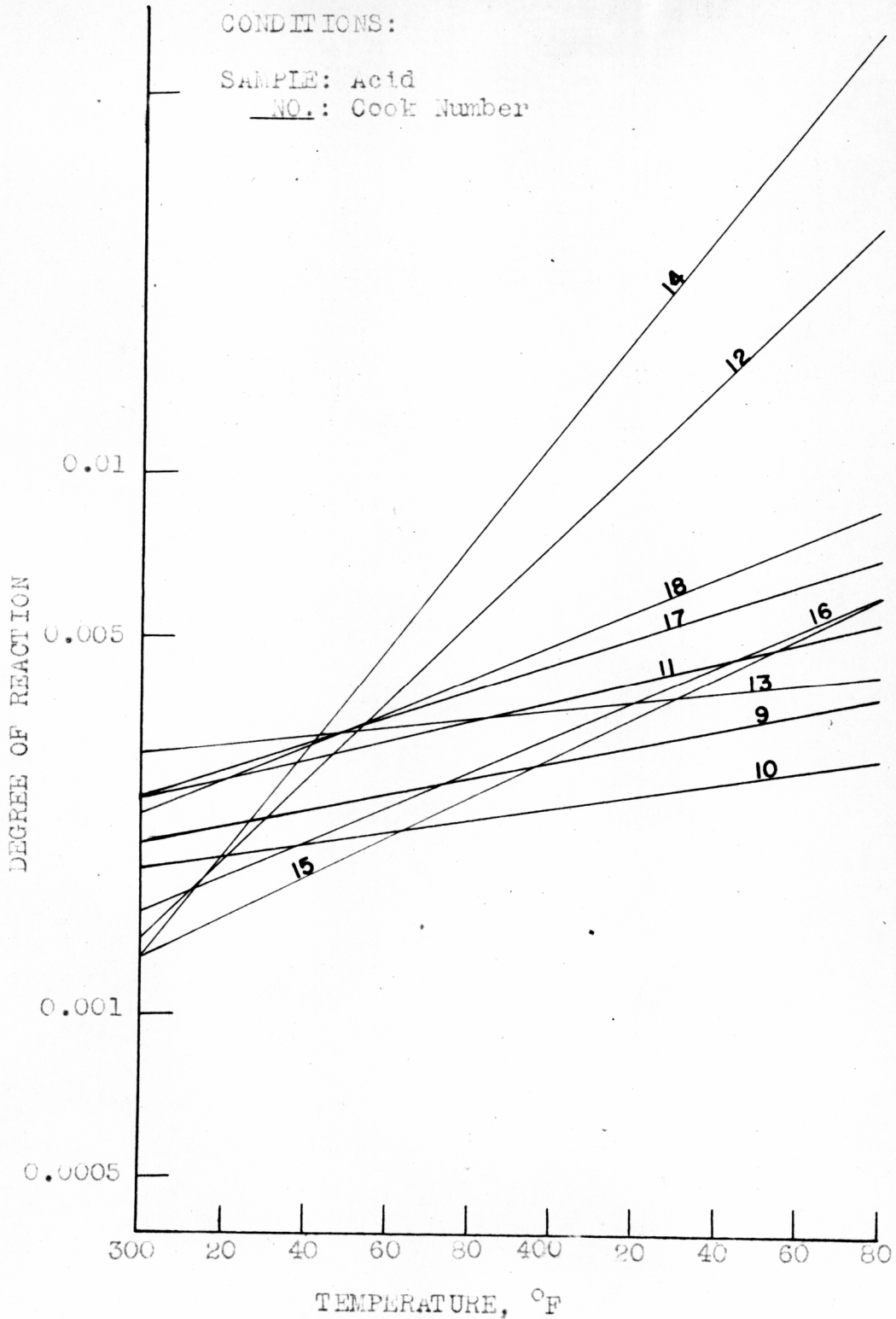


FIGURE 12. RELATIONSHIP OF DEGREE OF REACTION RATE TO TEMPERATURE IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

CONDITIONS:

