

PRODUCTION OF SODIUM CYANIDE

A Thesis

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by

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PRODUCTION OF SODIUM CYANIDE

Approved:

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## TABLE OF CONTENTS

CHAPTER	PAGE
I. INTRODUCTION . . . . .	1
II. DESCRIPTION OF APPARATUS . . . . .	8
III. REACTING MATERIALS . . . . .	9
IV. EXPERIMENTAL PROCEDURE . . . . .	10
V. ANALYSES . . . . .	11
VI. DISCUSSION OF RESULTS. . . . .	12
VII. RECOMMENDATIONS . . . . .	13
BIBLIOGRAPHY . . . . .	14
APPENDIX . . . . .	16

LIST OF TABLES

	Page
Table I      Data Sheet - Run No. 12 .....	17
Table II     Data Sheet - Run No. 13 .....	19
Table III    Data Sheet - Run No. 14 .....	21

## LIST OF FIGURES

		Page
Figure I	Flow Diagram .....	6
Figure II	Diagram of Reactor .....	7

## ABSTRACT

A preliminary study of the adaptability of the Bucher process ( $\text{Na}_2\text{CO}_3 + 4 \text{C} + \text{N}_2 = 2 \text{NaCN} + 3 \text{CO}$ ) to the fluidized bed technique was made.

Fourteen runs were attempted, of which eleven resulted in failure due to operational difficulties. The major difficulties experienced were in introducing the charge into the reactor, maintaining smooth fluidization, obtaining a significant reaction time, and maintaining isothermal conditions. Corrosion of the reactor added to the operational difficulties.

A technique of operation was developed such that significant data were obtained for the last three runs. The maximum conversion to cyanide was 19.9% of the sodium charged. From 3% to 6% of the sodium charged was converted to sodium ferrocyanide.

A favorable material balance was obtained for the last run in which 96.6% of the sodium charged was accounted for; 12.0% of this appeared as sodium cyanide and 4.1% as sodium ferrocyanide.

## PRODUCTION OF SODIUM CYANIDE

### I INTRODUCTION

#### Historical:

In 1838 Lewis Thompson was awarded the Gold Iris Medal of the Society of Arts for his work on Prussian Blue. By heating potash, coke, and iron turnings in an open crucible, he obtained cyanide which he converted into Prussian Blue. Thompson's works<sup>1</sup>, which were published in 1839, led to active discussion and investigation.

In 1843 a British patent<sup>2</sup> was taken out for the production of cyanide from atmospheric nitrogen. A commercial plant was set up at New Castle-on-Tyne. This plant had a capacity of one ton of potassium cyanide per day. Failure to appreciate the importance of iron as a catalyst necessitated operating at white heat and resulted in poor yields, slow action, and fast deterioration of the reactors. The process was abandoned in 1847, thus ending the first endeavor to fix atmospheric nitrogen on a commercial scale. Numerous other attempts were made during the latter part of the nineteenth century to put the process on a commercial basis, but all were without success.

The great need for fixed nitrogen which arose from the United State's efforts in the First World War led to much investigation in this

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<sup>1</sup> Lewis Thompson, "Improvements in the manufacture of Prussian Blue," Dingler's Polytechnisches Journal, 73, 281 (1839).

<sup>2</sup> British Patent, No. 9985 (1843).



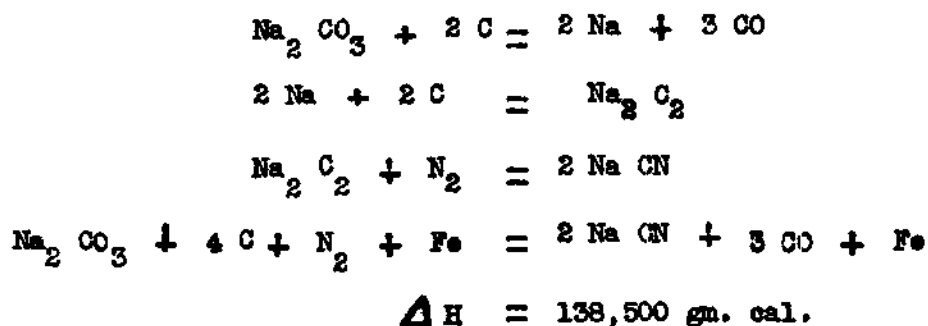
field. In 1917 James E. Bucher proposed a process for the production of sodium cyanide from sodium carbonate, nitrogen, and carbon with iron as a catalyst.<sup>3</sup>

