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The Effect of pH Variation on the Evolution of Non-Condensable Gases from Hardwood Black Liquors

Gregory Allen Goode
Western Michigan University

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**THE EFFECT OF PH VARIATION ON
THE EVOLUTION OF NON-CONDENSIBLE GASES
FROM HARDWOOD BLACK LIQUORS**

A Paper Submitted in **Partial** Fulfillment
For Senior Thesis
Paper 473

By: Gregory Allen Goode 

Advisor: Dr. Ellsworth Shriver

June 23, 1988

ABSTRACT

A distillation process utilizing hardwood black liquor samples from Mead in Escanaba was used to determine the effect of pH on non-condensable gas evolution.

pH was found to be highly negatively correlated to the volumetric production of non-condensable gas. The correlation for all the black liquor samples tested was found to be greater than -0.990 . This indicates that as pH increases the volumetric production of non-condensable gas decreases.

pH was found to be more correlated to the effect of gas volume reduction in the pH range of 11.5 to 12.5.

Non-condensable gases produced in the fifth and sixth effects were found to represent approximately 40 percent of the total amount of non-condensable gas evolved in the evaporator system at Mead in Escanaba

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INTRODUCTION

The current theme motivating the production philosophy for most paper mills is how to produce paper at the lowest cost. This is the premise under which the problem of non-condensable gas evolution has developed.

The Mead mill in Escanaba is currently promoting chemical savings in the kraft mill by lowering the active alkali charged to the digesters during hardwood cooks. As a result of this, lower amounts of residual alkali exist in the spent cooking liquor and subsequently the black liquor is at a lower pH than normal. Mead has noticed that the evaporator system has a lower capacity during evaporation of the liquor from these cooks. The mill has found that caustic adjustment of this black liquor entering the evaporators has a noticeable effect on increasing the capacity of the system.

OBJECTIVE

The objective of this thesis is to analyze the sensitivity of hardwood non-condensable gas evolution to pH variation. Analysis will be derived from variation of pH from 10 to 13.6 on two black liquor samples of similar solids levels. It is not the purpose of this thesis to solve the problem or make assessments on the chemical reactions that may be occurring during this operation. The purpose is to merely assess the relationship of pH and non-condensable gas evolution and determine some volumetric

measurement. A Conclusion will also be drawn on the amount of non-condensable gas produced in the industry by using the numbers determined in the lab.

BACKGROUND INFORMATION

The production of a large amount of non-condensable gases (NCG) can cause a vacuum system to become inadequate in venting surface condensers used in gas evacuation systems.¹ This occurs because NCG build up within the condenser blankets tubes and reduces the efficiency of the condensers. As a result of this build up of gas, the jet ejector systems become overloaded and result in a loss of vacuum. Figure 1 is an illustration of the gas evacuation system utilized at Mead. This system has two surface condensers and three coolers.

One of the most common problems encountered by an evaporator system is the loss of sufficient vacuum. Most multiple effect evaporators operate in a range of 23-29 ins. of Hg on the vapor body of the last effect.² Operating at insufficient vacuum reduces the overall working temperature difference (heat available for evaporation) resulting in a loss of evaporation capacity. Table 1 shows the premise of the heat transfer equation that governs the operation of evaporators.

Figure 1:

6TH EFFECT NON-CONDENSIBLE FLOW

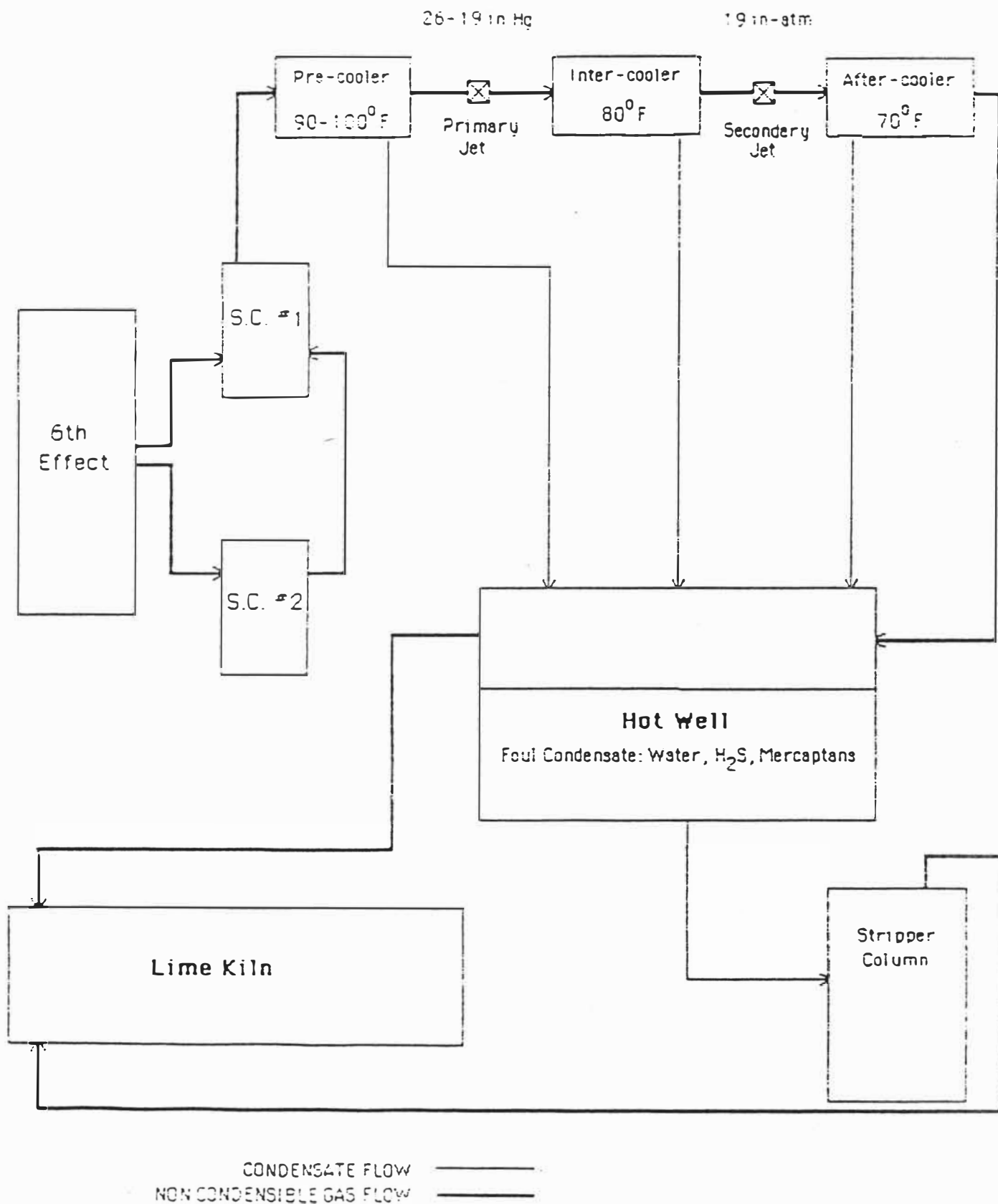


Table 1

Heat Transfer Equation

$$\text{Equation: } Q = UA(\Delta T)$$

where:

Q = BTU's transferred per hour

A = Area of heating surface in square feet

Delta T = Temperature drop (Steam temp. minus temp. of fluid heated)

U = Coefficient of heat transfer

Loss of vacuum on an evaporator can occur for reasons other than NCG loading on the system. The most common is scaling and fouling which occurs on the tubing inside the condenser. This cuts down the efficiency of heat transfer and gas volume reduction. Vacuum can also be lost due to leaks in piping and poor performance of ejector equipment. Operating personnel are able to counter vacuum loss temporarily by operating the system at higher first effect steam pressure and temperature.⁴ This increases the overall working temperature difference. This compensates for the loss of vacuum but steam economy for the system is lost.