SAMPLE: Neutral, O<sub>2</sub> Added  
 NO.: Cook Number

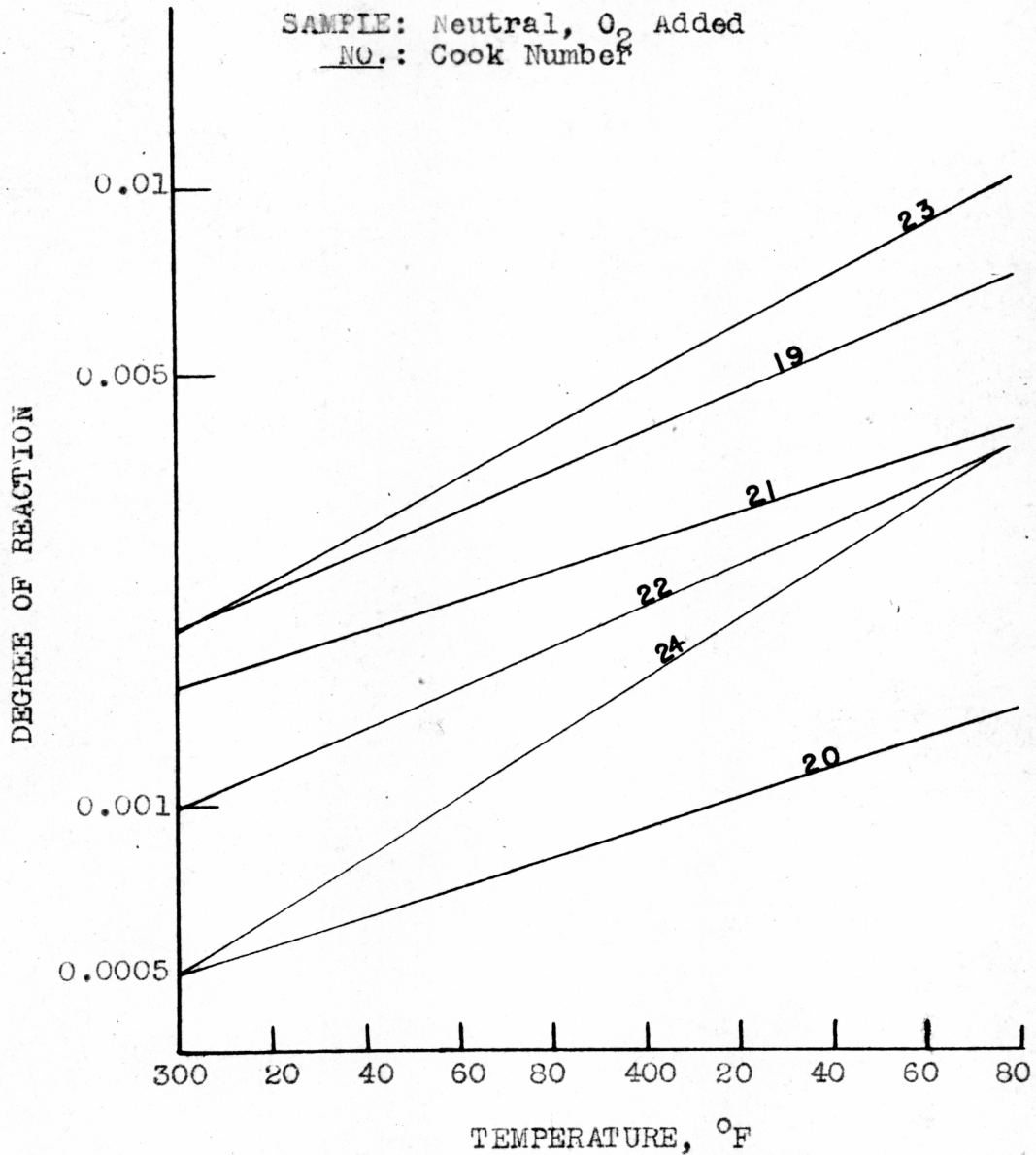


FIGURE 13. RELATIONSHIP OF DEGREE OF REACTION RATE TO TEMPERATURE IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

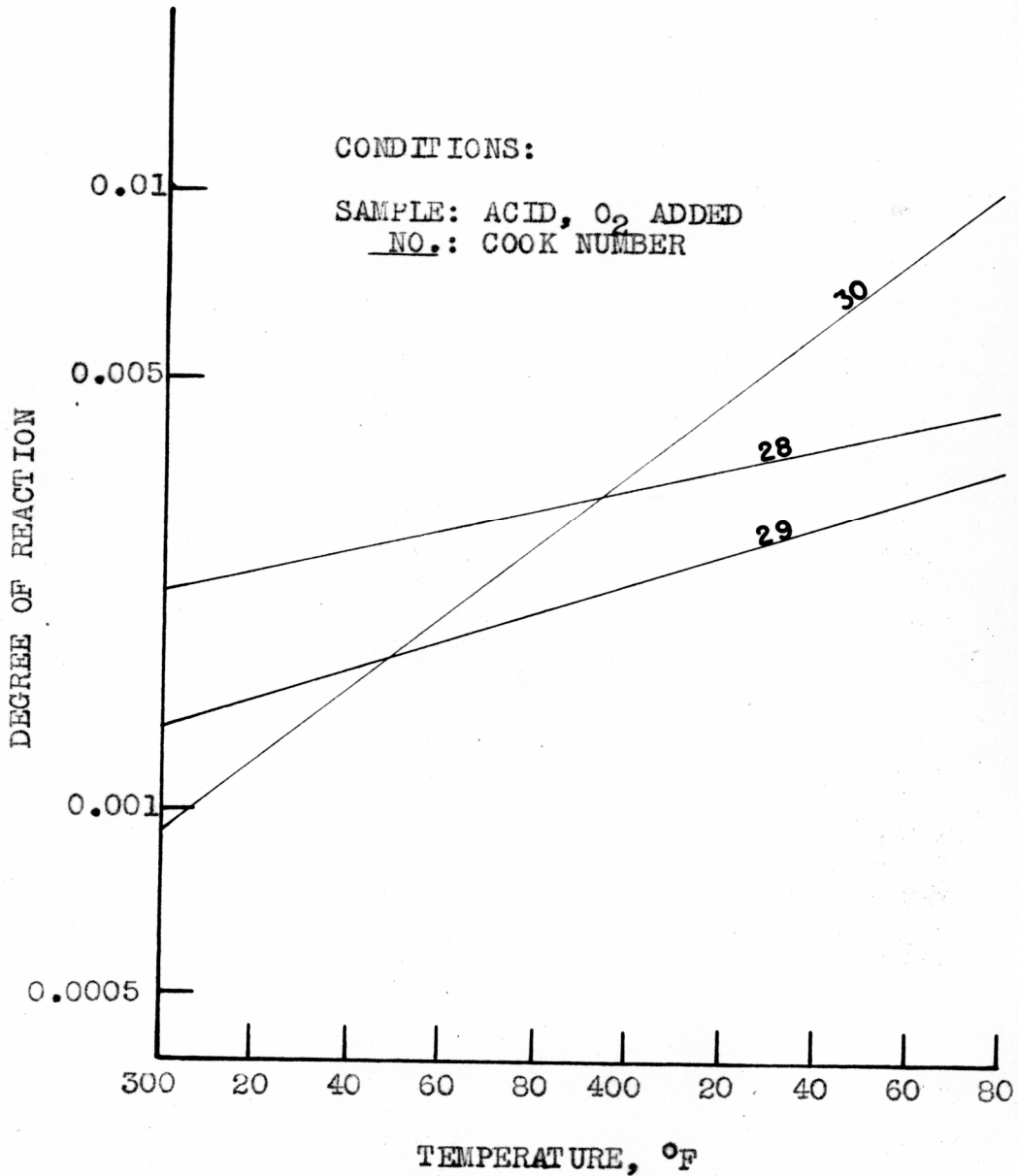


FIGURE 14. RELATIONSHIP OF DEGREE OF REACTION RATE TO TEMPERATURE IN HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

tests are shown in Tables 5 and 6, pages 55 and 56, and Figure 15, page 57.

The results obtained, as shown in Figure 15, can not be explained. It should, however, be noted that in all cooks where oxygen was added, the sum of the pressure due to the vapor pressure of water and the addition of oxygen was greater than the indicated gage pressure during the first 40 minutes of the cooks. For several cooks, number 25, 26, and 27, the degree of reaction rates were not calculated since their cooking times were equal to or less than 40 minutes.