The Bucher process, as it is called, has the following advantages: the reacting materials are cheap and abundant, the temperature is low enough for fuel heating to be used, and the apparatus is simple and of cheap construction.

The main difficulties of the Bucher process are of an engineering character and arise from the necessity of heating indirectly a solid of low heat capacity in which an endothermic reaction is taking place.

#### Theoretical Considerations:

The mechanism of the reaction as proposed by Guernsey is as follows:<sup>4</sup>




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<sup>3</sup> J. E. Bucher, "The Fixation of Nitrogen", *Industrial and Engineering Chemistry*, 9, 232-53 (1917).

<sup>4</sup> E. W. Guernsey, "The Mechanism of the Fixation of Nitrogen as Sodium Cyanide", *Industrial and Engineering Chemistry*, 18, 243-8 (1926).

Iron catalyzes the absorption of nitrogen by sodium carbide. It is also probable that the reduction of sodium carbonate by carbon is facilitated by iron.

Sodium carbide is not stable as a condensed phase, but it exists in a small concentration in the vapor phase in equilibrium with solid carbon and sodium vapor.<sup>5</sup>

The thermal stability of sodium cyanide itself must be considered. It has been shown that the vapor in equilibrium with sodium cyanide is not pure sodium cyanide but also contains sodium, sodium carbide, and nitrogen.<sup>6</sup> Thus the distillation of sodium cyanide from a charge, as has been suggested,<sup>7</sup> would result in decomposition of the cyanide. The fact that sodium cyanide is decomposed by heat suggests that a charge should not be treated any longer than necessary.

The equilibrium conversion of sodium carbonate to cyanide is determined by the temperature and the partial pressures of nitrogen and carbon monoxide. Guernsey gives the following relation between X (the ratio CN/Na) and the partial pressures of nitrogen and carbon monoxide:<sup>8</sup>

$$\log \left( \frac{4X^2}{1-X^2} \cdot \frac{P_{CO}^3}{P_{N_2}} \right) = \frac{K_1 + K_2}{T}$$

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<sup>5</sup>E. W. Guernsey and M. S. Sherman, "Thermal Dissociation of Sodium Carbide", Journal of the American Chemical Society, 48, 140-5 (1926).

<sup>6</sup>E. W. Guernsey and M. S. Sherman, "Thermal Dissociation of Sodium Cyanide", Journal of the American Chemical Society, 48, 695-704 (1926).

<sup>7</sup>J. E. Bucher, Industrial and Engineering Chemistry, 9, 233-53 (1917).

<sup>8</sup>E. W. Guernsey, "Some Factors Affecting the Fixation of Nitrogen as Cyanide", Industrial and Engineering Chemistry, 18, 243-8 (1926).

By fitting this equation to the data of Ingold and Wilson,<sup>9</sup> Guernsey found the values of the constants to be  $K_1 = 24.87$  and  $K_2 = 31.738$ .

#### Practical Considerations:

The preferred operating temperature is between  $1000^{\circ}\text{C}$  and  $1050^{\circ}\text{C}$ .<sup>10</sup> Higher temperatures would result in decomposition of the sodium cyanide. On the other hand, lower temperatures would shift the equilibrium unfavorably and lower the rate of reaction.

As has been stated, the main difficulty of the Bucher process arises from the necessity of heating indirectly a solid of low heat capacity in which an endothermic reaction is taking place. It is thought that the fluidized bed technique might alleviate this problem.