The Calcasieu Paper Company in Elizabeth, LA related its recovery problems during a trial period to low residual active alkali.⁵ The mill experienced lower evaporator capacity when they reduced active alkali charged to digesters and used time and temperature instead of kappa number for cooking control. The evaporators went from a 400 -425 gpm capacity to a 375 - 400 gpm capacity.

The mill approached the problem by increasing the number of boilouts on the system. This providing only temporary relief, and it was later determined that silica buildup on the evaporator tubes was not occurring and was not the cause of the problem. Low vacuum on the fifth effect prompted vacuum leak checks and steam ejector modifications. No leaks were found and the modifications did not alter the problem. The mill eventually solved the problem by increasing the residual alkali level in the spent liquor. They increased the level by five percent based on solids and capacity on the evaporator system was increased.

The mill attributed the increase and decrease in the capacity of the evaporators to the variation of liquor viscosity. At higher residual alkali the liquor had a lower viscosity and vice versa for lower residual alkali. It has been found that variation in liquor viscosity affects the rate of heat transfer and mass transfer, and changes the flow characteristics in the

recovery operations. More viscous liquors have slower flows and transfer heat at lower rates than less viscous liquors. The Calcasieu mill attributed its problem to this phenomenon.

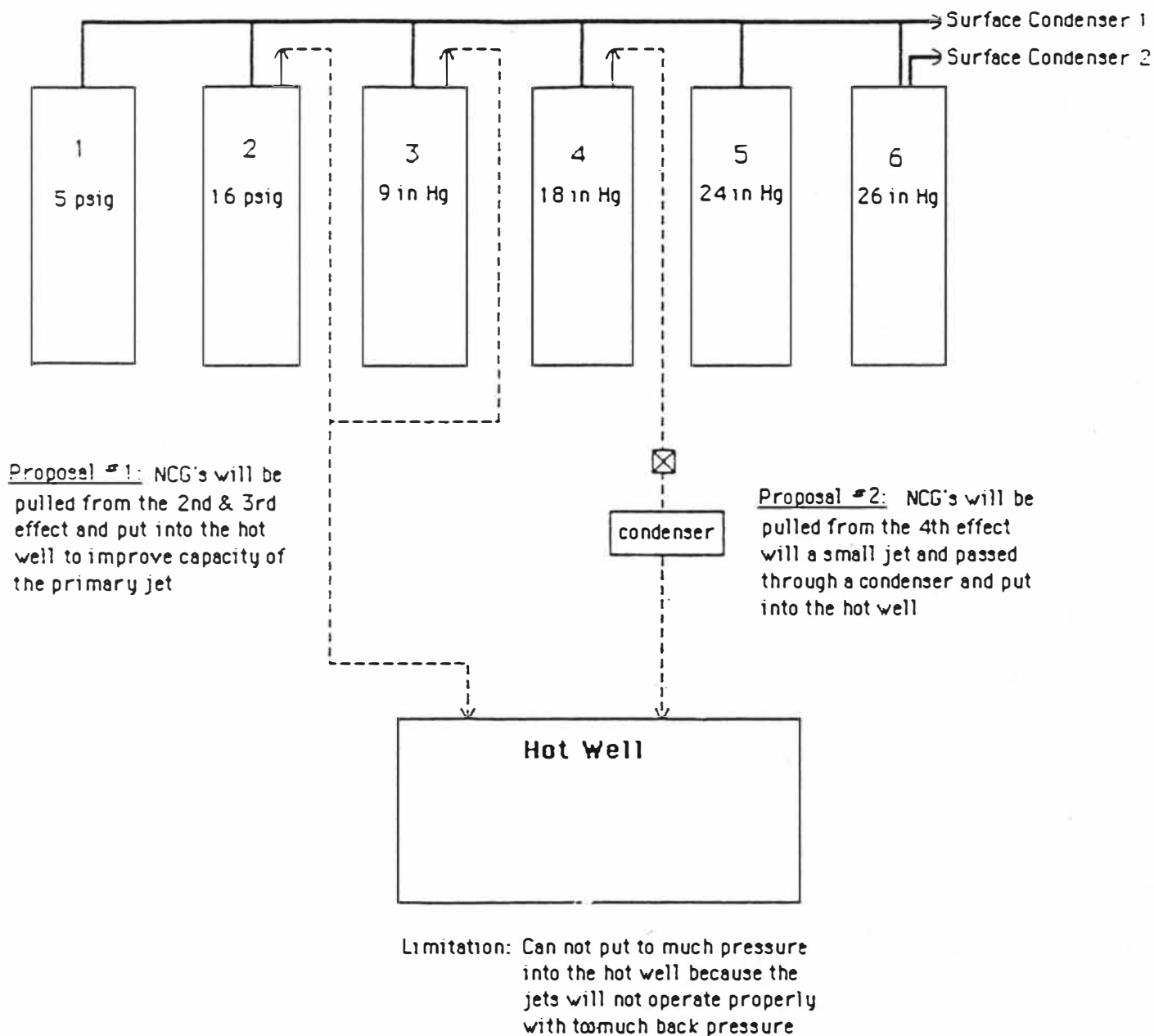
Mead's current belief is that they have a problem with non-condensable gas evolution in the fifth and sixth effects. They feel that the gas is loading up the system and lowering the capacity of their evaporators. Figure 2 shows the proposals under consideration and corrective action taken this far concerning the problem.

The mill has found that caustic adjustment on incoming black liquor increases the vacuum in the sixth effect which is lowered during hardwood black liquor runs. Figure 3 shows that residual alkali during hardwood black liquor runs is comparatively lower than during softwood runs. The gradual decrease in the residual alkali encountered during switch overs between wood types is due to hardwood and softwood liquors displacing liquors in the weak liquor storage tanks. A decreasing curve can be noticed as hardwood black liquor displaces softwood black liquor, and an increasing residual alkali can be seen as softwood liquor displaces hardwood liquor in the the weak liquor tank.

