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- (71) **Applicant:** INVISTA TECHNOLOGIES S. A R.L. [LU/CH]; Zweigniederlassung St. Gallen, Kreuzackerstrasse 9, CH-9000 St. Gallen (CH).
- (72) **Inventors:** FORSYTH, Stewart; 102 Yearsley Place, Wilmington, DE 19808 (US). LIU, Aiguo; 907 Lincoln Square, Apt. J, Elk Grove Village, IL 60007 (US). RENNER, Martin J.; 710 North Market Street, Hallettsville, TX 77964 (US). STAHLMAN, Brent J.; 1701 Victoria Station Drive, Apt. 708, Victoria, TX 77901 (US).
- (74) **Agent:** FURR, Robert B., Jr.; Three Little Falls Centre, 2801 Centerville Rd., Wilmington, DE 19808 (US).
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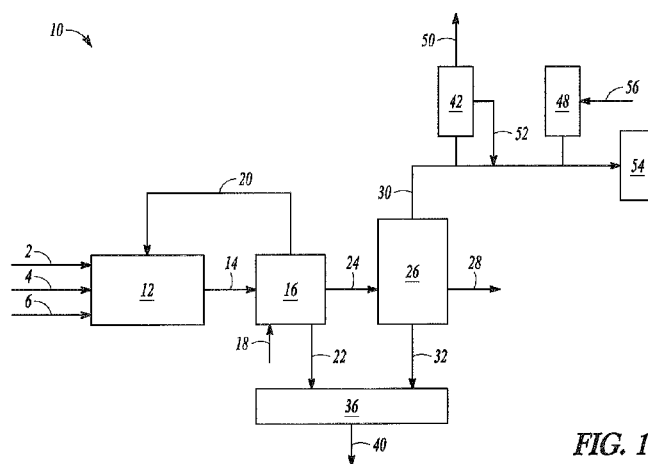


FIG. 1

(57) **Abstract:** Systems and methods for producing hydrogen cyanide via an Andrussov process are described. The system can include a reactor zone wherein oxygen, ammonia, and methane are allowed to react in the presence of a catalyst comprising platinum to provide at least hydrogen cyanide (HCN), hydrogen, and waste product. An HCN recovery zone can substantially remove the hydrogen cyanide from the hydrogen and the waste product. A flare zone can burn at least a combustible gas mixture in a hydrogen mode to produce at least carbon dioxide and water, the combustible gas mixture comprising at least a portion of the hydrogen and at least a portion of the waste product.

APPARATUS AND METHOD OF AN IMPROVED FLARE IN AN ANDRUSSOW PROCESS

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims the benefit of priority to U.S. Provisional Patent Application Serial No. 61/738,817 entitled "APPARATUS AND METHOD OF AN IMPROVED FLARE IN AN ANDRUSSOW PROCESS," filed December 18, 2012, the disclosure of which is incorporated herein in its entirety by reference.

TECHNICAL FIELD

[0002] The present disclosure is directed to a reactor scheme for the Andrussov process for the production of hydrogen cyanide (HCN) from methane, ammonia, and oxygen.

BACKGROUND

[0003] The Andrussov process can be used for gas phase production of hydrogen cyanide (HCN) from methane, ammonia, and oxygen over a platinum catalyst. Filtered ammonia, natural gas, and air are fed into a reactor and heated to about 800 °C to about 2,500 °C in the presence of a catalyst that includes at least platinum. The methane can be supplied from natural gas, which can be further purified. Hydrocarbons having at least two carbons can be present in natural gas. Air can be used as a source of oxygen. The reactor off-gas containing HCN and un-reacted ammonia can be quenched in a waste heat boiler to approximately 100 °C to 400 °C. The quenched reactor off-gas, containing HCN, can be sent through an ammonia absorption process to remove un-reacted ammonia, such as by contacting the reactor off-gas with ammonium phosphate solution, phosphoric acid, or sulfuric acid to remove the ammonia. From the ammonia absorber, the product off-gas can be sent through an HCN absorber, where cold water can be added to entrain the HCN. The HCN absorber can produce a gaseous waste stream that contains substantially hydrogen and by products. Due to the composition of the gaseous waste stream disposal can be an environmental concern, can be inefficient to flare off, or combinations thereof.

[0004] Various aspects of HCN production are described in the following articles: Eric L. Crump, U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards,

Economic Impact Analysis For the Proposed Cyanide Manufacturing NESHAP (May 2000), available online at <http://nepis.epa.gov/Exe/ZyPDF.cgi?Dockey=P100AHG1.PDF>, is directed toward the manufacture, end uses, and economic impacts of HCN; N.V. Trusov, *Effect of Sulfur Compounds and Higher Homologues of Methane on Hydrogen Cyanide Production by the Andrussov Method*, *Rus. J. of Applied Chemistry*, Vol. 74, No. 10, pp. 1693–97 (2001), is directed toward the effects of unavoidable components of natural gas, such as sulfur and higher homologues of methane, on the production of HCN by the Andrussov process; Clean Development Mechanism (CDM) Executive Board, United Nations Framework Convention on Climate Change (UNFCCC), *Clean Development Mechanism Project Design Document Form (CDM PDD)*, Ver. 3, (Jul. 28, 2006), available online at http://cdm.unfccc.int/Reference/PDDs_Forms/PDDs/PDD_form04_v03_2.pdf, is directed toward the production of HCN by the Andrussov process; and Gary R. Maxwell et al., *Assuring process safety in the transfer of hydrogen cyanide manufacturing technology*, *J. of Hazardous Materials*, Vol. 142, pp. 677–84 (2007), is directed toward the safe production of HCN.

SUMMARY

[0005] The present disclosure is directed to a solution to disposal of a gaseous waste stream from an HCN absorber in an Andrussov process. The solution can include a system and method for producing hydrogen cyanide via the Andrussov process. The system can include a reactor zone wherein oxygen, ammonia, and methane are allowed to react in the presence of a catalyst comprising platinum to provide at least hydrogen cyanide (HCN), hydrogen, and waste product. An HCN recovery zone can substantially remove the hydrogen cyanide from the hydrogen and the waste product. A flare zone can burn a combustible gas mixture in hydrogen mode to produce at least carbon dioxide and water, the combustible gas mixture comprising at least a portion of the hydrogen and at least a portion of the gaseous waste product. The combustible gas mixture can include natural gas.