#### Fluidization:

A fluidized mass of solid particles has been defined as one which exhibits the mobility and hydrostatic pressure characteristic of a fluid.<sup>11</sup> Thus the pressure drop across a fluidized bed will be approximately equal to the weight of the bed divided by the cross-sectional area; further evidence of smooth fluidization is a rhythmic jiggling of the pressure drop across the bed.

Channeling is the establishment of flow paths in the bed through

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<sup>9</sup> C. K. Ingold and Daniel Wilson, "The Reversibility of the Reaction between Nitrogen, Carbon, and Sodium Carbonate", *Journal of the Chemical Society*, (London), 121, 2278-84 (1922).

<sup>10</sup> Harry A. Curtis, "Fixed Nitrogen", A. C. S. Monograph No. 59, The Chemical Catalog Company, (1932).

<sup>11</sup> "Fluidization Nomenclature and Symbols", *Chemical Engineering*, 56, No. 5, p. 230 (1949).

which a disproportionate quantity of the fluid passes. The pressure drop across a bed in which channeling is occurring will be lower than that predicted from the above.

Slugging is a condition in which bubbles of the supporting fluid grow to the dimensions of the containing vessel. Thus portions of the bed which are trapped between bubbles rise in a piston like manner. Slugging causes erratic variations in the pressure drop across the bed.

The proper size distribution, i. e. the right proportion of fines and large particles, is necessary to good fluidization. With too many fines, particles tend to adhere to one another and channeling occurs. Slugging tends to occur in a bed which contains too few fines.

The advantages of carrying out the Bucher process in a fluidized bed are excellent control and uniformity of temperature, high over-all heat transfer, and excellent gaseous diffusion. Fluidization has the disadvantage of eliminating the possibility of countercurrent flow. Enough excess carbon must be added to keep the charge from sticking, which would disrupt smooth fluidization. Of course this increases the bulk of material to be handled.

LEGEND:      FIGURE NO. I

- A -        Rotameter
- B -        Manometer
- C -        Charging Chamber
- D -        Reactor
- E -        Product Receiver
- F -        Potentiometer
- G -        Cyclone
- H -        Water Bubbler
- I -        Glass Wool Plug