Mead currently adjusts its hardwood black liquor with purchased

Figure 2:

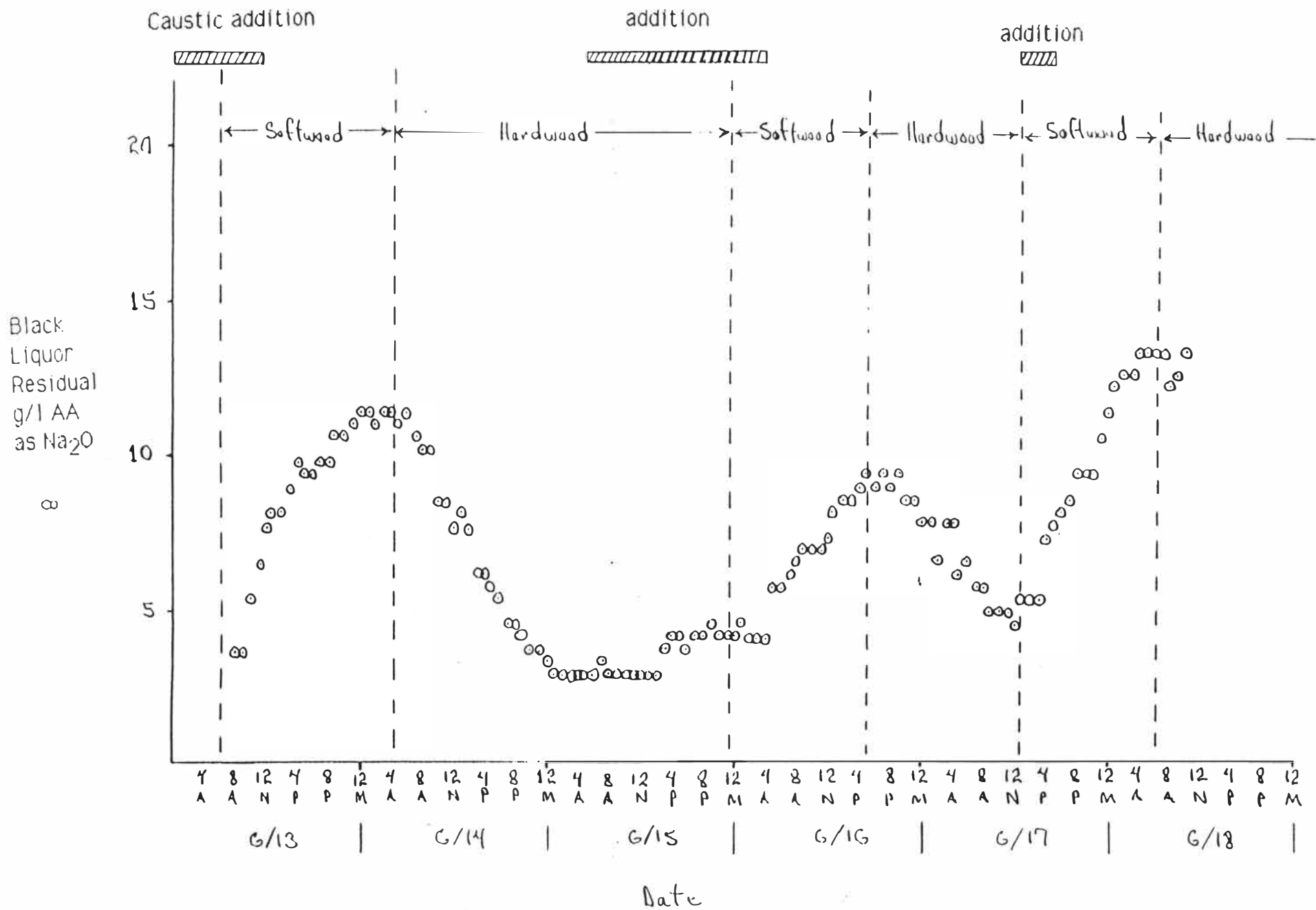
EVAPORATOR TRAIN



Corrective action taken this far:

1. Increased the size of the pre-cooler - gained 1/2 in of Hg
2. Installed Dip tubes in Surface condenser #1
3. Manometer on Jets
 - Secondary Jet - 22 in Hg to atm
 - Primary Jet - is not keeping the Secondary Jet supplied

Figure 3: Hardwood and Softwood Residual Active Alkali



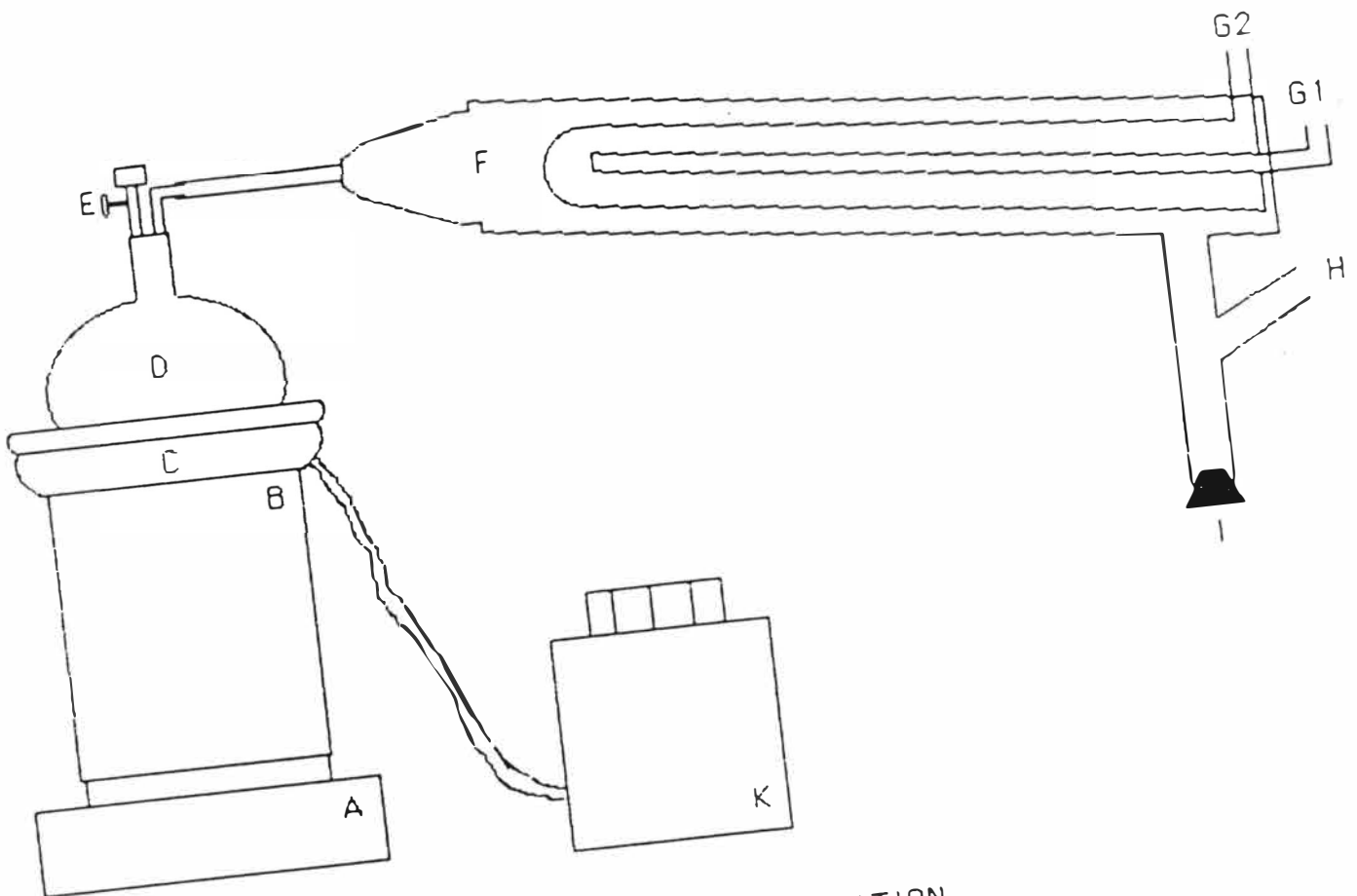
caustic. This is an expensive undertaking and defeats the purpose of promoting chemical savings in the kraft mill by using lower active alkali cooks. Under consideration at this time is the use of recycled white liquor for adjustment purposes.