[0006] A hydrocarbon mixture can provide the methane in the system. The hydrocarbon mixture can comprise natural gas, biogas, substantially pure methane, or mixtures thereof. The system can include a hydrogen recovery system, which can recycle, store, or utilize the recovered hydrogen downstream or upstream of the hydrogen recovery system. The system can

include an ammonia extraction zone wherein the ammonia is substantially removed from the HCN, hydrogen, and waste product.

[0007] A hydrogen cyanide recovery zone can provide a hydrogen cyanide product stream that can include a purity of at least about 98.5% hydrogen cyanide or provide the gaseous waste product stream such that less than about 1.5% HCN is present. The combustible gaseous waste mixture, fed to the flare, can comprise at least about 8 vol% hydrogen. The flare exit velocity can be less than about 37.2 meters per second, such as less than about $V_{\max}$, wherein $V_{\max}$ is defined by:

$$V_{\max} = (X_{H_2} - K_1) * K_2, \text{ wherein}$$

$V_{\max}$ = maximum permitted flare exit velocity, meters per second

K_1 = constant, 6.0 volume% hydrogen

K_2 = constant, 3.9 meters per second per volume % hydrogen

X_{H_2} = volume % hydrogen, on a wet basis, as calculated by using the

American Society for Testing and Materials (ASTM) Method D1946-77.

[0008] The flare zone can be configured to operate at temperatures of greater than about 1350 °C. The flare can be unassisted or stabilized. The flare zone can comprise at least a 3.0 inch diameter flare tip.

[0009] The flare zone further can be configured to flare a non-hydrogen combustible gas mixture burned in a non-hydrogen mode, such as a non-hydrogen combustible gas mixture of a heat content of at least about 200 BTU/scf. The system can include at least one liquid separator, upstream of the flare, to remove at least a portion of liquid present in the combustible gas mixture or non-hydrogen combustible gas mixture.

[0010] According to the present disclosure, a method for burning a waste product produced by an Andrussov process can include allowing oxygen, ammonia, and methane to react in the presence of a catalyst comprising platinum to give a product stream comprising at least hydrogen cyanide, hydrogen, and a waste product. Hydrogen cyanide can be extracted from the product stream to produce an HCN product stream and a gaseous waste stream comprising at least a portion of the hydrogen and the waste product and the gaseous waste stream can be feed to a hydrogen mode flare.

[0011] The method can include extracting ammonia from the product stream, such as prior to extracting hydrogen cyanide. At least a portion of liquid can be separated from the

gaseous waste stream prior to feeding the gaseous waste stream to the hydrogen mode flare. Hydrogen can be recovered from the gaseous waste stream. Such a gaseous waste stream with reduced hydrogen content can still be efficiently flared using the methods and apparatus described herein. The method can include supplementing the flared components with hydrogen or hydrocarbons, such as methane.

[0012] The method can include flaring the extracted stream at a flare exit velocity of less than about 37.2 meters per second, such as $V_{\max}$. The flare zone can operate at a temperature of greater than about 1650 °C. The method can include assisting or stabilizing the flare.

[0013] These and other examples and features of the present systems and methods will be set forth in part in the following Detailed Description. This Summary is intended to provide an overview of the present subject matter, and is not intended to provide an exclusive or exhaustive explanation. The Detailed Description below is included to provide further information about the present systems and methods.

BRIEF DESCRIPTION OF THE FIGURES

[0014] FIG. 1 is a schematic block flow diagram of an HCN production via the Andrussow process according to the present disclosure.

DETAILED DESCRIPTION

[0015] The synthesis of hydrogen cyanide by the Andrussow method (see, for example, Ullmann's Encyclopedia of Industrial Chemistry, Volume 8, VCH Verlagsgesellschaft, Weinheim, 1987, pp. 161–162) can be carried out in the vapor phase over a catalyst that comprises platinum or platinum alloys, or other metals. Catalysts suitable for carrying out the Andrussow process were discovered and described in the original Andrussow patent, published as U.S. Pat. No. 1,934,838, and elsewhere. In Andrussow's original work, he disclosed that catalysts can be chosen from oxidation catalysts that are infusible (solid) at the working temperature of around 1000°C; he included platinum, iridium, rhodium, palladium, osmium, gold or silver as catalytically active metals either in pure form or as alloys. He also noted that certain base metals, such as rare earth metals, thorium, uranium, and others, could also be used, such as in the form of infusible oxides or phosphates, and that catalysts could either be formed into nets (screens), or deposited on thermally-resistant solid supports such as silica or alumina.

[0016] In subsequent development work, platinum-containing catalysts have been selected due to their efficacy and to the heat resistance of the metal even in gauze or net form. For example, a platinum-rhodium alloy can be used as the catalyst, which can be in the form of a metal gauze or screen such as a woven or knitted gauze sheet, or can be disposed on a support structure. In an example, the woven or knitted gauze sheet can form a mesh-like structure having a size from 20-80 mesh, e.g., having openings with a size from about 0.18 mm to about 0.85 mm. A catalyst can comprise from about 85 wt% to about 95 wt% Pt and from about 5 wt% to about 15 wt% Rh, such as 85/5 Pt/Rh, 90/10, or 95/5 Pt/Rh. A platinum-rhodium catalyst can also comprise small amounts of metal impurities, such as iron (Fe), palladium (Pd), iridium (Ir), ruthenium (Ru), and other metals. The impurity metals can be present in trace amounts, such as about 10 ppm or less.

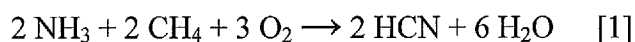
[0017] A broad spectrum of possible embodiments of the Andrussov method is described in German Patent 549,055. In one example, a catalyst comprising a plurality of fine-mesh gauzes of Pt with 10% rhodium disposed in series is used at temperatures of about 800 to 2,500° C, 1,000 to 1,500° C, or about 980 to 1050° C. For example, the catalyst can be a commercially-available catalyst, such as a Pt-Rh catalyst gauze available from Johnson Matthey Plc, London, UK, or a Pt-Rh catalyst gauze available from Heraeus Precious Metals GmbH & Co., Hanau, Germany.