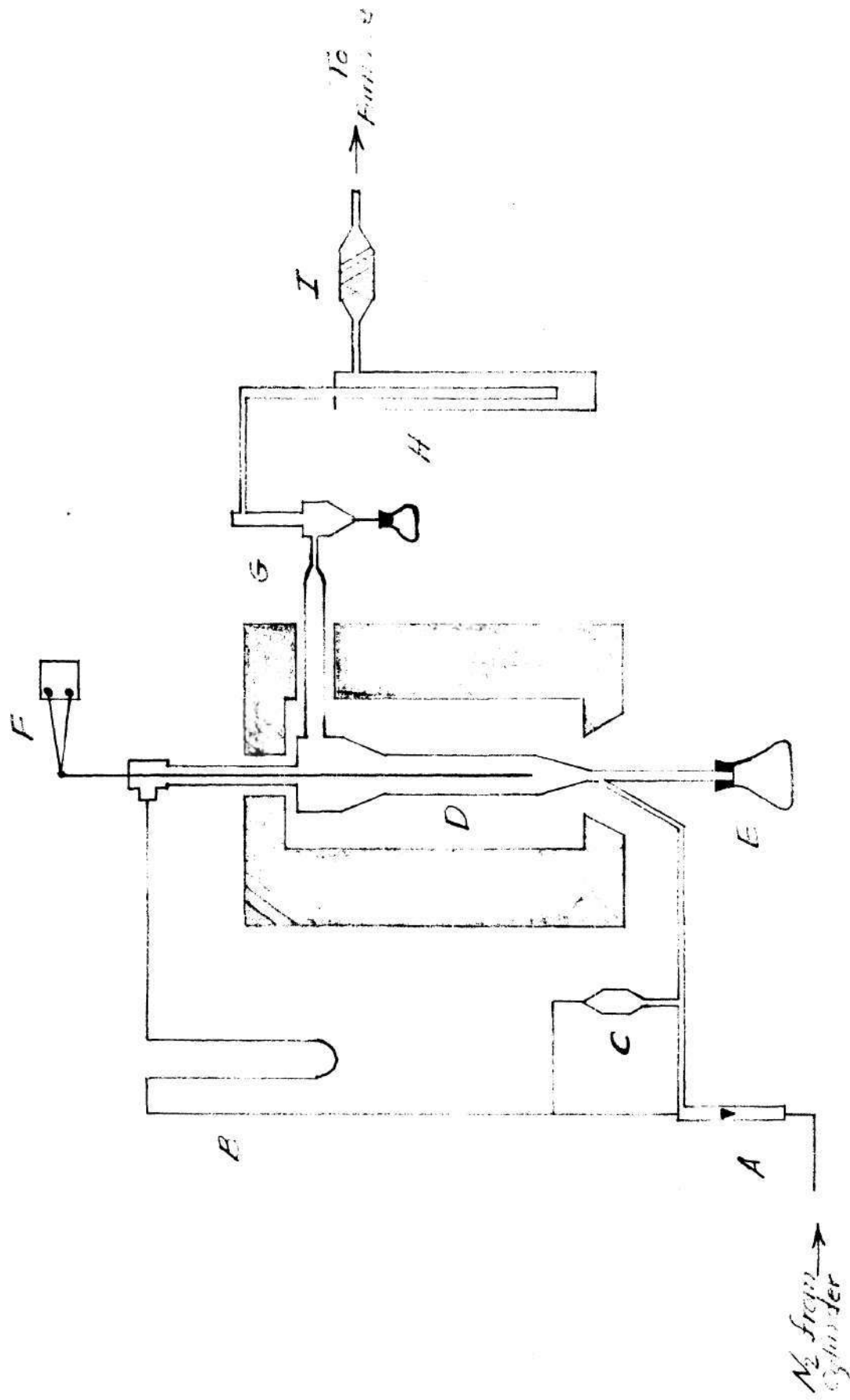
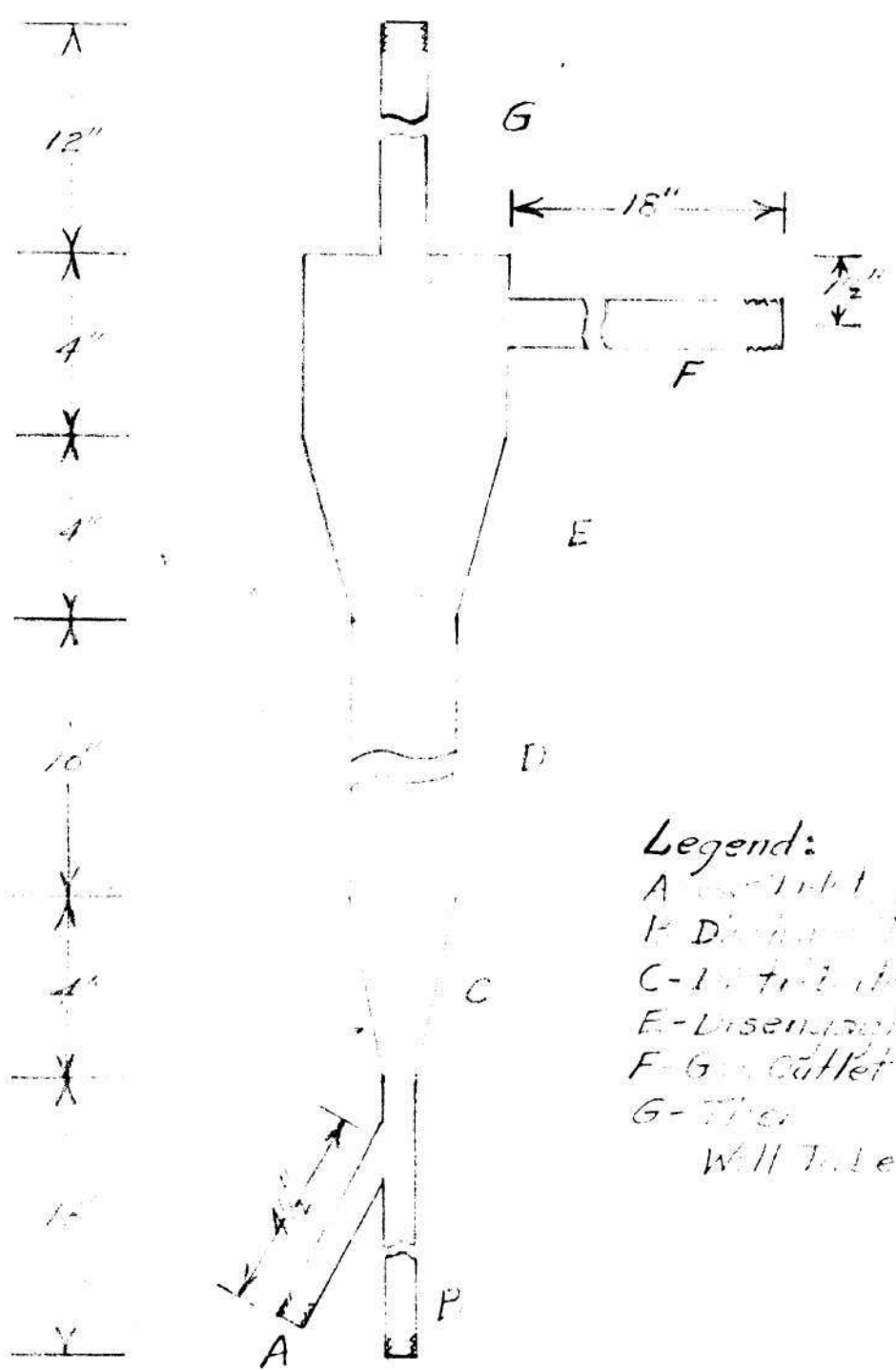


