PATENT SPECIFICATION

NO DRAWINGS

845,471

THE PATENT OFFICE LONDON

Date of Application and filing Complete Specification: March 11, 1958.

No. 7780/58.

Application made in United States of America on March 18, 1957.

Complete Specification Published: August 24, 1960.

Index at Acceptance:—Class 1(2), F(1X:4).

International Classification:—C01c.

COMPLETE SPECIFICATION

Method of Preparing Cyanogen

We, AMERICAN CYANAMID COMPANY, a corporation organised under the laws of the State of Maine, United States of America, of 30, Rockefeller Plaza, New York, State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method for preparing cyanogen and, more particularly, to the process for forming cyanogen by a flame synthesis in the absence or presence of an inert gaseous medium. Still more specifically, it is concerned with the combustion of gaseous hydrogen cyanide, chlorine and hydrogen to form cyanogen in high yield.

In the past, several processes have been suggested for preparing cyanogen. One such practice involves the reaction of anhydrous hydrogen cyanide and chlorine over a catalyst at elevated temperatures up to about 1000°C. Although the process is operable, a serious drawback is present due to the relatively slow reaction rate, even at these elevated temperatures. Consequently, a very large reaction space is needed to obtain commercially acceptable yields of cyanogen. Such large reaction volumes lead to problems of heat transfer, proper mixing of the gas reactants within the reaction vessel, and preclude use of higher temperatures which otherwise seem desirable.

Another procedure involves the reaction of cyanogen chloride and hydrogen at elevated temperatures up to 1000°C. Reaction times of more than 0·5 second are required. The principal shortcoming of this process is the requirement for preforming cyanogen chloride by reacting hydrogen cyanide and chlorine prior to reaction. This requires a necessary but wholly undesirable additional operational step.

It is a prime object of the present invention to overcome the disadvantages of the prior practices by devising a straight-forward procedure for directly reacting hydrogen cyanide and chlorine.

Such a process has many advantages. It would avoid the use of expensive equipment. For example, to withstand the corrosive effect of chlorine, stainless steel or other chlorine-resistant fabricating material was required for the reactor of prior art processes. However, in normal usage such reactor, by its physical nature, may be safely heated to temperatures not in excess of about 550°C., for otherwise chlorine becomes excessively corrosive. At such temperatures, reaction between chlorine and hydrogen cyanide is relatively slow and, therefore, is not wholly satisfactory. To elevate the temperature, say above 550°C., the reactor is soon rendered worthless.

To avoid this result, quartz which is resistant to chlorine corrosion was used as a fabricating material. However, at elevated temperatures in excess of about 1000°C., spalling occurred due to temperature fluctuations and quartz therefore was found unsuitable. Although quartz tubes and other suitably thermally insulated reactors have been previously suggested, the prior art has effected reaction within a relatively large volume or space, rather than in a concentrated or pin-pointed area independent of the proximity of wall enclosures, as proposed by the present invention.

According to this invention, there is provided a method of preparing cyanogen which comprises: igniting a gaseous mixture of hydrogen cyanide and chlorine in the presence of hydrogen at a temperature of at least 1200°C., and recovering cyanogen.

In the process of the invention, the reactants are fed to a suitable burner vessel where they are ignited. Advantageously, the reactants are preheated prior to reaction. For instance, hydrogen cyanide, chlorine and

[*Price 3s. 6d.*]

hydrogen may be preheated to temperatures of at least 200°C. or higher and are fed to a burner vessel where they are ignited at temperatures of 1200°C. and above. The process may be carried out in almost any type of burner vessel which is pressure tight and capable of being heated to 1200°C. Resultant gaseous products can be removed from the vessel so as to be further processed to recover cyanogen in good yield and purity. Cyanogen may be recovered in a variety of ways. For example, the gaseous products withdrawn from the burner vessel are passed through a water scrubber at ambient temperature to remove acidic and other impurities. Cyanogen is recovered by collecting the gas issuing from the scrubber.