EXPERIMENTAL APPROACH

The production of the non-condensable gas was done through the application of a distillation process. A 100 gm sample (approximately 100 ml) of black liquor was introduced into a 250 ml flask and heat was applied. An asbestos heating basket plugged into a rheostat was used to insure even heating of the sample at a controlled rate. Heat was applied until initial boiling began and the rheostat was cut back to 65 percent of delivered power. The gases from the flask were then passed through a condensing unit which had tap water at approximately 68⁰ F passing through the inner liner. Figure 4 shows an illustration of the lab set up.

The collection of gas for the determination of the volume produced was accomplished by passing the gas into an ordinary sandwich baggie.(larger bags were used for the total volume determinations) The bags were then tied off and immersed in a container of water. The displaced water was then funneled into a graduated cylinder. The amount of water displaced

Figure 4: Lab Set up



PARTS IDENTIFICATION

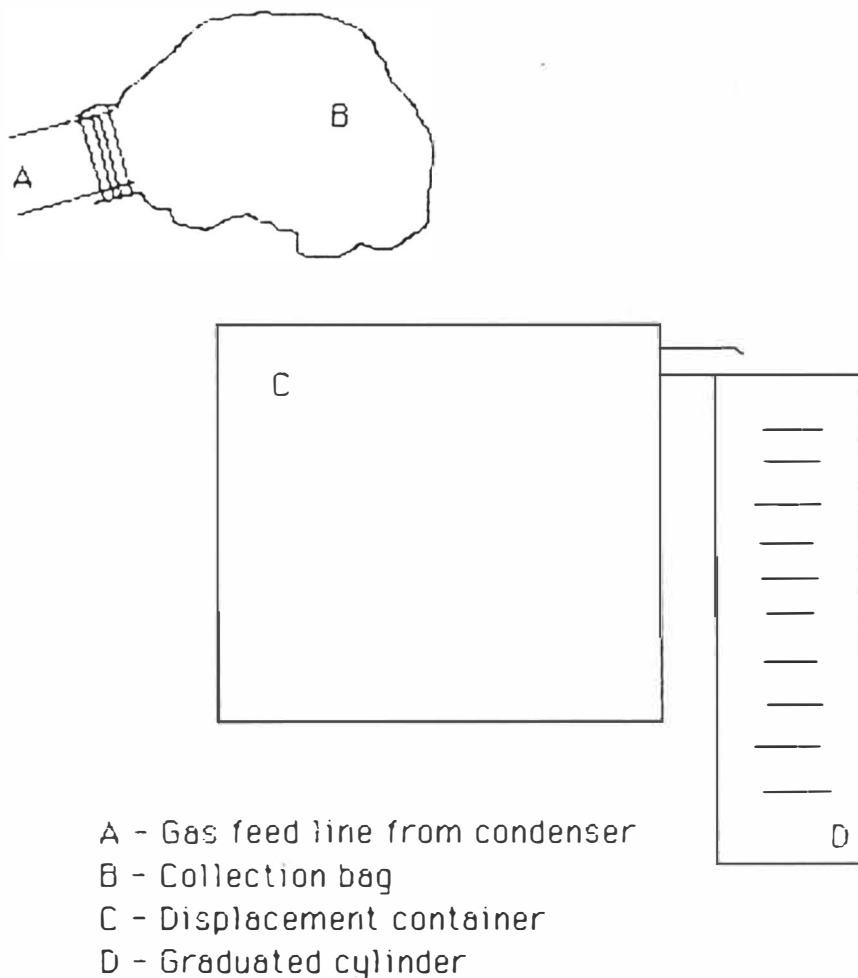
A - Mettler Balance
 B - Flask stand
 C - Heating Basket
 D - 250 ml Flask
 E - Fill Valve
 F - Condenser

G - Water inlet
 H - Water outlet
 I - NCG Flow
 J - Condensate Stopper
 K - Rheostat

and measured in the cylinder represented the gas amount in the bag.

Figure 5 shows this procedure.

Figure 5: Water Displacement Method



Careful consideration was given to the choosing of the displacing container. This was done to insure that a minimum amount of back pressure was put on the bag containing the gas. This procedure was taken to minimize the compressing of the gas in the bag since the compressibility of the gas was unknown.

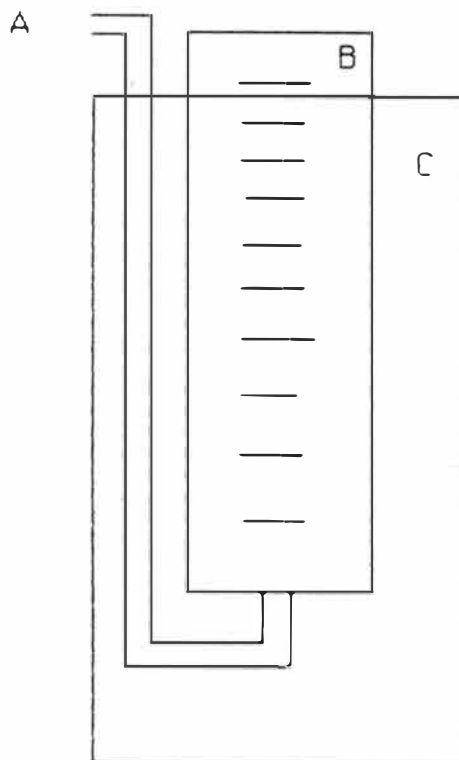
A number of other gas collection methods were utilized during the beginning stages of the project. These methods were:

APPROACH 1: (Figure 6)

The initial gas collection method utilized an inverted graduated cylinder immersed in a water bath. A tube from the condenser was then passed into the open space existing at the top of the inverted cylinder. Gas production would then force the water from the inverted cylinder into the surrounding water. This would allow for a direct gas measurement.

This method did produce some results but the actual utilization of the procedure proved to be cumbersome and time consuming. For these reasons it was not used.