Figure I  
Flow Diagram



Legend:  
 A - Inlet, 3/4"  
 B - Discharge Tube, 1"  
 C - Diffuser section  
 E - Discharge, 5" dia  
 F - Gas Inlet, 3/4"  
 G - Filter  
 Will take 3/4"

Figure II

Diagram of Reactor

## II DESCRIPTION OF APPARATUS

Figure 1, page 6, is a flow diagram of the process. Cylinder nitrogen throttled by a diaphragm valve was metered by a Fisher and Porter No. 2A rotameter.

The charge was introduced into the nitrogen stream before it entered the reactor from a charging chamber mounted above the gas inlet pipe. A rubber hose connected from the rotameter to the top of the charging chamber equalized the pressure thus facilitating dropping the charge into the nitrogen stream.

The reactor (Figure II, page 7) was made of Schedule 40 type 304 stainless steel pipe. The charge, suspended in the nitrogen stream, was blown into the bottom of the reactor where it built up a bed. The conical distributor section at the bottom distributed the gas flow. Particles which were blown out of the bed fell back due to the decreased velocity in the disengaging section at the top. The charge was dropped from the reactor through the discharge tube by decreasing the nitrogen flow.

The pressure drop across the bed was measured by a water manometer. The temperature was measured by a chromel-alumel thermocouple which was enclosed in a 5/16 inch stainless steel tube and was passed through a packing gland at the top.

Entrained particles in the exit gases were separated in a cyclone. After the cyclone the gases were passed through a water bubbler to remove the finer particles and vapors. The gases were then passed through a glass wool plug and discharged into the furnace.

The furnace was made of Johns Mansville "Fireblok" and was heated with four locomotive type gas burners.



### III REACTING MATERIALS

Water pumped nitrogen bottled by the National Cylinder Gas Company supplied nitrogen for the reaction and supported the fluidized bed. The company claimed better than 99.9 per cent purity.