Gas Produced. The quantity of gas produced during a cook and the amount of  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{O}_2$  and  $\text{H}_2\text{S}$  present in the gas is shown in Table 3, columns XV, XVI, XVII and XVIII. No correlation could be found between the volume of gas produced or the components of the gas and B.O.D. reduction as shown in Figures 2, 3, 4 and 5.

#### Limitations

The material limitations observed during the investigation were, pressure in autoclave, pH of the raw liquor, type of waste, and amount of oxygen added.

Pressure Maintained in Autoclave. Since the pressure gage used in the experimental part of the investigation was capable of readings only to 800 psi, it was decided that the highest pressure maintained in the series of cooks should

TABLE 5

PERCENT OF OXYGEN PRESENT IN A GAS PRODUCED DURING  
A COOK OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE TO  
WHICH OXYGEN HAD BEEN ADDED.

Conditions:

Sample = 465 ml., Neutral.

Grams of Oxygen added = 9.4

Time (min.)	O <sub>2</sub> (percent)	Gage Pressure (psi)
0	21.3	120
10	30	100
20	26	140
30	56	160
40	34	190
50	24	210
60	6	250
70	12	280
80	0	290
90	0	330



TABLE 6

PERCENT OF OXYGEN PRESENT IN A GAS IN CONTACT WITH SEMI-CHEMICAL NEUTRAL SULFITE WASTE LIQUOR TO WHICH OXYGEN HAD BEEN ADDED.

Conditions:

Sample = 465 ml., Neutral.

Grams of oxygen used = 7.4

Time (min.)	O <sub>2</sub> (percent)	Gage Pressure (psi)
0	32	100
20	31	100
40	58	90
50	66	85
1015	78	50

## CONDITIONS:

SAMPLE: 465 ml. Neutral  
 O : 9.4 Gms O<sub>2</sub> Added  
 X : 7.4 Gms O<sub>2</sub> Added

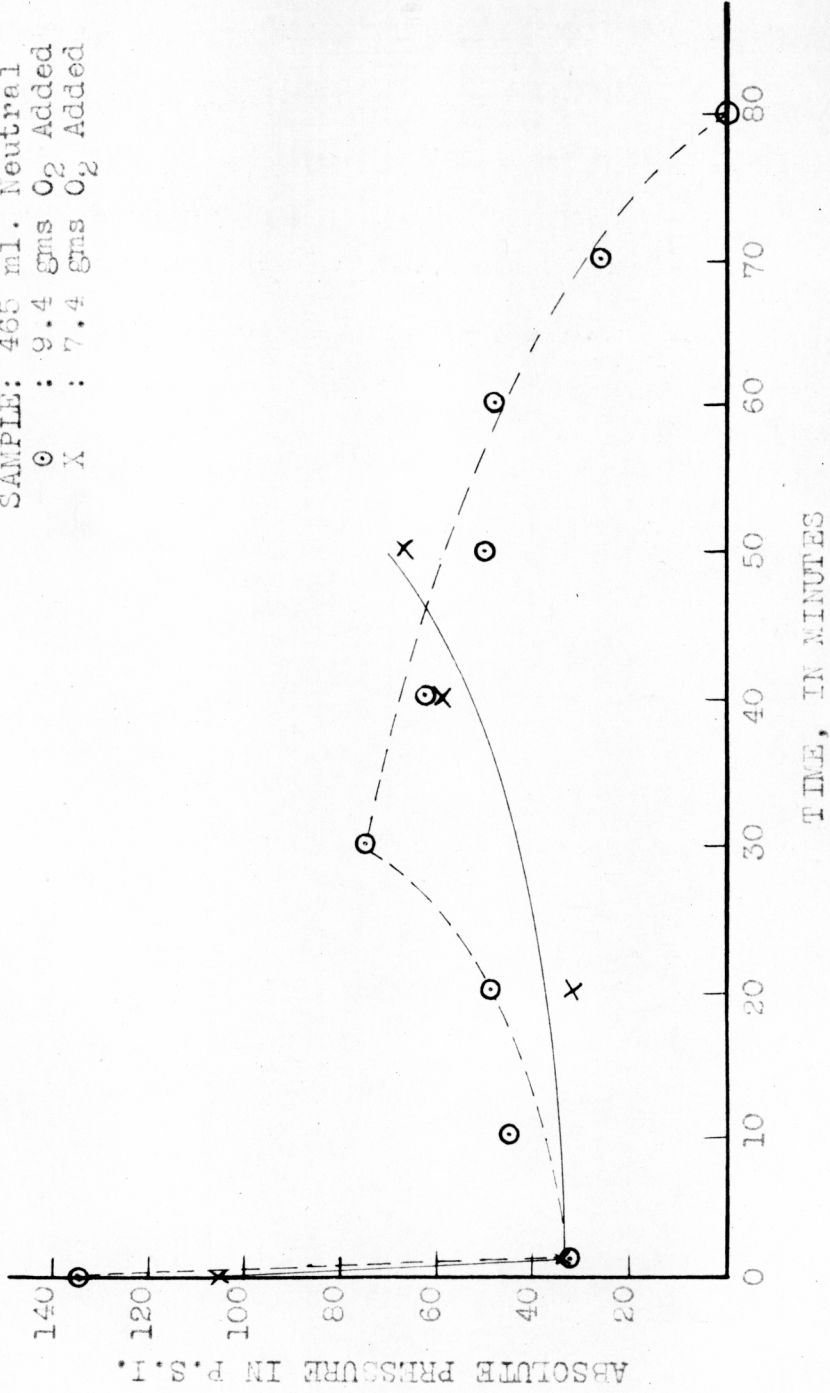


FIGURE 15. EFFECT OF ADDITION OF OXYGEN ON THE CONCENTRATION OF OXYGEN IN THE GAS PRODUCED DURING HEAT HYDROLYSIS OF SEMI-CHEMICAL NEUTRAL SULFITE WASTE.

be 600 psi.

pH of Raw Liquor. The pH of the sample used was not adjusted except in the acid series where it was lowered to pH of 3 by the addition of concentrated sulfuric acid.