Figure 6: Option 1 - Inverted Cylinder



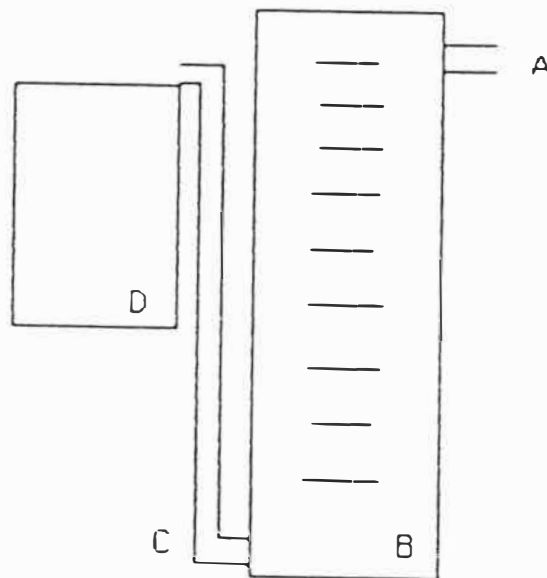
- A - Feed line from condenser
- B - Graduated cylinder
- C - Water bath

Approach 2: (Figure 7)

This gas collection procedure utilized a graduated cylinder with an upper gas inlet and lower water outlet. The cylinder was charged with ordinary tap water and balanced by an exit tube that contained a column of water of equal height. The gas entering the top would then displace the water and force it out the exit tube at the bottom. A direct measurement of the gas produced could then be taken.

This approach proposed many problems. The water inside the cylinder exerted a back pressure on the system causing an improper weight reading on the scale and percent solids determination during the test. This approach was scrapped.

Figure 7: Option 2 - Column water Displacement



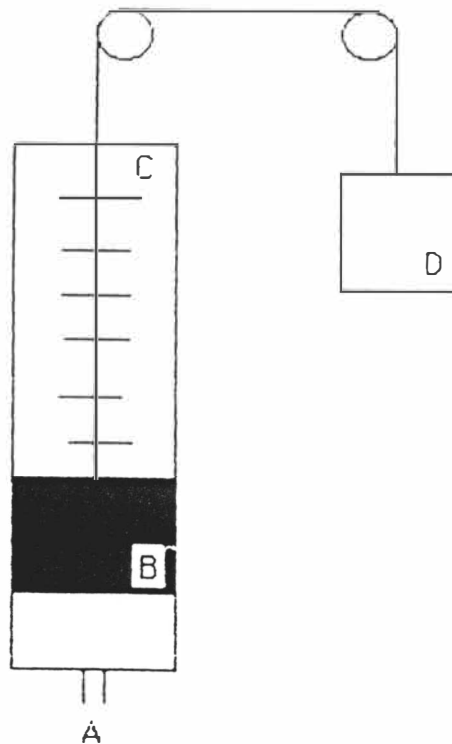
- A - Feed line from condenser
- B - Graduated cylinder
- C - Water exit line
- D - Displaced water collector

Approach 3: (Figure 8)

This approach consisted of the fabrication of a float type apparatus. A float was made out of styrofoam and wrapped in electricians tape. It was then inserted into a lubricated glass cylinder providing an air tight seal. A hole was then drilled into the bottom of the cylinder and a gas entry line was attached. A counter balance was fixed to the float to account for the friction necessary to overcome the force required to begin initial movement of the float. .

It was found that the pressure necessary to raise the float was too large. Attempts made to make the system more sensitive by increasing the counter balance weight caused uncontrolled moving of the float. Thus, this approach was not used.

Figure 8 - Option 3 - Float



- A - Gas feed line
- B - Float
- C - Graduated cylinder
- D - Counter weight

Readings for gas volume were taken at 50 and at 18-20 percent solids. The 50 percent solids reading represented the NCG developed in a six effect evaporator system and 18-20 percent solids represented NCG produced in the fifth and sixth effects. Development of NCG for the fifth and sixth effects is the same because Mead utilizes a split parallel feed to these two effects from the weak liquor storage tank. The initial condition of the black liquor used for testing can be found in Table 2.

Table 2

Initial Conditions

Sample #1 : Solids level - 15.5 %

 Ph level - 11.8

Sample # 2: Solids level - 14.2%

 Ph level - 12.1

Three data points were recorded at ph levels from 10 to 13.8 in increments of .2. Ph adjustment toward the basic scale was accomplished by a 30 percent solution of NaOH and the lower ph's were developed by addition of a weak Hcl solution. A model 7 Ph meter was used to monitor ph and a buffer solution for the 10 to 13 range was used to calibrate the meter after every sixth test. The raw data collected and averages can be found in Appendix I-IV.

It was necessary for the higher and lower ph's to recalculate the solids level. Adjustments back to the original solids levels were accomplished by addition of water or higher solids black liquor of the same sample. An example of this type of calculation and adjustment can be found in Table 3.

Table 3

Percent Solids Adjustment

Ph adjustment from 11.9 to 12.4

100 gms of liquor sample at 15.5% solids

Black liquor : 15.5 gms of solid + 84.5 gms of water

NaOH : 3.0 gms of solid + 7.0 gms of water

Current solids = $18.5 \text{ gms solid} / 110 \text{ gms tot.} = 16.8 \% \text{ solids}$

Adjustment: add 9.4 gms of water

Adjusted Solids: $18.5 \text{ gms solid} / 119.4 \text{ gms tot.} = 15.5 \% \text{ solids}$

DISCUSSION OF RESULTS

Experimental Results:

Table 4 consists of the results for total and 18-20 percent gas volume production for samples one and two.

Table 4

Tabulated Results

Volumetric values are in ml

Ph	<u>Sample 1 (15.5% Solids)</u>		<u>Sample 2 (14.2% Solids)</u>	
	Total Vol.	18-20% Vol	Total Vol.	18-20 Vol
10.0	638	263	684	279
10.2	636	264	676	272
10.4	636	262	682	264
10.6	632	255	681	259
10.8	630	249	675	257
11.0	624	244	667	254
11.2	619	244	650	248
11.4	621	242	641	247
11.6	611	239	636	243
11.8	598	238	622	241
12.0	603	241	611	241
12.2	586	235	597	238
12.4	583	235	589	238
12.6	586	229	582	222
12.8	572	227	577	223
13.0	548	218	564	219
13.2	534	219	560	213
13.4	528	208	560	215
13.6	529	209	558	207

Evaluation of Results:

Statistical analysis on ph as related to total gas produced and gas produced in the 18-20 percent range shows a very high negative

correlation. A Spearman Correlation test shows that for both samples the correlation was in excess of -0.990 . (Appendix V) This states that as pH is increased the volume of gas evolved is reduced with a very high correlation existing. Plots for the variation in pH and gas evolution can be found in Appendix VI.

A residual analysis on this data was also performed. The residual consists of a plot of the data points and determines how far these points vary from the equation of the line plotted in the regression analysis. The regression plots are the ones found in Appendix VI and the residual plots are found in Appendix VII. A point on the residual plot that is closer to the zero line represents a point that is more highly correlated to the independent variable (pH) than one that is farther away. Close examination of the residual plots for both samples at the 50 percent level and 18-20 percent show that there is a higher correlation between pH and gas evolution in the 11.5 to 12.5 pH range.