[0018] The composition of a gaseous waste stream from an Andrussov process can make disposal of the stream difficult. For example, the gaseous waste stream can be such that flaring is inefficient, harmful to the environment, outside the parameters of local or federal regulations, or combinations thereof. The present disclosure is directed to an apparatus and method for the synthesis of HCN via an Andrussov process, in which an improved flare can dispose of the gaseous waste stream taking into consideration environmental concerns, efficiency concerns, or governmental regulations.

[0019] FIG. 1 is a schematic block flow diagram of an example process 10 for the production of hydrogen cyanide (HCN) via the Andrussov process, according to the present disclosure. In the example process 10, a HCN reaction zone 12 is supplied with an ammonia (NH₃) stream 2, a methane (CH₄) stream 4, and an air stream 6 (which includes oxygen gas (O₂)). Air can include air and other oxygen-containing gas mixtures, including oxygen-enriched air having oxygen concentrations above about 21 vol%. Examples herein can be particularly

useful when an Andrussov process is adapted to employ a gaseous oxygen feed stream **6** that contains somewhat higher levels of oxygen than air. For example, such a gaseous oxygen feed stream **6** can contain at least about 25 vol% oxygen, at least about 30 vol% oxygen, at least about 40 vol% oxygen, at least about 50 vol% oxygen, at least about 60 vol% oxygen, at least about 70 vol% oxygen, at least about 80 vol% oxygen, at least about 90 vol% oxygen, at least about 95 vol% oxygen, at least about 98 vol% oxygen. The process **10** can include an air Andrussov process, e.g., an air stream **6** that includes about 21 vol% oxygen, an air-enriched Andrussov process, e.g., an air stream **6** that includes greater than about 21 vol% oxygen but less than about 100 vol% oxygen, or an oxygen Andrussov process, e.g., an air stream **6** that includes about 100% oxygen.

[0020] The three feed streams **2, 4, 6**, are mixed and reacted in one or more reactors to be converted to hydrogen cyanide and water in the presence of a catalyst according to Equation 1:



[0021] The one or more reactors can include a HCN catalyst, such as platinum (Pt) or a platinum alloy, such as an alloy of platinum with rhodium (Rh) or palladium (Pd) containing at least about 85% platinum by weight. Alloys used in the Andrussov process can include, but are not limited to, 10 wt% Rh – 90 wt% Pt, 8 wt% Rh – 92 wt% Pt, 5 wt% Pd – 5 wt% Rh – 90 wt% Pt, or 5 wt% Rh – 95 wt% Pt. Alloys containing up to about 5 wt% iridium (Ir) can be used. In an example, the HCN catalyst can be designed to reduce by products, such as nitrous-oxide by products, and therefore can have an increase rhodium (Rh) content, or other materials, such as cobalt (Co). The HCN catalyst can be contained in a packed bed, such as in a packed bed reactor, or be formed as a gauze, such as by weaving or knitting metal filaments into gauze-like structures. Such formed catalysts are well established in the art and can contain the catalytic materials described herein.

[0022] The HCN catalyst can be a commercially-available catalyst, such as a Pt-Rh catalyst gauze available from Johnson Matthey Plc, London, UK, or a Pt-Rh catalyst gauze available from Heraeus Precious Metals GmbH & Co., Hanau, GERMANY.

[0023] The resulting product stream **14** from the HCN reaction zone **12** can be fed into an ammonia recovery system **16** that is configured to recover unreacted NH₃. Ammonia can be recovered by NH₃ absorption via providing one or more of phosphoric acid (H₃PO₄), sulfuric

acid (H_2SO_4), or an ammonium phosphate solution to the product stream **14** that can absorb NH_3 from the product stream **14**. In the example shown in **FIG. 1**, a phosphoric acid stream **18** within the ammonia recovery system **16** can absorb NH_3 . Ammonia can be removed from the $\text{H}_3\text{PO}_4/\text{NH}_3$ solution using one or more strippers to separate the NH_3 from the H_3PO_4 . The NH_3 can be recycled back to the HCN reaction zone **12** via an NH_3 recycle stream **20**. The H_3PO_4 and other waste can be purged as a wastewater stream **22**, while an NH_3 -stripped HCN stream **24** can be fed to an HCN recovery system **26**.

[0024] The HCN recovery system **26** can include one or more unit operations configured to separate and purify HCN from the HCN stream **24**. As a result of the HCN recovery system **26**, a purified HCN product stream **28** is produced. The HCN recovery system **26** can also produce a waste gas **30** or a wastewater stream **32**. Wastewater streams **22**, **32** can be fed to waste water treatment **36** for further processing, such as recovery of ammonia or hydrogen cyanide. The final wastewater stream **40** from the waste water treatment **36** can be further processed, stored, or disposed.

[0025] As illustrated in **FIG. 1**, HCN recovery system **26** can include a gaseous waste stream **30**. Gaseous waste stream **30** can include hydrogen, carbon monoxide, nitrogen, carbon dioxide, hydrogen cyanide, or mixtures thereof. In an example, gaseous waste stream **30** can include additional waste streams not illustrated. For example, a waste stream from reactor **12** or other operation can be combined with stream **30**. The composition of gas waste stream **30** can vary according to a number of factors, including composition of the feed streams **2**, **4**, **6**, efficiency of the HCN reaction zone **12**, efficiency of the ammonia recovery system **16**, efficiency of the HCN recovery system **26**, operating conditions, or combinations thereof.