Waste Used for Investigation. The only waste used in the investigation was neutral-sulfite, mixed-hardwoods, semi-chemical spent liquor.

Amount of Oxygen Added. When a 700 ml. sample was used 5.1 grams of oxygen was added, while 7.5 grams of oxygen was added when a 465 ml. sample was used. This was accomplished by the addition of oxygen to the autoclave until the oxygen exerted 100 psi gage pressure.

## VI CONCLUSIONS

The following are the conclusions obtained from the experiment:

1. The largest B.O.D. reduction possible is 60.2 percent. This reduction, although large, does not reduce the B.O.D. content of the liquor sufficiently to allow discharge without further treatment.

2. Adjusting the pH of the liquor to a value of 3 will result in a 13.4 percent greater reduction in the B.O.D. than obtained when the pH was not changed.

3. The addition of oxygen to Acid samples will give an 8.3 percent increase in the B.O.D. reduction; to 700 ml. Neutral sample a B.O.D. 16.5 percent greater than the raw sample; to 465 ml. Neutral sample, a B.O.D. reduction 5 percent less than that obtained in new cook without the addition of oxygen.

4. Dilution of liquor 1:1 will decrease the B.O.D. reduction in the Acid, Neutral, and Neutral-Oxygen added series of cooks, but increase it in the Acid-Oxygen added series. Dilutions of 2:1 decreases the B.O.D. reduction in the Neutral series while Acid, Acid-Oxygen added and Neutral-Oxygen added series show an increase.

5. No correlation can be made between B.O.D. reduction and degree of reaction rate.

6. No correlation can be found between B.O.D. reduction and any of the data obtained.

## VII SUMMARY

The object of this experiment was to determine if and to what extent the B.O.D. of S.N.W. waste could be reduced by Heat Hydrolysis. This process gave B.O.D. reduction up to 80 percent when used on Sulfite waste at Oregon State College and it was hoped that similar results could be obtained using S.N.W. liquor.

The experimental part of the investigation was conducted to determine (1) if the B.O.D. content of S.N.W. liquor could be reduced by Heat Hydrolysis; (2) if pH, dilution and the addition of oxygen were factors that affected the reduction of B.O.D. These factors were determined by adjusting the raw liquor to the desired concentration and cooking the liquor in a closed container until certain conditions were obtained, namely, that of constant pressure with constant temperature.

Analysis of the raw and cooked liquor consisted of determining pH, total solids, and B.O.D., while analysis of the gas created during the cook was limited to the total amount of gas created and the amount of  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{O}_2$  and  $\text{H}_2\text{S}$  in the gas.

Four different series of cooks were conducted on each sample. They were, Neutral (raw liquor), Acid (raw liquor pH adjusted, Neutral-Oxygen added (raw liquor with oxygen added) and Acid-Oxygen added (raw liquor pH adjusted with

oxygen added). The samples used were 700 ml., 465 ml., dilution 1:1 (232 ml. liquor plus 232 ml. distilled water) and dilution 2:1 (310 ml. distilled water plus 155 ml. liquor).

Industry requires a 90 percent or greater reaction in the B.O.D. content of the liquor before it will consider the use of a process. The best reduction obtained by Heat Hydrolysis was 60.2 percent, far below the minimum standard of industry.

When Heat Hydrolysis was employed acidifying the waste liquor gave a slight increase in the B.O.D. reduction; addition of oxygen resulted in an increase in B.O.D. reduction when the sample was acidify, a B.O.D. greater than the raw liquor for 700 ml. Neutral sample, and a reduction in the B.O.D. of the raw liquor when 465 ml. Neutral sample was used. Dilution 1:1 resulted in a decrease in B.O.D. reduction in comparison to that obtained in non-diluted liquor, while dilution 2:1 provided an increase in B.O.D. reduction when the same comparison was made.

No correlation was found to exist between B.O.D. reduction and total solids reduction, length of cook, highest temperature of cook, highest pressure of cook, amount of gas produced, the kind of gas produced or degree of reaction rate of the gas.

## VIII ACKNOWLEDGEMENTS

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## X VITA

Mr. Robert George Butler was born in Taft, California on February 12, 1924. After attending elementary school in Los Angeles, California, he graduated from Hollywood High School, Hollywood, California in January 1943.

In February 1943, the author entered the United States Army Air Forces and after attending Armorment School, he was assigned to the First Air Commando Group, and spent two years in India, Burma and China Theater of Operations with this organization.

In 1947, he was discharged from the service and enrolled at the University of California at Los Angeles for one and a half years. He then transferred to the University of Southern California where he received his B.E. degree in Civil Engineering in February, 1950. After graduation, he was employed by the Department of Water and Power, City of Los Angeles until June 1951 as a Sanitary Engineer.

To continue his studies, he enrolled at Virginia Polytechnic Institute in June 1951, and became a candidate for the Master of Science degree in Sanitary Engineering.

*Robert George Butler*

XI APPENDIX

Data Obtained During Cook of Sample

## COOK NO. 1

Sample: 700 ml. S.N.W.

pH Raw: 8.3

pH Cook: 8.6

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	81	0	0.81	0	
25	308	74.7	75.5	- 0.8	
40	343	234.7	123	111.7	
75	359	314.7	151	163.7	
85	328	314.7	100.5	214.2	
115	335	329.7	110.5	219.2	
130	323	304.7	93.5	211.2	
145	327	329.7	99	230.7	
160	340	354.7	118	236.7	
167	335	324.7	110.5	214.2	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00003917x - 0.01061$ .

Average deviation of data from above equation: + 0.00052.

## COOK NO. 2

Sample: 700 ml. S.N.W.

pH Raw: 8.4

pH Cook: 8.15

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	-	0	-		
10	241	39.7	25.5	14.2	
20	301	134.7	68.5	66.2	
39	369	464.7	171	293.7	
60	334	414.7	109	305.7	
90	333	444.7	107.5	337.2	
120	329	434.7	102	332.7	
150	326	424.7	97.5	327.2	
180	328	394.7	100.5	294.2	
210	317	414.7	86	328.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.000057x - 0.01409$ .

Average deviation of data from above equation: +0.00034.