Lab To Industry Comparison:

The volume of gas determined from the lab can be converted to Mead's production level by the use of the following equation:

$$a. \frac{1500 \text{ gal}}{\text{min}} \times \frac{\text{liter}}{.2642 \text{ gal}} \times \frac{1000 \text{ ml}}{\text{liter}} \times \frac{\text{Quantity of Gas Produced}}{100 \text{ ml of black liq sample}} = \frac{\text{ml gas}}{\text{min}}$$

$$b. \frac{\text{ml gas}}{\text{min}} \times \frac{\text{liter}}{1000 \text{ ml}} \times \frac{1 \text{ ft}^3}{28.32 \text{ liters}} = \text{Gas produced ft}^3 \text{ per minute}$$

* This is the flow of weak black liquor to the evaporator system at Mead. This flow is split and 750 gpm flow to the six effect and 750 gpm flow to the fifth effect.

The total gas volume for the evaporator system at Mead determined from the experimental results ranges from 1054-1371 cubic feet per minute of non-condensable gases. It was found that gases from the fifth and sixth effect (the 18-20% gas volume determination) combined ranges from 417-559 cubic feet per minute. The gas volume from the initial feed of black liquor into the fifth and sixth effects represents 39-40 percent of the total volume of non-condensable gases evolved in the entire evaporator system at Mead.

CONCLUSION/RECOMMENDATIONS

From the data analyzed in the thesis it can be said that ph variation has a definite influence on the production of non-condensable gases. Higher ph black liquors produce less non-condensable gases, but adjustment of liquors outside of a 13 ph range have a less correlated effect on reducing the

volumetric production of gas than a pH adjustment between 11.5 -12.5. It has also been determined that a large portion of the non-condensable gases evolved in an evaporator system are produced in the fifth and sixth effects, approximately 40 percent of the total.

Further study on black liquor non-condensable gas evolution should involve a larger sample size than what was evaluated in this thesis. This would allow one to determine more specifically the range at which caustic adjustment begins to affect gas evolution. The sample size considered for this thesis was large enough to determine general tendencies but for higher statistical verification more data points should have been considered. Time constraints limited the implication of this.

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APPENDIX I

Total Volume Results for 15.5 % Solids Sample

Values read in ml

Ph	Sample #1	Sample #2	Sample #3	Average value
10.0	644	634	636	638
10.2	633	637	640	636
10.4	640	634	635	636
10.6	635	630	631	632
10.8	636	628	626	630
11.0	632	622	618	624
11.2	626	614	617	619
11.4	617	624	622	621
11.6	615	608	612	611
11.8	591	607	596	598
12.0	600	601	608	603
12.2	590	584	585	586
12.4	580	579	584	583
12.6	588	582	590	586
12.8	573	568	576	572
13.0	548	542	555	548
13.2	541	529	532	534
13.4	538	542	537	528
13.6	532	528	525	529

APPENDIX II

18-20% Gas Volume for 15.5% Solids Sample

Values read in ml

Ph	Sample #1	Sample #2	Sample #3	Average value
10.0	268	260	262	263
10.2	270	265	258	264
10.4	269	254	265	262
10.6	255	259	250	255
10.8	246	251	252	249
11.0	248	242	244	244
11.2	247	245	240	244
11.4	243	241	244	242
11.6	242	237	239	239
11.8	238	244	234	238
12.0	245	243	236	241
12.2	232	240	235	235
12.4	238	234	230	234
12.6	228	232	228	229
12.8	226	231	224	227
13.0	216	222	215	218
13.2	223	221	213	219
13.4	211	207	205	208
13.6	210	205	213	209

APPENDIX III

Total Volume Results for 14.2% Solids Sample

Values read in ml

Ph	Sample #1	Sample #2	Sample #3	Average value
10.0	689	681	684	684
10.2	683	675	671	676
10.4	684	681	683	682
10.6	683	677	684	681
10.8	672	678	674	675
11.0	670	664	667	667
11.2	654	648	649	650
11.4	648	638	636	641
11.6	638	634	637	636
11.8	625	617	627	622
12.0	618	606	610	611
12.2	602	590	600	597
12.4	590	580	593	589
12.6	585	579	584	582
12.8	578	574	578	577
13.0	562	568	563	564
13.2	557	563	561	560
13.4	559	565	557	560
13.6	556	562	558	558

APPENDIX IV

18-20% Gas Volume for 14.2% Solids Sample

Values read in ml

Ph	Sample #1	Sample #2	Sample #3	Average value
10.0	283	278	276	279
10.2	265	273	277	272
10.4	270	263	258	264
10.6	265	257	255	259
10.8	261	254	256	257
11.0	252	253	258	254
11.2	247	251	245	248
11.4	253	247	243	247
11.6	242	246	240	243
11.8	245	235	239	241
12.0	237	240	245	241
12.2	238	234	241	238
12.4	232	229	227	229
12.6	225	222	218	222
12.8	220	227	223	223
13.0	219	224	215	219
13.2	214	209	216	213
13.4	220	213	212	215
13.6	209	202	214	207

APPENDIX V

Spearman Correlation

15.5% Black liquor Sample: Total Vol. vs. Ph

Variable: ph

Variable: Total Volume

Paired Observations: 20

Test: Spearman Correlation

Statistic: -0.99248

Significance: 0.000

15.5% Black liquor Sample: 18-20% Gas Vol. vs. Ph

Variable: ph

Variable: 18-20% Vol.