[0026] In an example, gaseous waste stream **30** can feed a hydrogen recovery system **42**. In general, the gaseous waste stream **30** can contain residual amounts of HCN, as well as significant hydrogen gas or a variety of gases including unreacted methane, carbon dioxide, carbon monoxide, water, nitrogen, and a variety of organonitriles. Removal of HCN can be substantially complete, not only so that valuable HCN is not lost to the waste stream, but also for health and environmental concerns, and because significant amounts of HCN can complicate processing of the waste stream. For example, some pressure swing apparatus absorbents that can be used for hydrogen recovery can have operational limits of less than about 2.0% HCN, or less than about 1.5% HCN. Hence, the gaseous waste stream can have less than about 2.0% HCN, or

less than about 1.5% HCN, or less than about 1% HCN, or less than about 0.5% HCN, or less than about 0.2% HCN, or less than about 0.1% HCN. In some situations, the HCN content of the gaseous waste stream **30** can vary, for example, between about 0.5% to about 1.0%. For example, gaseous waste stream **30** can include 40-75 vol% H₂, 15-35 vol% CO, 5-15 vol% N₂, 1-2 vol% CO₂, 1-2 vol% CH₄, or 0-1 vol% HCN.

[0027] The recovered hydrogen stream **50** can be stored for future processing or sale, sent to additional hydrogen processing units, recycled to a point upstream or downstream, used to supplement a flare **48** (as discussed further below), or combinations thereof. Hydrogen recovery system **42** can regulate a level of hydrogen that is recovered from combined gaseous waste stream **20**. For example, if combined gaseous stream **30** is hydrogen rich, such as greater than about 40 vol%, hydrogen recovery system **42** can recover a larger percentage of hydrogen. While the recovered hydrogen stream **50** need not be 100% pure, removal of at least the majority of the carbon dioxide, carbon monoxide, or nitriles from hydrogen can be desirable to avoid side products and contaminants when the recovered hydrogen is used, for example, for hydrogenation reactions. For example, the recovered hydrogen gas can be at least about 90% pure, or at least about 91 % pure, or at least about 92% pure, or at least about 93 % pure, or at least about 94% pure, or at least about 95 % pure, or at least about 96% pure, or at least about 97 % pure, or at least about 98% pure, or at least about 99% pure hydrogen. Further, if a processing unit downstream, such as flare **48**, is designed to operate at a range of hydrogen concentrations, hydrogen recovery system **42** can be optimized to provide a processed gaseous waste stream **52** to a processing unit downstream. In an example, the hydrogen recovery system **42** can produce a processed gaseous waste stream including at least about 8 vol% hydrogen. Gaseous waste stream **52** can include less than about 1.5% HCN. A valve can be located downstream of hydrogen recovery system **42** but upstream of processed gaseous waste stream **52**, such that gaseous waste stream **30** feeds directly and only to the hydrogen recovery system **42**.

[0028] In an example, system **10** can include the flare **48** to burn off gaseous waste stream **30**. Flare **48** can be designed to thoroughly destroy gaseous waste. If the gaseous waste is dilute, stream **30** can be enriched with hydrocarbon fuel **56**, such as natural gas, to increase a heating value of the resulting blend in order to ensure that the waste is thoroughly destroyed. Hydrogen has a lower net heating value than many hydrocarbon fuels. For example, the net heating value of methane is about 913 BTU/scf. On the other hand, the net heating value of

hydrogen is only about 275 BTU/scf. In view of this disparity, one would not expect that a gaseous mixture containing a relatively low concentration of hydrogen would be sufficient to thoroughly destroy the dilute gaseous waste with which it is blended. However, unexpectedly, the present inventors discovered that a minimum amount of about at least 5 vol %, of about at least 7 vol%, of about at least 8 vol%, of about at least 10 vol % of hydrogen in the gaseous mixture flared was sufficient to ensure destruction of the gaseous waste.

[0029] In an example, a detector that can quantify components in the combustible gas mixture. A valve can be operably linked to the detector, where the valve is configured to permit supplementing the combustible gas mixture **30** with a hydrogen-containing enrichment fuel, such as hydrogen or methane, when a set value of non-combustible components, corresponding to a lower threshold of heat content, are detected in the combustible gas mixture. The amount of hydrogen-containing enrichment fuel needed to thoroughly destroy the dilute gaseous waste material upon combustion also depends, in part, on the heating value of the dilute gaseous waste material flared. The enrichment stream can contain a sufficient amount of hydrogen or hydrocarbons such that, when blended with the gaseous waste stream **30**, the resulting blend can have a sufficient heating value to thoroughly destroy the dilute gaseous waste material upon combustion in the flare **48**.

[0030] In an example, a valve can be operably linked to the detector, where the valve is configured to permit supplementing the oxygen permitted to react when a hydrogen concentration below a threshold value is detected in the combustible gas-mixture **30**. An increase in the amount of oxygen **6** can result in an increase in hydrogen concentration in the combustible gas mixture **30**. In an example, the oxygen can be provided by air stream **6** or an oxygen supplement stream upstream of or connected to the HCN reactor **12**. Surprisingly, providing greater oxygen concentrations to the HCN reactor **12**, and consequently lower inert gas concentrations (e.g., N₂), can provide the benefit of more combustible gas waste **30** that can be utilized in flare **48**. Typically, inert gases are utilized to provide a heat transfer benefit during the reaction. For example, the presence of nitrogen in the reactor can aid in decreasing temperature spikes. The present inventors have invented an HCN process capable of handling greater oxygen concentrations in a feed stream by using a flare, such as flare **48**, to increase temperature control of the system. Further, greater oxygen concentrations in the feed stream can

provide the benefit of reducing an amount of fuel, such as methane 4 required for the HCN reactor 12.

[0031] In an example flare 48 can be designed to meet local environment or federal environmental velocity requirements of the flare. For example, a flare exit velocity can be less than about 37.2 meters per second. Flare exit velocity is the velocity of the flare at the tip of the flare device 48. In another example, the flare velocity can be designed to be less than a $V_{\max}$, where $V_{\max}$ is defined by

$$V_{\max} = (X_{H_2} - K_1) * K_2, \text{ wherein}$$

$V_{\max}$ = maximum permitted flare exit velocity, meters per second

K_1 = constant, 6.0 volume% hydrogen

K_2 = constant, 3.9 meters per second per volume % hydrogen

X_{H_2} = volume % hydrogen, on a wet basis, as calculated by using the

American Society for Testing and Materials (ASTM) Method D1946-77. Increasing the amount of oxygen allowed to react in the HCN reactor 12 can provide the benefit of increasing the concentration of hydrogen in the waste stream 30, consequently increasing the flare velocity, as described above.