## COOK NO. 3

Sample: 700 ml. S.N.W.

pH Raw: 8.5

pH Cook: 7.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	77	0	0.46	0	
5	187	64.7	8.9	55.8	
20	334	124.7	109	15.7	
35	359	334.7	151	183.7	
40	353	414.7	140	274.7	
65	297	314.7	64.5	250.5	
80	348	484.7	131.5	353.2	
95	326	442.7	97.5	345.2	
125	302	334.7	69.5	265.2	
155	332	434.7	106	328.7	
185	313	314.7	83.4	231.3	
210	333	414.7	107.5	307.2	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.000004615x + 0.002136$ .

Average deviation of data from above equation: -0.000109.

## COOK NO. 4

Sample: 700 ml. S.N.W.

pH Raw: 8.3

pH Cook: 8.6

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	80	0	0.5	0	
15	292	74.7	59.5	15.2	
20	321	164.7	91	73.7	
30	385	414.7	208	206.7	
65	356	414.7	146	268.7	
95	358	414.7	149.5	265.2	
125	349	414.7	133	281.7	
155	343	424.7	123	301.7	
185	342	404.7	121.5	283.2	
215	341	404.7	119.5	285.2	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.0000465x - 0.01263$ .

Average deviation of data from above equation: + 0.000044.



## COOK NO. 5

Sample: 700 ml. S.N.W.

pH Raw: 7.9

pH Cook: 8.45

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	-	0	-	-	
10	242	44.7	26	18.7	
15	288	64.7	56	8.7	
20	312	104.7	80	24.7	
30	327	154.7	99	53.7	
40	345	224.7	126.5	98.2	
50	370	334.7	173	161.7	
60	390	484.7	222	262.7	
65	409	584.7	274	310.7	
67	409	614.7	274	340.7	
68	415	644.7	293	351.7	
70	410	654.7	278	376.7	
73	411	674.7	280	394.7	Gas went out Re-lit
75	392	614.7	226	388.7	
80	393	594.7	227	367.7	
83	386	584.7	213	371.7	
85	372	544.7	177	367.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.0000367x - .00941$ .

Average deviation of data from above equation: - 0.000023.

## COOK NO. 6

Sample: 465 ml. S.N.W.

pH Raw: 8.0

pH Cook: 8.8

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	-	0	-	-	
5	-	0	-	-	
10	312	34.7	80	312	
20	325	124.7	96.5	325	
30	345	179.7	126	345	
40	355	264.7	143.5	355	
50	375	354.7	185	375	
55	383	414.7	204	383	
60	380	447.7	196	380	
65	385	474.7	208	385	
70	390	509.7	222	390	
75	390	529.7	222	390	
80	390	554.7	222	390	
85	395	584.7	234	395	
90	395	584.7	234	395	
95	395	584.7	234	395	
100	398	594.7	242	398	

Equation for degree of reaction rate obtained by method of least squares:  $y = 0.00004x - 0.01116$ .

Average deviation of data from above equation:  $+ 0.000323$ .

## COOK NO. 7

Sample: 464 ml. (232 ml. S.N.W. 232 ml.  
distilled water).

pH Raw : 7.65

pH Cook: 8.3

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	-	0	-	-	
10	284	64.7	52.5	12.2	
20	313	104.7	81.5	23.2	
30	325	159.7	96.5	61.2	
40	338	224.7	115	107.7	
50	353	324.7	140	184.7	
60	341	349.7	120	229.7	
70	348	384.7	131	253.7	
80	355	422.7	144	278.7	
90	362	454.7	157	297.9	
100	364	484.7	161	323.7	
110	369	499.7	171	326.7	
120	369	529.7	171	358.7	
130	370	549.7	173	376.7	
140	370	564.7	173	391.7	
150	372	579.7	177	402.7	
160	363	584.7	159	425.7	
170	363	584.7	159	425.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00003066x - 0.007399$ .

Average deviation of data from above equation: + 0.00034.

COOK NO. 8

Sample: Dilution 2:1 (155 ml. liquor  
310 ml. distilled water).

pH Raw: 7.98

pH Cook: 6.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	-	0	-	-	
10	246	44.7	27.5	17.2	
20	303	94.7	70.5	24.2	
30	322	144.7	92.5	52.2	
40	330	194.7	103.5	91.2	
50	337	244.7	113.5	131.2	
60	348	334.7	115	219.7	
70	357	414.7	147.5	267.2	
80	370	504.7	173	331.7	
90	382	594.7	202	392.7	
95	365	534.7	163	371.7	
100	358	494.7	149.5	345.2	
110	344	464.7	124.5	340.2	
120	355	454.7	143.5	311.2	
130	356	454.7	145.5	309.2	
140	352	449.7	138.5	311.2	
150	360	464.7	153	311.7	
160	361	466.7	155	311.7	
165	361	466.7	155	311.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.0000215x - 0.00403$ .

Average deviation of data from above equation:  $+0.000025$ .

## COOK NO. 9

Sample: 700 ml. S.N.W.

pH Raw: 7.7

pH after Sulfuric Acid Added: 3.15

pH Cook: 5.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	40	0	-	0	
5	81	0	51	-51	
20	213	39.7	15	24.7	
35	326	98.7	97.5	1.2	
50	384	274.7	207	67.7	
63	388	414.7	217	197.7	
65	409	454.7	274	180.7	
70	364	414.7	161	253.7	
100	344	419.7	123	296.7	
130	349	424.7	133	291.7	
160	349	414.7	133	281.7	
190	334	409.7	109	300.7	
220	347	414.7	129.5	285.2	

Equation for degree of reaction rate obtained by method of least squares:  $y = 0.00000856x - 0.000446$ .

Average deviation of data from above equation: + 0.0000071.

## COOK NO. 10

Sample: 700 ml. S.N.W.

pH Raw: 7.7

pH after Sulfuric Acid Added: 3.05

pH Cook: 5.5

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	81	0	0.52	-	
15	210	39.7	14.1	25.6	
30	334	109.7	109	0.7	
50	420	414.7	309	105.7	
90	357	439.7	147.5	292.2	
120	351	429.7	137	292.7	
165	344	419.7	124.5	284.7	
170	328	409.7	115	284.7	
200	353	439.7	140	299.7	
220	333	384.7	107.5	277.2	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00000442x + 0.000659$ .