Paired Observations: 20

Test: Spearman Correlation

Statistic: -0.98947

Significance: 0.000

APPENDIX V

Con't

14.2% Black liquor Sample: Total Vol. vs. Ph

Variable: ph

Variable: Tot Vol 2

Paired Observations: 20

Test: Spearman Correlation

Statistic: -0.99511

Significance: 0.000

14.2% Black liquor Sample: 18-20% Gas Vol. vs. Ph

Variable: ph

Variable: 18-20% vol 2

Paired Observations: 20

Test: Spearman Correlation

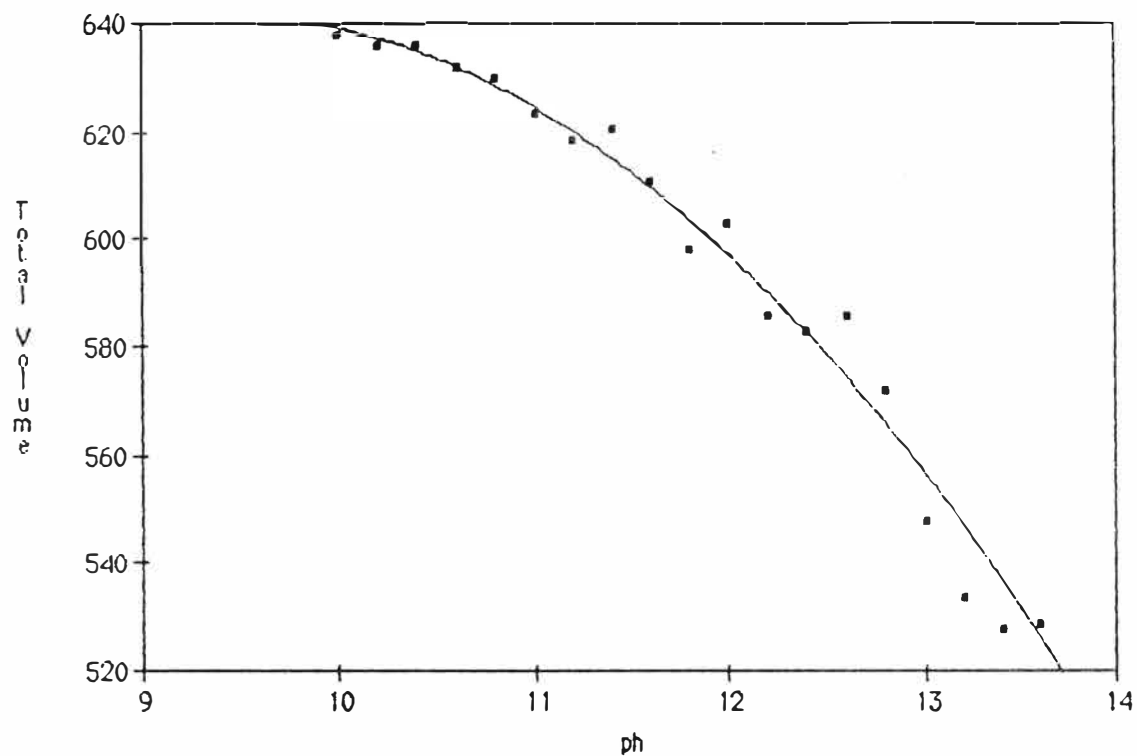
Statistic: -0.99511

Significance: 0.000

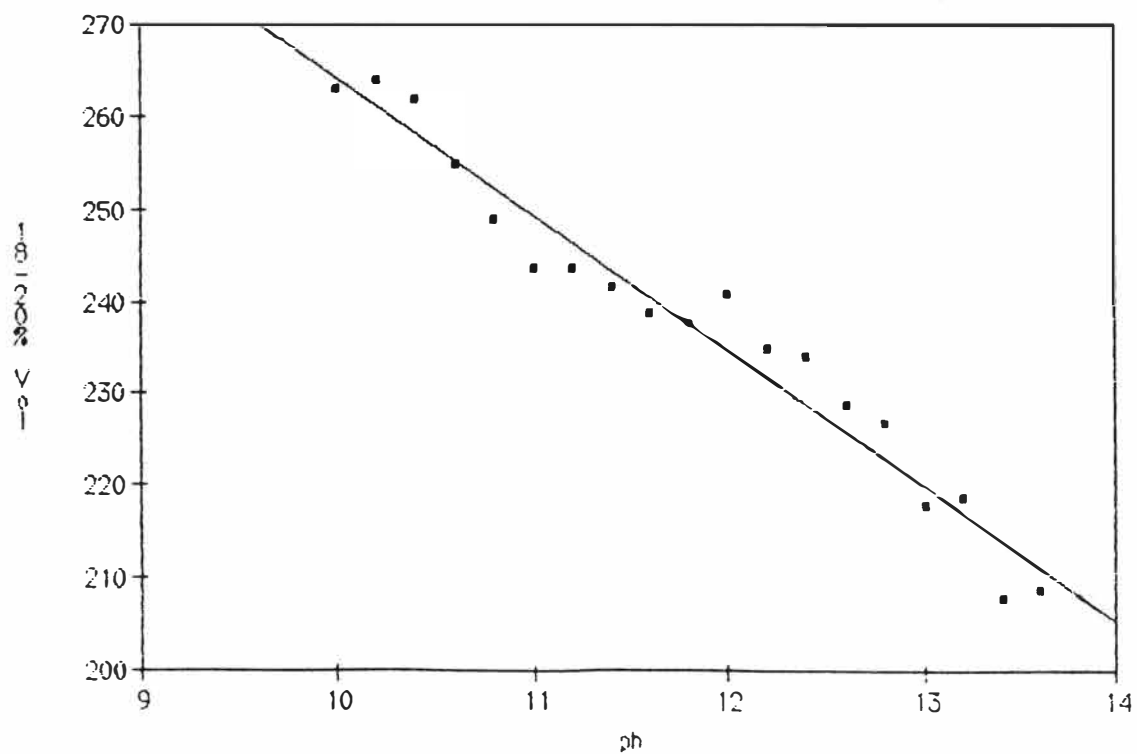
APPENDIX VI

Regression Plots

15.5% Black liquor Sample: Total Vol. vs. Ph



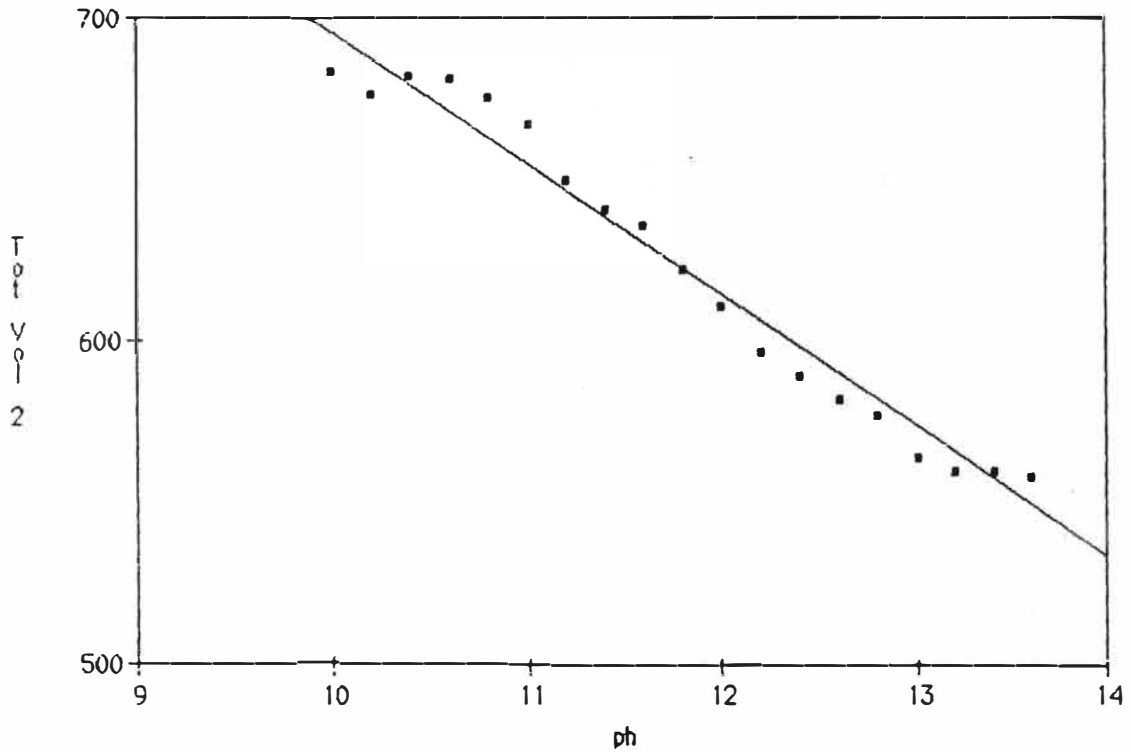
15.5% Black liquor Sample: 18-20% Gas Vol. vs. Ph