[0032] In an example, the flare 48 can be designed to burn the combustible gaseous mixture at temperatures of greater than about 1000 °C, greater than about 1200 °C, of greater than about 1350 °C, of greater than about 1500 °C, of greater than about 1600 °C, or greater than about 1800 °C. The flare burn temperature can include a temperature greater than a combustion temperature of one or more component of the combustible gaseous mixture. In an example, the combustible gaseous mixture can include supplemental hydrocarbons, such as natural gas, or hydrogen, to increase the combustibility of the combustible gaseous mixture.

[0033] The flare 48 can include any flare suitable for destruction of the combustible gaseous mixture. For example, the flare 48 can include an unassisted flare or an assisted flare, or stabilized or non-stabilized flare. An assisted flare includes an additional stream, such as steam assisted or air assisted, to provide for an increased flare exit velocity, increased combustible composition, more consistent combustible composition, or combinations thereof. A stabilized flare can include an additional stream, such as hydrocarbon stabilized or air stabilized, to increase flare consistency, such as exit velocity, or temperature. A flare nozzle of the flare 48

can be in compliance of any designed regulations, such as a flare tip or nozzle dimension. In an example, the flare zone can include an at least a 3.0 inch diameter flare tip.

[0034] In an example, the flare zone further can be configured to operate with a non-hydrogen combustible gas mixture burned in a non-hydrogen mode. The non-hydrogen combustible gas mixture can include a heat value of at least about 200 BTU/scf. The flare 48 can switch between a hydrogen mode, such as burning a minimum percentage of hydrogen, or a non-hydrogen mode, such as zero vol% hydrogen, or combinations thereof. The flare 48 can provide the benefit of increased operational flexibility.

[0035] In an example, the system 10 can include a scrubber downstream or upstream of the hydrogen recovery system 42 to remove at least a portion of liquid, such as water, present in gaseous waste stream 30. Removing liquid provided to the flare 48 can provide a steady flare, a more predictable heat capacity of the feed provided to the flare 48. Combustible gaseous waste 30 can be fed to additional processes 54 of system 10, such as a boiler that can be utilized downstream to recycle heat energy.

[0036] According to the present disclosure, a method for the production of hydrogen cyanide can include allowing oxygen, ammonia, and methane to react in the presence of the catalyst comprising platinum to give a product stream comprising at least hydrogen cyanide, hydrogen, and the waste product. The method can further include extracting the hydrogen cyanide from the product stream to produce the HCN product stream (FIG. 1, element 28) and the gaseous waste stream (FIG. 1, element 30) comprising at least a portion of the hydrogen and the waste product and feeding the gaseous waste stream to a hydrogen mode flare (FIG. 1, element 48). The gaseous waste stream can include more than one gaseous waste stream combined with the gaseous waste stream, such that at least a portion of the waste from the system is flared, for example in flare 48.

[0037] In an example, the method can include separating at least a portion of liquid in the gaseous waste stream, such as by a vapor-liquid separator, flash drum, knock-out drum, knock-out pot, compressor suction drum, compressor inlet drum, demister, or combinations thereof.

[0038] In an example, the method includes extracting ammonia from the product stream, such as by an ammonia scrubber. The method can also include recovering hydrogen from the gaseous waste stream or combined gaseous waste stream. For example, the hydrogen composition of the gaseous waste stream may be in excess of hydrogen mode flaring levels. In

such instances, the excess hydrogen can be recovered and utilized elsewhere in a plant, stored for future use or sale, or combinations thereof.

[0039] The method can include regulating the flare, such that the flare exit velocity is less than about 37.2 meters per second. Regulating flare exit velocity can provide the benefit of a stable flare, increasing the destruction of the elements or compounds burned. Further, the method can include flaring the gaseous waste stream at a temperature of greater than about 1650 ° C.

EXAMPLES

Example 1: Comparison Air, Air Enriched, and Oxygen Andrussov Process Waste Gas Compositions

[0040] This Example illustrates that an Andrussov process that uses an enriched source of oxygen generally produces a waste stream with higher hydrogen content than one that employs air as an oxygen source.

[0041] The method can include regulating the flare, such that the flare exit velocity is less than about 37.2 meters per second. Regulating flare exit velocity can provide the benefit of a stable flare, increasing the destruction of the elements or compounds burned. Further, the method can include flaring the gaseous waste stream at a temperature of greater than about 1650 ° C.

[0042] A 4 inch internal diameter stainless steel reactor with ceramic insulation lining inside is used for pilot scale test. Forty sheets of 90 wt% Pt/10 wt% Rh 40 mesh gauze from Johnson Matthey (USA) are loaded as catalyst bed. Perforated alumina tile is used for catalyst sheet support. The total flow rate is set at 2532 SCFH (standard cubic foot per hour). Hydrogen cyanide is produced via multiple Andrussov processes. One process, the air Andrussov process, employs an oxygen-containing gas (air) that includes 21 vol% oxygen. A second process, the air-enriched Andrussov process, employs an oxygen-containing gas having greater than about 21 vol% oxygen and less than about 100 vol% oxygen. A third process, the oxygen Andrussov process, employs an oxygen-containing gas that is about 100 vol% oxygen.

[0043] Ammonia is separately removed from each of the product streams in a process involving absorption into an ammonium phosphate stream. Hydrogen cyanide is then removed from the ammonia-depleted product stream in a process involving acidified water, thereby

separately generating a hydrogen cyanide product and a gaseous waste stream for each of the processes.

[0044] The composition of the gaseous waste streams from the air, air enriched, and oxygen processes are shown below in Table 1.

Table 1

	Reactant Feed stream (O₂ Concentration)	Waste Gas Stream (H₂ Concentration)
Air Process	20.95% Oxygen	16.7% Hydrogen
Air Enriched Process	25% Oxygen	21.2% Hydrogen
	35% Oxygen	32.3% Hydrogen
	45% Oxygen	42.6% Hydrogen
	55% Oxygen	51.8% Hydrogen
	65% Oxygen	59.6% Hydrogen
	75% Oxygen	66.3% Hydrogen
	80% Oxygen	69.2% Hydrogen
	85% Oxygen	71.9% Hydrogen
	90% Oxygen	74.4% Hydrogen
	95% Oxygen	76.7% Hydrogen
Oxygen Process	Pure Oxygen	78.8% Hydrogen

[0045] As illustrated, an Andrussov process that employs an enriched oxygen stream as a source of the oxygen reactant generates significantly more hydrogen than an Andrussov process that employs air as a source of the oxygen reactant.