Anhydrous sodium carbonate, Baker's G. P. reagent, was used. The sodium carbonate was sieved in a rotap. The size range which passed a 100 mesh screen and was retained on a 270 mesh screen was found to fluidize best. The sodium carbonate as mixed in the charge titrated to 93 per cent purity on a basis of total basicity (titrated to a bromo cresol blue end point).

Wood Charcoal with a size range which passed a 45 mesh screen and was retained on a 100 mesh screen was used as a source of carbon. It is believed that the charcoal volatilized to a small degree when it was exposed to the temperatures of the reaction.

The iron catalyst was supplied as powdered reduced metallic iron.

The charge was mixed dry; enough excess charcoal was added to prevent sticking of the charge, which would have interfered with smooth fluidization.

## IV EXPERIMENTAL PROCEDURE

With the apparatus set up as described in Chapter II, the furnace and reactor were heated to 100°C above the temperature at which the reaction was to be carried out. The desired gas flow was then established and the temperature of the gas stream was checked.

The charge was introduced into the inlet gas stream at a rate such as not to form slugs that would blow over into the cyclone separator. Considerable difficulty was experienced in getting a bed of known quantity into the reactor and yet obtaining a significant run time.

The gas velocity had to be great enough to keep the bed from falling through the bottom discharge tube and not in excess of that which would blow the charge into the cyclone separator. The flow was adjusted within this range so as to maintain fluidization in the reactor as evidenced by a rhythmic jiggling of the manometer which measured the pressure drop across the bed. Difficulty was experienced in maintaining these conditions, particularly during the charging period.

To compensate for changes in heat requirements, adjustments were made on the burners in an effort to maintain an isothermal reaction. The art of maintaining a constant temperature was not perfected during this work.

At the end of the run the burners were turned off and the exhaust fan which drew air through the furnace was started so as to cool the system as rapidly as possible. The gas velocity was increased and decreased in an effort to drop the bed through the discharge tube. It was necessary to use the thermocouple well to facilitate dropping the bed from the reactor.

## V ANALYSES

The products obtained from the bottom discharge tube, cyclone, water bubbler, and reactor washings were analyzed separately for total basicity, cyanide, and sodium ferrocyanide.

The total basicity was determined by titrating an aliquot of the sample to a bromo cresol blue end point with hydrochloric acid. The total basicity was attributed to the sodium which appeared as cyanide, unreacted carbonate, and metallic sodium. The validity of this analysis was substantiated by titrating known mixtures of cyanide, carbonate, and ferrocyanide.

The cyanide was determined by the Lundell-Bridgman Method.<sup>12</sup> An aliquot of the sample was neutralized with hydrochloric acid and then made slightly alkaline with ammonium hydroxide. A dimethyl gloxine indicator was added and the solution was titrated with standard nickel sulfate solution until a permanent red precipitant formed.

The sodium ferrocyanide was determined gravimetrically by precipitating as ferri ferrocyanide. An aliquot of the sample was acidified with hydrochloric acid and then treated with an excess of ferric chloride.

The total equivalents of sodium were taken as the sum of that attributed to the total basicity and that attributed to the sodium ferrocyanide.

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<sup>12</sup>

G. E. F. Lundell and J. A. Bridgman, "A New Method for the Determination of Hydrocyanic Acid and the Alkali Cyanides", *Industrial and Engineering Chemistry*, 6 554-6 (1914).

## VI DISCUSSION OF RESULTS

Fourteen runs were attempted, of which eleven resulted in failure due to operational difficulties. The major difficulties experienced were in introducing the charge into the reactor, maintaining smooth fluidization, obtaining a significant reaction time, and maintaining isothermal conditions. Corrosion of the reactor added to the operational difficulties.

A technique of operation was developed such that significant data were obtained for the last three runs. These data are presented in the appendix on pages 17-22.

The fact that from a quarter to a third of the run time was spent in charging and that the retention time of that part of the bed which was blown from the reactor was unknown complicates the calculation of a significant run time. Because of the temperature variation with time (800-1000°C), no definite temperature can be assigned to a run.