In general, while the reacting gases may be ignited and burned directly, preheating of the gases prior to reaction has been found to be advantageous. Thus preheating to temperatures from 200°C. to 600°C. is a good practice. Preheating may be accomplished conventionally by heat exchange as between incoming gases and outgoing reacted gases. Another alternative method for preheating the gaseous mixture is to electrically heat the incoming gases to the desired temperature before introducing the latter gases to the burner. Ignition of the gases at the burner can be achieved by a variety of methods such as a retractable torch using oxygen and hydrogen, an electric spark or a heated platinum wire.

According to the stoichiometry of the hydrogen cyanide-chlorine reaction, two mols of hydrogen cyanide will react with one mol of chlorine to produce cyanogen. However, it has been surprisingly found that a lesser mol quantity of hydrogen cyanide can be used. For instance, as little as one and one-half mols of hydrogen cyanide per mol of chlorine can be reacted with good effect.

To supply heat and sustain the flame of reaction, the presence of hydrogen has presently been found to be prerequisite. The mol ratio of hydrogen may be widely varied, namely, from one-quarter (0.25) to two (2) mols of hydrogen per mol of chlorine.

It is an advantage of the present invention that hydrogen cyanide, chlorine and hydrogen may be separately fed to the burner and ignited. Alternatively, they may be variously premixed, such as by mixing hydrogen cyanide and chlorine prior to reaction. In addition, the reaction may occur in the presence or absence of an inert gas, such as nitrogen or helium.

The flame temperature, usually within the range of from 1200°C. to 1800°C., can be employed. The selection of such temperature depends upon the volume of hydrogen gas introduced. For instance, the higher the mol ratio of hydrogen to chlorine, the lower the temperature. In this practice, the reaction at the above flame temperatures is substantially completed within 0·001 to 0·00001 second or less.

The invention will be further illustrated in the following examples.

EXAMPLES

Several runs were made by continuously passing preheated gaseous hydrogen cyanide, chlorine, and hydrogen to a silica burner and ignited by means of a heated platinum wire. Resultant gases are removed, and cooled to ambient temperature in each run of the examples. Cyanogen is recovered by removing undesirable by-products in a water scrubber. It is then collected as the issuing gas from the scrubber and identified by infrared analysis.

The conditions of reaction and results obtained are recorded in the table below:

TABLE

Run	Gas Rate, ml/Min.				Preheat Temp. °C.	Flame Temp. °C.	Reaction Time Sec.	Yield* (%)
	H_2	HCN	Cl_2	N_2				
1	850	1040	750	0	About 400°C.	About 1200°C.	1×10^{-3}	19·7
2	120	1040	510	130	About 400°C.	About 1500°C.	1×10^{-4}	26·8
3	210	1040	510	130	About 400°C.	About 1500°C.	1×10^{-4}	28·6
4	185	1040	750	0	About 500°C.	About 1800°C.	1×10^{-5}	36·8

* %=Conversion to $(CN)_2$ based on HCN fed.

It is an advantage of the process of the invention that the time of reaction is markedly increased over the previous practices. Hence, efficiency of process and overall yield of product are increased while permitting the operation to remain independent of reaction volume considerations. Thus, the cost for equipment which is required for effecting reaction is relatively inexpensive. The process as described and claimed lends itself to good commercial exploitation.

WHAT WE CLAIM IS:—

1. A method of preparing cyanogen which comprises: igniting a gaseous mixture of hydrogen cyanide and chlorine in the presence of hydrogen at a temperature of at least 1200°C., and recovering cyanogen.

2. A method according to Claim 1 in which the gases are ignited in an atmosphere of nitrogen.

3. A method according to Claim 1 or 2 in which prior to ignition the mixture of chlorine, hydrogen cyanide and hydrogen in the mol ratio of 1:1·5-2·0:0·25-2, respectively, is preheated.

4. A method according to Claim 3 in which the gases are preheated at temperatures within the range 200°C. to 600°C.

5. A method according to any one of the preceding Claims in which the temperature of reaction is within the range 1200°C. to 1800°C.

6. A method according to Claim 5 in which the reaction time is between 1 x 10^{-3} and 1 x 10^{-5} second.

7. A method of preparing cyanogen substantially as hereinbefore described with reference to the Examples.

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Leeds: Printed for Her Majesty's Stationery Office, by W. A. Smith (Leeds) Ltd.—1960.
Published by The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.