Example 2: Comparison BTU values for Air, Air Enriched and Oxygen Andrussov Process Waste Gas Compositions

[0046] This Example illustrates that an Andrussov process that uses an enriched source of oxygen generally produces a waste stream with higher heating value than one that employs air as an oxygen source.

[0047] Three processes, as outlined in Example 1, are used to produce HCN. The heating value of the gaseous waste streams from the air, air enriched, and oxygen processes are shown below in Table 2.

Table 2

	Reactant Feed stream (O₂ Concentration)	Waste Gas Stream – Heating Value (BTU/SCF)
Air Process	20.95% Oxygen	67.6
Air Enriched Process	25% Oxygen	83.2
	35% Oxygen	120.3
	45% Oxygen	153.3
	55% Oxygen	181.5
	65% Oxygen	205.0
	75% Oxygen	224.4
	80% Oxygen	232.7
	85% Oxygen	240.3
	90% Oxygen	247.1
	95% Oxygen	253.3
Oxygen Process	Pure Oxygen	258.9

[0048] As illustrated, an Andrussow process that employs an enriched oxygen stream as a source of the oxygen reactant generates a waste stream with a significantly lower heating value than an Andrussow process that employs air as a source of the oxygen reactant.

[0049] The above Detailed Description is intended to be illustrative, and not restrictive. For example, the above-described examples (or one or more elements thereof) can be used in combination with each other. Other examples can be used, such as by one of ordinary skill in the art upon reviewing the above description. Also, various features or elements can be grouped together to streamline the disclosure. This should not be interpreted as intending that an unclaimed disclosed feature is essential to any claim. Rather, inventive subject matter can lie in less than all features of a particular disclosed example. Thus, the following claims are hereby incorporated into the Detailed Description, with each claim standing on its own as a separate example. The scope of the invention should be determined with reference to the appended claims, along with the full scope of equivalents to which such claims are entitled.

[0050] In the event of inconsistent usages between this document and any documents so incorporated by reference, the usage in this document controls.

[0051] In this document, the terms “a” or “an” are used, as is common in patent documents, to include one or more than one, independent of any other instances or usages of “at least one” or “one or more.” In this document, the term “or” is used to refer to a nonexclusive or, such that “A or B” includes “A but not B,” “B but not A,” and “A and B,” unless otherwise indicated. In this document, the terms “including” and “in which” are used as the plain-English equivalents of the respective terms “comprising” and “wherein.” Also, in the following claims, the terms “including” and “comprising” are open-ended, that is, a system, device, article, composition, formulation, or process that includes elements in addition to those listed after such a term in a claim are still deemed to fall within the scope of that claim. Moreover, in the following claims, the terms “first,” “second,” and “third,” etc. are used merely as labels, and are not intended to impose numerical requirements on their objects.

[0052] Method examples described herein can be machine or computer-implemented, at least in part. Some examples can include a computer-readable medium or machine-readable medium encoded with instructions operable to configure an electronic device to perform methods or method steps as described in the above examples. An implementation of such methods or method steps can include code, such as microcode, assembly language code, a higher-level

language code, or the like. Such code can include computer readable instructions for performing various methods. The code may form portions of computer program products. Further, in an example, the code can be tangibly stored on one or more volatile, non-transitory, or non-volatile tangible computer-readable media, such as during execution or at other times. Examples of these tangible computer-readable media can include, but are not limited to, hard disks, removable magnetic disks, removable optical disks (e.g., compact disks and digital video disks), magnetic cassettes, memory cards or sticks, random access memories (RAMs), read only memories (ROMs), and the like.

[0053] The Abstract is provided to comply with 37 C.F.R. §1.72(b), to allow the reader to quickly ascertain the nature of the technical disclosure. It is submitted with the understanding that it will not be used to interpret or limit the scope or meaning of the claims.

[0054] Although the invention has been described with reference to exemplary examples, workers skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention.

[0055] The invention has been described broadly and generically herein. Each of the narrower species and subgeneric groupings falling within the generic disclosure also form part of the invention. This includes the generic description of the invention with a proviso or negative limitation removing any subject matter from the genus, regardless of whether or not the excised material is specifically recited herein. In addition, where features or aspects of the invention are described in terms of Markush groups, those skilled in the art will recognize that the invention is also thereby described in terms of any individual member or subgroup of members of the Markush group.

[0056] The following statements describe some of the elements or features of the invention. Because this application is a provisional application, these statements may become changed upon preparation and filing of a nonprovisional application. Such changes are not intended to affect the scope of equivalents according to the claims issuing from the nonprovisional application, if such changes occur. According to 35 U.S.C. § 111(b), claims are not required for a provisional application. Consequently, the statements of the invention cannot be interpreted to be claims pursuant to 35 U.S.C. § 112.

STATEMENTS OF THE INVENTION:

[0057] 1. A system for producing hydrogen cyanide via the Andrussow process, comprising:

a reactor zone wherein oxygen, ammonia, and methane are allowed to react in the presence of a catalyst comprising platinum or platinum alloy to provide a product stream comprising at least hydrogen cyanide (HCN), hydrogen, and waste product;

an HCN recovery zone wherein the hydrogen cyanide is substantially removed from the product stream to generate a hydrogen cyanide product and a combustible gas mixture comprising the hydrogen and the waste product; and

a flare zone wherein at least the combustible gas mixture is burned in a hydrogen mode to produce at least carbon dioxide and water.

[0058] 2. The system of statement 1, wherein a hydrocarbon mixture comprises the methane.

[0059] 3. The system of statement 2, wherein the hydrocarbon mixture comprises natural gas, biogas, substantially pure methane, or mixtures thereof.

[0060] 4. The system of any one of statements 1-3, further comprising a hydrogen recovery system that recovers at least some hydrogen from at least one of the product stream and the combustible gas mixture.