Failure to obtain a perfect sodium balance may be attributed to small losses of sodium from the system and the reaction of sodium with impurities in the reactants. A trace of hydrogen sulfide was noticed when the product was acidified, indicating that sulfur from some source had been evolved during the reaction.

Metallic sodium was formed during the reaction as evidenced by its burning when the product was exposed to air. Reaction of sodium with water in the bubbler accounts for the hydrogen present in the exit gases.

## VII RECOMMENDATIONS

The following recommendations are offered for guidance in any further work on this problem:

1. A more expeditious method should be devised for charging the reactor.
2. The exit gas stream should be metered and should be analyzed more often so as to follow the kinetics of the reaction.
3. A method should be devised for keeping a more nearly constant temperature.
4. The apparatus should be constructed of low carbon steel because of its ease of fabrication over stainless steel.
5. The possibilities of carrying out the reaction in two stages as suggested by Guernsey<sup>13</sup> should be investigated.

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<sup>13</sup> Harry A. Curtis, "Fixed Nitrogen", A. C. S. Monograph, No. 59, the Chemical Catalog Company, (1932).

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**APPENDIX**



TABLE I

Data Sheet - Run No. 12

Charge:		Wt.	Size
Charcoal		150 gms.	45-100 mesh
Sodium Carbonate		75 " (93% Pure)	100-270 "
Iron		25 "	powder

Time (Sec.)	Temp. (°C)	Nitrogen Flow (cfm-68°F, 1 atm)	Press. Drop (in. water)	Remarks
0	992			
30	994	0.50	0.2	
60	982	"	1.2	
90	856	"	3.0	
120	788	"	1.4	
150	808	0.66	0.8	
180	822	"	2.0	
210	802	"	1.6	
240	792	"	3.0	Tended to Slug
270	812	"	4.0	
300	822	"	2.0	
330	846	"	2.8	
360	846	"	2.4	
390	854	"	4.4	
420	832	"	5.0	
450	844	"	3.2	
465	874	"	3.0	
530	912	"	3.0	Feed Complete
560	932	"	3.0	
590	944	"	2.8	
620	954	"	2.6	
650	956	"	2.8	
700	964	"	2.8	
800	966	"	2.6	
900	974	"	2.8	
1000	982	"	2.6	
1100	982	"	2.6	
1200	982	"	2.6	
1300	986	"	2.4	
1400	986	"	2.6	
1500	990	"	2.6	

TABLE I - Continued

Time (Sec.)	Temp. (°C)	Nitrogen Flow (cfm-68°F, 1 atm)	Press. Drop (in. water)	Remarks
1600	992	0.66	2.4	
1700	998	"	2.4	
1800	1000	"	2.6	
1900	1002	"	2.6	Turned off heat
2000	876	---	---	
2100	740	---	---	
2200	650	---	---	
2300	522	---	---	
2400	490	---	---	
2500	406	---	---	
2600	386	---	---	

## Analyses:

Source	Basicity	Sodium Cyanide ( gram equivalents of sodium)	Sodium Ferrocyanide
Product from bottom of Reactor- Cyclone	0.506	0.124	0.011
Bubbler	0.245	0.031	0.011
Reactor Washings	0.079	0.029	Negligible
	<u>0.275</u>	<u>0.007</u>	<u>0.013</u>
	1.105	0.191	0.035
Total equivalents of sodium accounted for -			1.14
Percent of sodium charged accounted for -			86.7 %
Percent of sodium charged converted to sodium cyanide			14.5 %
Percent of sodium charged converted to sodium ferrocyanide			2.7 %

TABLE II

Data Sheet - Run No. 13

Charge:		Wt.	Size
Charcoal		150 gms.	45-100 mesh
Sodium Carbonate		75 " (93% pure)	100-270 "
Iron		25 "	powder