Average deviation of data from above equation: - 0.0000285.

## COOK NO. 11

Sample: 700 ml. S.N.W.

pH Raw: 8.3

pH after Sulfuric Acid Added: 3.15

pH Cook: 5.35

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	84	0	0.58	0	
15	225	39.7	19	20.7	
20	275	54.7	45.5	9.2	
30	320	134.7	89.5	45.2	
45	389	424.7	217.5	207.5	
85	370	424.7	174	250.7	
115	358	384.7	149.5	235.2	
145	382	429.7	202.5	227.2	
175	369	434.7	171	263.7	
205	365	414.7	163	251.7	
235	372	414.7	178	234.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00001693x^2 - 0.00353$ .

Average deviation of data from above equation: -0.00004.

## COOK NO. 12

Sample: 700 ml. S.N.W.

pH Raw: 8.4

pH after Sulfuric Acid Added: 3.15

pH Cook: 5.01

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	42.5	0	0.2	-	
10	229	39.7	20.5	19.2	
25	328	204.7	100.5	104.3	
35	381	414.7	199	215.7	
75	354	444.7	142	302.7	
105	341.5	394.7	120.5	274.2	
135	352	409.7	138.5	271.2	
165	359	409.7	117	282.7	
195	319	389.7	88.5	301.2	
205	381.5	414.7	105	309.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00006138x - 0.01702$ .

Average deviation of data from above equation: - 0.00022.



## COOK NO. 13

Sample: 700 ml. S.N.W.

pH Raw: 8.5

pH after Sulfuric Acid Added: 3.1

pH Cook: 4.7

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	80	0	0.5	-0.5	
10	333	124.7	107	17.7	
15	408	264.7	272.5	-7.8	
20	426	414.7	330	81.7	
25	439	554.7	375	179.7	
45	342	414.7	121.5	293.2	
75	357	414.7	147.5	267.2	
105	359	454.7	151	283.7	
135	349	394.7	133	261.7	
165	360	444.7	153	291.7	
200	373	424.7	180	244.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00000635x + 0.001243$ .

Average deviation of data from above equation: + 0.000055.

COOK NO. 14

Sample: 700 ml. S.N.W.

pH Raw: 8.2

pH after Sulfuric Acid Added: 3.15

pH Cook: 5.05

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	88	0	0.65	0	
15	328	274.7	100.5	174.2	
25	384	414.7	207.5	207.2	
35	356	414.7	145.5	269.2	
65	352	414.7	138.2	276.5	
95	345	414.7	126	288.7	
125	352	404.7	138.2	266.5	
155	360	424.7	153	271.7	
185	357	454.7	147.5	307.2	
205	349	414.7	133	281.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.000152x - 0.04925$ .

Average deviation of data from above equation: + 0.00011.

## COOK NO. 15

Sample: 700 ml. S.N.W.

pH Raw: 7.5

pH after Sulfuric Acid Added: 3.15

pH Cook: 4.7

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	50	0	0.2	0	
10	121	0	1.7	-1.7	
20	213	34.7	15	19.7	
30	290	89.7	57.5	31.2	
40	346	199.7	128	71.7	
52	385	394.7	208	86.7	
55	385	414.7	208	206.7	
58	381	414.7	199	215.7	
60	373	409.7	180	229.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.0000131x - 0.00136$ .

Average deviation of data from above equation: -0.000125.

## COOK NO. 16

Sample: 465 ml. S.N.W.

pH Raw: 7.78

pH after Sulfuric Acid Added: 2.98

pH Cook: 5.1

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0		0	0	0	
10	265	54.7	38.5	16.2	
20	295	79.7	62	17.7	
30	322	124.7	92.5	32.2	
40	350	184.7	135	49.7	
50	360	254.7	153	101.7	
60	380	344.7	196	148.7	
70	390	399.7	222	177.7	
80	391	439.7	224	215.7	
90	395	474.7	234	240.7	
100	395	504.7	234	270.7	
110	395	534.7	234	300.7	
120	393	564.7	228	336.7	
130	393	564.7	228	356.7	
140	393	604.7	228	376.7	
150	405	629.7	263	366.7	
160	375	554.7	185	369.7	Gas lowered
170	385	544.7	208	336.7	
180	385	544.7	208	336.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00001798x - 0.0041$ .

Average deviation of data from above equation: + 0.00013.

## COOK NO. 17

Sample: 464 ml. (232 ml. liquor  
232 ml. distilled water).

pH Raw: 7.7

pH after Sulfuric Acid Added: 3.0

pH Cook: 4.7

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0		0			
10	245	54.7	27.5	27.2	
20	278	79.7	47.5	32.2	
30	313	149.7	81.5	68.2	
40	331	214.7	104.5	110.2	
50	355	314.7	143.5	171.2	
60	373	414.7	180	234.7	
70	382	524.7	203	321.7	
85	382	544.7	203	341.7	
90	382	549.7	203	346.7	
100	384	564.7	207	357.7	
110	392	574.7	228	346.7	
120	377	579.7	189	390.7	
135	382	594.7	202	392.7	
140	383	596.7	203	393.7	
145	386	599.7	211	388.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00001864x - 0.003024$ .

Average deviation of data from above equation: -0.000021.

## COOK NO. 18

Sample: 465 ml. (155 ml. liquor  
310 ml. distilled water).

pH Raw: 7.8

pH after Sulfuric Acid Added: 2.8

pH Cook: 4.5

Time (Min.)	Temp. (°F)	Gage Pres. Absol.(psi)	Pres. H <sub>2</sub> O Absol.(psi)	Partial Pres. Absol.(psi)	Remarks
0	74	0	-	0	
10	228	39.7	20.2	9.5	
20	291	74.7	58.5	16.2	
30	308	109.7	75.5	34.2	
40	317	154.7	86	68.7	
50	324	199.7	95	104.7	
60	331	264.7	104.5	160.2	
70	336	324.7	112	212.7	
80	350	384.7	135	249.7	
90	362	464.7	157	307.7	
100	363	514.7	159	355.7	
110	365	564.7	163	401.7	
120	365	594.7	163	431.7	
130	372	609.7	178	431.7	
135	373	609.7	180	429.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00002475x - 0.005072$ .