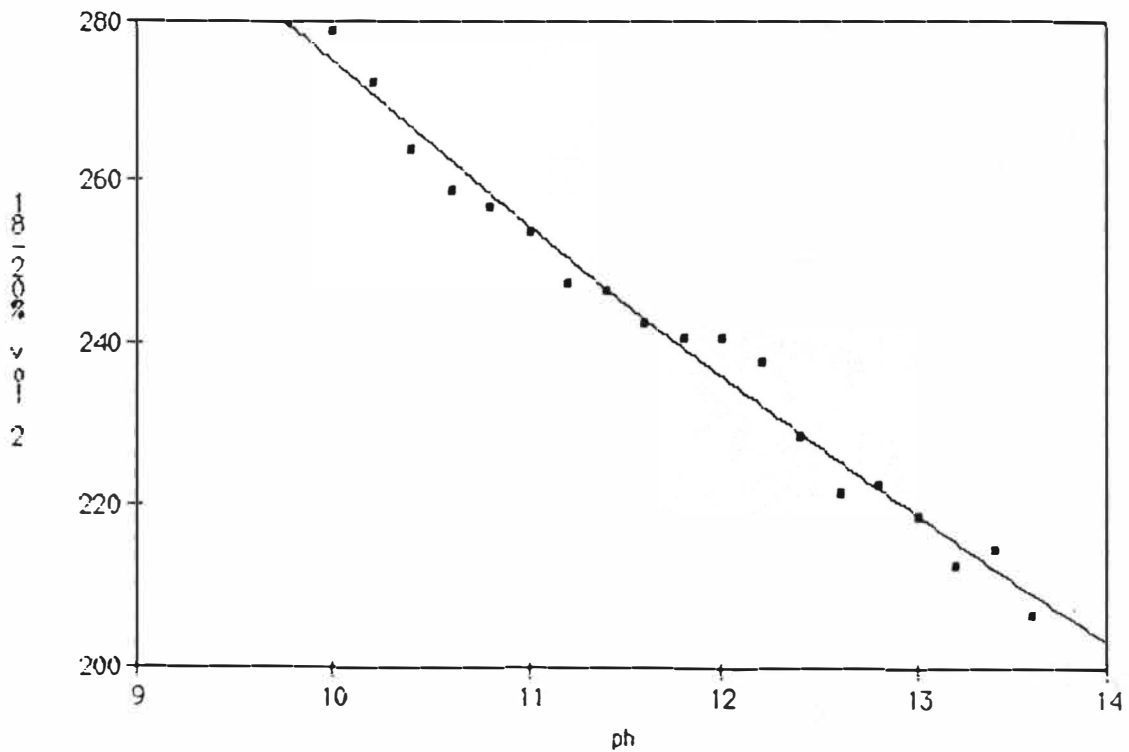
APPENDIX VI

Con't

14.2% Black liquor Sample: Total Vol. vs. Ph



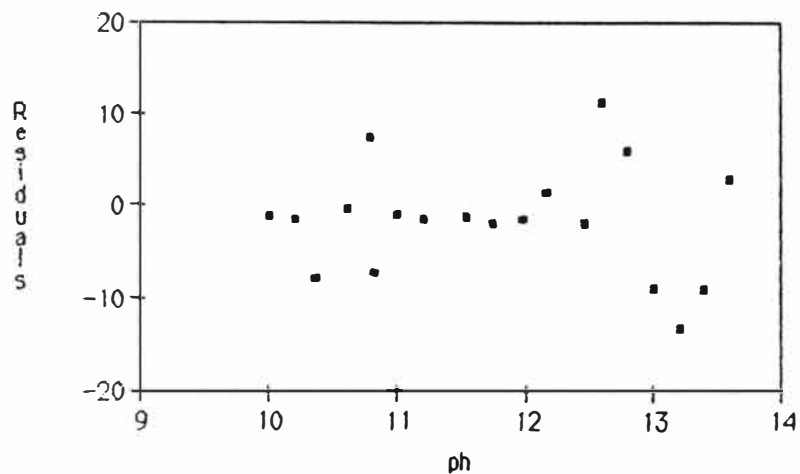
14.2% Black liquor Sample: 18-20% Gas Vol. vs. Ph



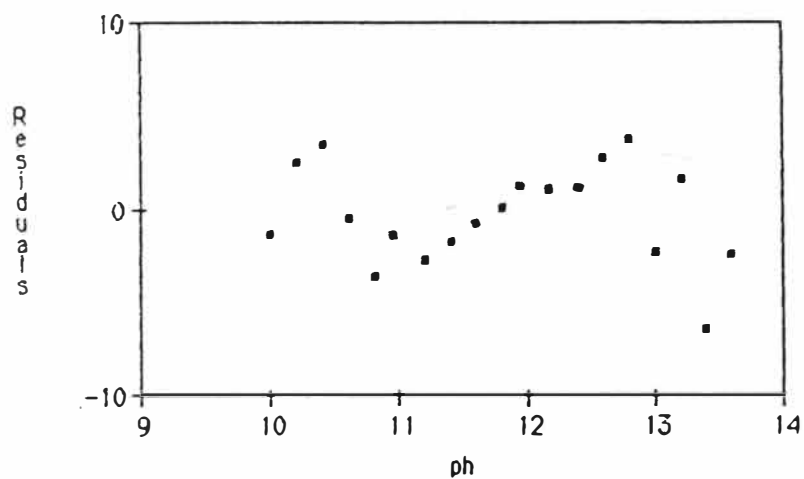
APPENDIX VII

Residual Plots

15.5% Black liquor Sample: Total Vol. vs. Ph



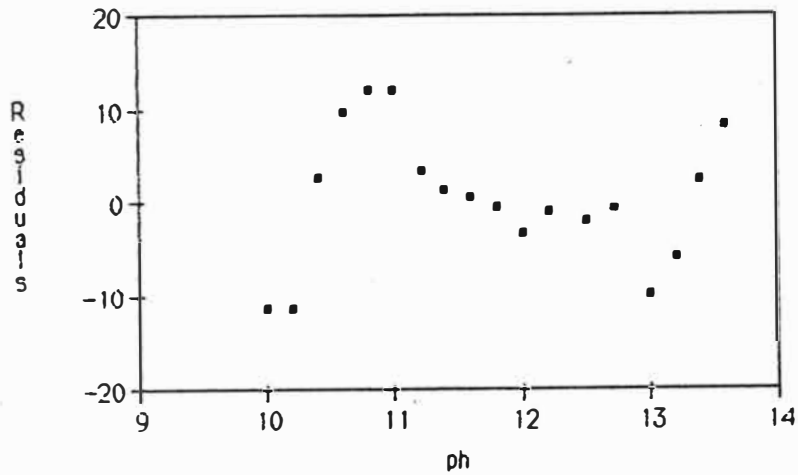
15.5% Black liquor Sample: 18-20% Gas Vol. vs. Ph



APPENDIX VII

Con't

14.2% Black liquor Sample: Total Vol. vs. Ph



14.2% Black liquor Sample: 18-20% Gas Vol. vs. Ph