[0061] 5. The system of any one of statements 1-4, further comprising an ammonia recovery system that substantially removes ammonia from the product stream.

[0062] 6. The system of any one of statements 1-5, wherein the hydrogen cyanide product is at least about 98.5 vol% pure hydrogen cyanide.

[0063] 7. The system of any one of statements 1-6, wherein the combustible gas mixture comprises at least about 8 vol% hydrogen.

[0064] 8. The system of any one of statements 1-7, wherein the flare has a flare exit velocity of less than about 37.2 meters per second.

[0065] 9. The system of any one of statements 1-8, wherein the flare has a flare exit velocity of less than about $V_{\max}$, wherein $V_{\max}$ is defined by:

$$V_{\max} = (X_{\text{H}_2} - K_1) * K_2, \text{ wherein}$$

$V_{\max}$ = maximum permitted flare exit velocity, meters per second

K_1 = constant, 6.0 volume% hydrogen

K_2 = constant, 3.9 meters per second per volume % hydrogen

X_{H_2} = volume % hydrogen, on a wet basis, as calculated by using the American Society for Testing and Materials (ASTM) Method D1946-77.

[0066] 10. The system of any one of statements 1-9, wherein the flare zone comprises temperatures of greater than about 1000 °C, greater than about 1200 °C, of greater than about 1350 °C, of greater than about 1500 °C, of greater than about 1600 °C, or greater than about 1800 °C

[0067] 11. The system of any one of statements 1-10, wherein the flare zone comprises an unassisted flare.

[0068] 12. The system of any one of statements 1-11, wherein the flare zone comprises at least a 3.0 inch diameter flare tip.

[0069] 13. The system of any one of statements 1-12, further comprising a flare stabilizer.

[0070] 14. The system of any one of statements 1-13, wherein the flare zone further comprises natural gas.

[0071] 15. The system of any one of statements 1-14, further comprising a hydrogen source that adds hydrogen to the combustible gas mixture.

[0072] 16. The system of any of statements 1-15, further comprising a detector that quantifies components in the combustible gas mixture.

[0073] 17. The system of statement 16, further comprising a valve operably linked to the detector, where the valve is configured to permit supplementing the combustible gas mixture with hydrogen or methane when a set value of non-combustible components, corresponding to a lower threshold of heat content, are detected in the combustible gas mixture.

[0074] 18. The system of statement 16, further comprising a valve operably linked to the detector, where the valve is configured to permit supplementing the oxygen permitted to react when a hydrogen concentration below a threshold value is detected in the combustible gas-mixture.

[0075] 19. The system of statement 18, wherein the threshold value is based on a heat content value of the combustible gas mixture.

[0076] 20. The system of statement 18 or 19, wherein the valve is operably linked to an air feed stream.

- [0077] 21. The system of statement 18 or 19, wherein the valve is operably linked to an oxygen feed stream.
- [0078] 22. The system of statement 18 or 19, wherein the valve is operably linked to an oxygen enriched air feed stream.
- [0079] 23. The system of any one of statements 1-22, wherein the flare zone further comprises a non-hydrogen combustible gas mixture burned in a non-hydrogen mode.
- [0080] 24. The system of statement 23, wherein the non-hydrogen combustible gas mixture comprises a heat value of at least about 200 BTU/scf.
- [0081] 25. The system of any one of statements 1-24, wherein the waste product comprises less than about 1.5% HCN.
- [0082] 26. The system of any one of statements 1-25, further comprising at least one liquid separator, upstream of the flare, to remove at least a portion of liquid present in the combustible gas mixture or non-hydrogen combustible gas mixture.
- [0083] 27. A method for burning a waste product produced by an Andrussov process, comprising:
allowing oxygen, ammonia, and methane to react in the presence of a catalyst comprising platinum to give a product stream comprising at least hydrogen cyanide, hydrogen, and a waste product;
extracting the hydrogen cyanide from the product stream to produce a hydrogen cyanide stream and a gaseous waste stream comprising at least a portion of the hydrogen and the waste product; and
feeding the gaseous waste stream to a hydrogen mode flare.
- [0084] 28. The method of statement 27, further comprising extracting ammonia from the product stream.
- [0085] 29. The method of statement 27 or 23, further comprising separating at least a portion of liquid from the gaseous waste stream prior to feeding the extracted stream to the hydrogen mode flare.
- [0086] 30. The method of any one of statements 27-29, further comprising recovering hydrogen from the gaseous waste stream.
- [0087] 31. The method of any one of statements 27-30, further comprising extracting ammonia prior to extracting the hydrogen cyanide.

[0088] 32. The method of any one of statements 27-31, further comprising flaring the gaseous waste stream at a flare exit velocity of less than about 37.2 meters per second.

[0089] 33. The method of any one of statements 27-32, further comprising flaring the gaseous waste stream at a flare exit velocity of less than about $V_{\max}$, wherein $V_{\max}$ is defined by:

$$V_{\max} = (X_{H_2} - K_1) * K_2, \text{ wherein}$$

$V_{\max}$ = maximum permitted flare exit velocity, meters per second

K_1 = constant, 6.0 volume% hydrogen

K_2 = constant, 3.9 meters per second per volume % hydrogen

X_{H_2} = volume % hydrogen, on a wet basis, as calculated by using the

American Society for Testing and Materials (ASTM) Method D1946-77.

[0090] 34. The method of any one of statements 27-33, further comprising flaring the gaseous waste stream at a temperature of greater than about 1650 °C.

[0091] 35. The method of any one of statements 27-34, further comprising stabilizing the hydrogen mode flare with a flare stabilizer.

[0092] 36. The method of any one of statements 27-35, further comprising increasing a hydrogen concentration of the gaseous waste to increase a heating value of the gaseous waste.

[0093] 37. The method of any one of statements 27-36, further comprising supplementing the gaseous waste with a hydrogen supplement stream.

[0094] 38. The method of statement 36, further comprising increasing an amount of the oxygen allowed to react.

[0095] 39. The method of any one of statements 27-37, further comprising: detecting a hydrogen concentration of the gaseous waste; and increasing an amount of oxygen allowed to react when the hydrogen concentration is detected below a threshold value.