Average deviation of data from above equation: + 0.000025.

## COOK NO. 19

Sample: 700 ml. S.N.W.

pH Raw: 8.5

pH Cook: 8.3

Grams of Oxygen Added: 5.1

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres.H <sub>2</sub> O Absol. (psi)	Pres.O <sub>2</sub> Absol. (psi)	Partial Pres.Absol. (psi)	Remarks
0	80	114.7	0.55	114.7	- 0.55	
16	289	114.7	56.5	65	- 6.8	
25	342	164.7	121.5	84.8	-41.6	
30	312	134.7	80	107	-52.3	
40	329	234.7	102	90.5	42	
54	341	414.7	120	71.3	223.4	
60	321	424.7	91	55	278.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00002086x - 0.0043$ .

Average deviation of data from above equation: - 0.00043.

## COOK NO. 20

Sample: 700 ml. S.N.W.

pH Raw: 8.5

pH Cook: 6.45

Grams of Oxygen Added: 5.1

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres. H <sub>2</sub> O Absol. (psi)	Pres. O <sub>2</sub> Absol. (psi)	Partial Pres. Absol. (psi)	Remarks
0	141	114.7	2.8	114.7	- 2.8	
5	277	134.7	47	95.5	-17.6	
10	365	164.7	163	60.3	- 8.6	
15	392	214.7	227.5			
20	398	294.7	242.5	70	-17.8	
25	416	414.7	297.5	83.3	13.9	
38	408	409.7	272.5	99.2	38.0	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.000003738x - 0.00059$ .

Average deviation of data from above equation: +0.0000033.



## COOK NO. 21

Sample: 700 ml. S.N.W.

pH Raw: 8.35

pH Cook: 8.05

Grams of Oxygen Added: 5.1

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres.H <sub>2</sub> O Absol. (psi)	Pres.O <sub>2</sub> Absol. (psi)	Partial Pres.Absol. (psi)	Remarks
0	78	114.7	0.49	114.7	- 0.49	
7	189	114.7	9.1	74.8	30.8	
12	303	129.7	70.5	62.4	- 3.2	
17	342	174.7	121.5	70	-16.8	
22	356	234.7	145.5	78.8	10.4	
27	362	274.7	167	95	12.7	
32	364	304.7	161	107.2	36.5	
37	356	364.7	145.5	100	119.2	
42	352	414.7	138.5	90.6	185.6	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.000011x - 0.00174$ .

Average deviation of data from above equation: -0.000125.

## COOK NO. 22

Sample: 465 ml. S.N.W.

pH Raw: 8.2

pH Cook: 7.25

Grams of Oxygen Added: 7.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres. H <sub>2</sub> O Absol. (psi)	Pres. O <sub>2</sub> Absol. (psi)	Partial Pres. Absol. (psi)	Remarks
0	80	114.7	0.6	114.7	- 0.6	
10	235	124.7	22.7	74	28.0	
20	375	184.7	165	120	- 0.3	
30	400	264.7	250	96	-81.3	
40	375	272.7	165	77.8	29.9	
50	380	304.7	196	75.5	33.2	
60	390	344.7	221	41.0	82.7	
70	393	404.7	227	0	177.7	
80	395	444.7	234	0	210.7	
90	400	484.7	249	0	235.7	
100	385	504.7	207	0	297.7	
110	380	499.7	196	0	303.7	
120	382	489.7	203	0	286.7	
130	383	484.7	204	0	280.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $\bar{z} = 0.00001183 - 0.00259.$

Average deviation of data from above equation:  $-0.000216.$

## COOK NO. 23

Sample: 464 ml. (232 ml. S.N.W. liquor  
232 ml. distilled water).

pH Raw: 7.65

pH Cook: 7.8

Grams of Oxygen Added: 7.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres.H <sub>2</sub> O Absol. (psi)	Pres.O <sub>2</sub> Absol. (psi)	Partial Pres.Absol. (psi)	Remarks
0	79	114.7	0.49	114.7	- 0.49	
10	221	74.7	17.8	56	0.9	
20	328	119.7	100.5	69	-49.8	
30	326	144.7	97.5	110	-62.8	
40	318	164.7	87.5	89	-11.8	
50	298	184.7	65	72	47.7	
60	322	284.7	92.5	69.6	122.6	
70	342	404.7	121.5	38.6	244.6	
80	369	544.7	171	0	373.7	Turned Gas down
90	364	564.7	161	0	403.7	
95	364	594.7	161	0	433.7	
100	364	604.7	161	0	443.7	
105	365	614.7	163	0	451.7	
110	367	624.7	167	0	457.7	
115	375	624.7	185	0	439.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00004018x - 0.010156$ .

Average deviation of data from above equation:  $\pm 0.00016$ .

COOK NO. 24

Sample: 465 ml. (155 ml. S.N.W.  
310 ml. distilled water).

pH Raw: 7.9

pH Cook: 5.19

Grams of Oxygen Added: 7.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres. H <sub>2</sub> O Absol. (psi)	Pres. O <sub>2</sub> Absol. (psi)	Partial Pres. Absol. (psi)	Remarks
0	74	114.7	0.43	114.7	0.43	
10	246	134.7	27.5	58.2	49.0	
20	358	179.7	149.5	73.5	-33.3	
30	381	124.7	199	121	-95.3	
40	388	279.7	217	98.4	-35.7	
50	402	344.7	252	80.6	12.1	
60	416	414.7	297	79	38.7	
70	428	499.7	335	43.3	121.4	
80	428	559.7	335	0	224.7	
90	423	584.7	318	0	266.7	
100	402	584.7	253	0	331.7	
105	406	584.7	264	0	320.7	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00001504x - 0.00395$ .

Average deviation of data from above equation: + 0.000341.