Time (Sec.)	Temp. (°C)	Nitrogen Flow (cfm-68°F, 1 atm)	Press. Drop (in. water)	Remarks
—	1008	—	0.2	
0	998	0.66	0.6	
30	974	"	1.4	
60	878	"	0.6	
90	796	"	1.4	
120	792	"	2.0	
150	822	"	1.0	
180	832	"	1.4	
210	846	"	1.4	
240	844	"	3.8	
270	842	"	4.0	
300	846	"	3.0	
330	862	"	3.0	
360	862	"	4.8	Feed Complete
390	866	"	3.2	
420	894	"	3.0	
450	916	"	2.8	
480	932	"	2.8	
510	944	"	3.0	
540	952	"	3.0	
570	962	"	3.0	
600	966	"	2.8	
700	982	"	2.8	
800	998	"	2.8	
1000	998	"	2.6	
1200	992	"	2.4	
1400	994	"	2.2	
1600	994	"	2.2	
1800	992	"	2.2	Turned off heat.
1900	896	—	—	
2000	730	—	—	
2100	588	—	—	
2200	486	—	—	
2300	438	—	—	

TABLE II - Continued

## Analyses:

Source	Basicity ( gram equivalents of Sodium)	Sodium Cyanide	Sodium Ferrocyanoide
Product from bottom of Reactor-	0.577	0.186	0.033
Cyclone	0.186	0.020	0.016
Bubbler	0.083	0.034	0.013
Reactor Washings	<u>0.304</u>	<u>0.021</u>	<u>0.019</u>
	1.150	0.261	0.081
Total equivalents of sodium accounted for -			1.23
Percent of sodium charged accounted for -			93.5 %
Percent of sodium charged converted to sodium cyanide-			19.9 %
Percent of sodium charged converted to sodium ferrocyanide			6.2 %

TABLE III

Data Sheet - Run No. 14

Charge:		Wt.	Size
Charcoal		150 gms.	45-100 mesh
Sodium Carbonate		75 " (93% Pure)	100-270 "
Iron		25 "	powder

Time (Sec.)	Temp. (°C)	Nitrogen Flow (cfm-68°F, 1 atm)	Press. Drop (in. water)	Remarks
—	1008	0.66	0.2	
0	1008	"	1.0	
30	872	"	2.6	
60	788	0.50	2.6	
90	788	"	3.0	
120	778	"	3.4	
150	774	"	3.4	1st Gas Sample
180	782	"	3.8	
210	782	"	4.0	
240	782	"	2.6	
270	824	"	2.2	Feed Complete
300	856	"	2.2	
330	874	"	2.0	
360	892	"	1.8	
390	912	"	1.8	2nd Gas Sample
420	926	"	1.8	
450	936	"	1.8	
480	942	"	1.8	
510	946	"	2.0	3rd Gas Sample
540	946	"	2.0	
570	952	"	1.8	
600	952	"	2.0	
700	966	"	2.4	4th Gas Sample
800	972	0.97	2.2	5th Gas Sample
900	966	"	2.4	Turned off heat
1000	906	"	—	
1100	730	—	—	
1200	570	—	—	
1400	514	—	—	

TABLE NO. III - Continued

## Analyses:

Source	Basicity ( gram equivalents of Sodium )	Sodium Cyanide	Sodium Ferrocyanide
Product from bottom of reactor-	0.595	0.158	0.016
Cyclone	0.144	0.003	0.004
Bubbler	0.017	0.001	0.007
Reactor Washings	<u>0.457</u>	<u>0.010</u>	<u>0.025</u>
	1.213	0.152	0.052
Total equivalents of sodium accounted for-			1.27
Percent of sodium charged accounted for-			96.6 %
Percent of sodium charged converted to sodium cyanide-			12.0 %
Percent of sodium charged converted to sodium ferrocyanide-			4.1 %

Gas Samples:	1st	2nd	3rd	4th	5th
% CO	20.4	15.6	9.2	4.2	1.0
% H <sub>2</sub>	17.5	3.0	1.7	1.0	0.3
% N <sub>2</sub>	58.9	81.4	89.1	94.8	98.7
% CO <sub>2</sub>	3.2	—	—	—	—