## COOK NO. 25

Sample: 700 ml. S.N.W. Liquor

pH Raw: 8.25

pH after Sulfuric Acid Added: 3.15

pH Cook: 4.45

Grams of Oxygen Added: 5.1

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres.H <sub>2</sub> O Absol. (psi)	Pres.O <sub>2</sub> Absol. (psi)	Partial Pres.Absol. (psi)	Remarks
0	84	114.7	0.59	114.7	- 0.59	
10	263	134.7	37.5	58.5	38.7	
15	268	194.7	170	70.1	-45.4	
20	409	264.7	273	76.6	-84.9	
25	434	354.7	358	93.6	-96.9	
28	447	414.7	410	107	-102.3	
34	436	424.7	365	114	-54.3	
37	442	434.7	390	110	-65.3	
44	413	424.7	387	91.6	-43.9	

Since all but one partial pressure was negative, no degree of reaction rate could be calculated.

## COOK NO. 26

Sample: 700 ml. S.N.W. Liquor

pH Raw: 7.96

pH after Sulfuric Acid Added: 3.1

pH Cook: 4.2

Grams of Oxygen Added: 5.1

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres. H <sub>2</sub> O Absol. (psi)	Pres. O <sub>2</sub> Absol. (psi)	Partial Pres. Absol. (psi)	Remarks
0	71	114.7	0.39	114.7	- 0.39	
5	94	114.7	0.80	83.4	29.5	
10	210	124.7	14.1	55.4	55.2	
15	323	154.7	100.5	68.3	-14.1	
20	382	209.7	203	76	-69.3	
25	420	309.7	310	94.5	-94.6	
30	442	414.7	390	127	-102.3	
35	440	414.7	385	115	- 85.3	

Since all but two partial pressures were negative no degree of reaction rate could be calculated.

## COOK NO. 27

Sample: 700 ml. S.N.W. Liquor

pH Raw: 7.8

pH after Sulfuric Acid Added: 3.0

pH Cook: 4.3

Grams of Oxygen Added: 5.1

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres. H <sub>2</sub> O Absol. (psi)	Pres. O <sub>2</sub> Absol. (psi)	Partial Pres. Absol. (psi)	Remarks
0	84	114.7	0.59	114.7	- 0.59	
5	184	114.7	8.4	94.5	11.8	
10	320	144.7	89.5	63.2	- 8.0	
15	370	214.7	174	70.3	-29.6	
20	414	314.7	289	77	-51.3	
25	446	414.7	408	95.2	-88.5	
27	451	454.7	428	102.1	-75.4	
30	442	424.7	390	124.5	-89.8	

Since all but one partial pressures were negative no degree reaction rate could be calculated.

## COOK NO. 28

Sample: 485 ml. S.N.W. Liqueur

pH Raw: 7.78

PH after Sulfuric Acid Added: 3.1

PH Cook: 4.91

Grams of Oxygen Added: 7.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres. H <sub>2</sub> O Absol. (psi)	Pres. O <sub>2</sub> Absol. (psi)	Partial Pres. Absol. (psi)	Remarks
0	75	114.7	0.43	114.7	- 0.43	
10	250	154.7	30	58.4	46.3	
20	375	199.7	183.5	74.8	-58.5	
30	400	284.7	246.5	111.8	-73.6	
35	420	379.7	310	118	-42.3	
40	415	394.7	293	101	0.7	
50	400	404.7	246.5	80.5	74.7	
60	390	438.7	220	76.2	140.5	
68	396	464.7	234	51.2	179.5	
70	395	474.7	234	41.6	198.1	
75	390	494.7	220	0	274.7	
80	384	511.7	208	0	305.7	
85	388	524.7	215	0	309.7	
90	358	534.7	215	0	319.7	
95	380	564.7	198	0	368.7	
100	384	574.7	208	0	368.7	
108	374	564.7	181	0	383.7	
110	373	544.7	179	0	365.7	
115	365	534.7	162	0	372.7	
120	362	524.7	157	0	367.7	
125	365	522.7	162	0	360.7	
130	365	519.7	162	0	357.7	
135	362	516.7	157	0	359.7	
140	362	514.7	157	0	357.7	
145	366	514.7	164	0	350.1	
150	368	514.7	169	0	345.1	

Turned  
gas down

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.000019403x - 0.00444$ .

Average deviation of data from above equation: -0.000258.



## COOK NO. 29

Sample: 464 ml. (232 ml. S.N.W.  
232 ml. distilled water)  
Dilution 1:1

pH Raw: 7.7

pH after Sulfuric Acid Added: 2.95

pH Cook: 4.4

Grams of Oxygen Added: 7.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres.H <sub>2</sub> O Absol. (psi)	Pres.O <sub>2</sub> Absol. (psi)	Partial Pres.Absol. (psi)	Remarks
0	73	114.7	0.41	114.7	- 0.41	
10	282	134.7	50.5	61.2	23.0	
20	398	264.7	242	77	-54.3	
30	422	354.7	318	124	-77.3	
35	428	414.7	338	113	36.3	
40	441	484.7	385	105	- 5.3	
45	445	544.7	442	93.5	81.7	
50	456	614.7	448	85	105.3	
55	444	584.7	398	81.4	85.2	
60	454	599.7	440	74.5		

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00001106 - 0.002053.$

Average deviation of data from above equation: -0.00002.

COOK NO. 30

Sample: 465 ml. (155 ml S.N.W. Liquor  
310 ml. distilled water)

Dilution 2:1

pH Raw: 7.9

pH after Sulfuric Acid Added: 2.98

pH Cook: 4.05

Grams of Oxygen Added: 7.4

Time (Min.)	Temp. (°F)	Gage Pres. Absol. (psi)	Pres. H <sub>2</sub> O Absol. (psi)	Pres. O <sub>2</sub> Absol. (psi)	Partial Pres. Absol. (psi)	Remarks
0	80	114.7	0.51	114.7	- 0.51	
10	256	144.7	53.5	63.6	57.6	
20	375	204.7	185	74.4	-54.7	
30	392	264.7	227	118.5	-80.8	
40	389	324.7	218	97.5	19.2	
50	388	364.7	217	78.5	69.2	
60	385	399.7	207	75	117.7	
70	371	424.7	175	40	209.7	
80	364	439.7	161	0	278.7	
90	345	439.7	126.5	0	313.2	

Equation for degree of reaction rate obtained by method  
of least squares:  $y = 0.00003686 - 0.01015x$ .

Average deviation of data from above equation: + 0.00076.