[0096] 40. The method of statement 39, further comprising increasing an oxygen concentration of an air feed stream.

[0097] 41. The system or method of any one or any combination of statements 1-40 is optionally configured such that all elements or options recited are available to use or select from.

CLAIMS

What is claimed is:

1. A system for producing hydrogen cyanide via an Andrussov process, comprising:
a reactor zone wherein oxygen, ammonia, and methane are allowed to react in the presence of a catalyst comprising platinum to provide a product stream comprising at least hydrogen cyanide (HCN), hydrogen, and waste product;
an HCN recovery zone wherein the hydrogen cyanide is substantially removed from the product stream to generate a hydrogen cyanide product and a combustible gas mixture comprising the hydrogen and the waste product; and
a flare zone wherein at least the combustible gas mixture is burned in a hydrogen mode to produce at least carbon dioxide and water.
2. The system of claim 1, wherein a hydrocarbon mixture comprises the methane.
3. The system of claim 2, wherein the hydrocarbon mixture comprises natural gas, biogas, substantially pure methane, or mixtures thereof.
4. The system of any one of claims 1-3, further comprising a hydrogen recovery system connected to the reactor zone that recovers at least some hydrogen from at least one of the product stream and the combustible gas mixture.
5. The system of any one of claims 1-4, further comprising an ammonia recovery system that substantially removes ammonia from the product stream.
6. The system of any one of claims 1-5, wherein the hydrogen cyanide product is at least about 98.5 vol% pure hydrogen cyanide.
7. The system of any one of claims 1-6, wherein the combustible gas mixture comprises at least about 8 vol% hydrogen.

8. The system of any one of claims 1-7, wherein the flare has a flare exit velocity of less than about 37.2 meters per second.

9. The system of any one of claims 1-8, wherein the flare has a flare exit velocity of less than about $V_{\max}$, wherein $V_{\max}$ is defined by:

$$V_{\max} = (X_{H_2} - K_1) * K_2, \text{ wherein}$$

$V_{\max}$ = maximum permitted flare exit velocity, meters per second

K_1 = constant, 6.0 volume% hydrogen

K_2 = constant, 3.9 meters per second per volume % hydrogen

X_{H_2} = volume % hydrogen, on a wet basis, as calculated by using the

American Society for Testing and Materials (ASTM) Method D1946-77.

10. The system of any one of claims 1-9, wherein the flare zone comprises temperatures of greater than about 1350 °C.

11. The system of any one of claims 1-10, wherein the flare zone comprises an unassisted flare.

12. The system of any one of claims 1-11, wherein the flare zone comprises at least a 3.0 inch diameter flare tip.

13. The system of any one of claims 1-12, further comprising a flare stabilizer.

14. The system of any one of claims 1-13, wherein the flare zone further comprises natural gas.

15. The system of any one of claims 1-14, further comprising a hydrogen source that adds hydrogen to the combustible gas mixture.

16. The system of any of claims 1-15, further comprising a detector that quantifies components in the combustible gas mixture.
17. The system of claim 16, further comprising a valve operably linked to the detector, where the valve is configured to permit supplementing the combustible gas mixture with hydrogen or methane when a set value of non-combustible components, corresponding to a lower threshold of heat content, are detected in the combustible gas mixture.
18. The system of claim 16, further comprising a valve operably linked to the detector, where the valve is configured to permit supplementing the oxygen permitted to react when a hydrogen concentration below a threshold value is detected in the combustible gas-mixture.
19. The system of claim 18, wherein the threshold value is based on a heat content value of the combustible gas mixture.
20. The system of claim 18 or 19, wherein the valve is operably linked to an air feed stream.
21. The system of claim 18 or 19, wherein the valve is operably linked to an oxygen feed stream.
22. The system of claim 18 or 19, wherein the valve is operably linked to an oxygen enriched air feed stream.
23. The system of any one of claims 1-22, wherein the flare zone further comprises a non-hydrogen combustible gas mixture burned in a non-hydrogen mode.
24. The system of claim 23, wherein the non-hydrogen combustible gas mixture comprises a heat value of at least about 200 BTU/scf.
25. The system of any one of claims 1-24, wherein the waste product comprises less than about 1.5 vol% HCN.

26. The system of any one of claims 1-25, further comprising at least one liquid separator, upstream of the flare, to remove at least a portion of liquid present in the combustible gas mixture or non-hydrogen combustible gas mixture.
27. A method for burning a waste product produced by an Andrussov process, comprising:
allowing oxygen, ammonia, and methane to react in the presence of a catalyst comprising platinum to give a product stream comprising at least hydrogen cyanide, hydrogen, and a waste product;
extracting the hydrogen cyanide from the product stream to produce a hydrogen cyanide stream and a gaseous waste stream comprising at least a portion of the hydrogen and the waste product; and
feeding the gaseous waste stream to a hydrogen mode flare.
28. The method of claim 27, further comprising extracting ammonia from the product stream.
29. The method of claim 27 or 28, further comprising separating at least a portion of liquid from the gaseous waste stream prior to feeding the extracted stream to the hydrogen mode flare.
30. The method of any one of claims 27-29, further comprising recovering hydrogen from the gaseous waste stream.
31. The method of any one of claims 27-30, further comprising extracting ammonia prior to extracting the hydrogen cyanide.
32. The method of any one of claims 27-31, further comprising flaring the gaseous waste stream at a flare exit velocity of less than about 37.2 meters per second.

33. The method of any one of claims 27-32, further comprising flaring the gaseous waste stream at a flare exit velocity of less than about $V_{\max}$, wherein $V_{\max}$ is defined by:

$$V_{\max} = (X_{H_2} - K_1) * K_2, \text{ wherein}$$

$V_{\max}$ = maximum permitted flare exit velocity, meters per second

K_1 = constant, 6.0 volume% hydrogen

K_2 = constant, 3.9 meters per second per volume % hydrogen

X_{H_2} = volume % hydrogen, on a wet basis, as calculated by using the

American Society for Testing and Materials (ASTM) Method D1946-77.

34. The method of any one of claims 27-33, further comprising flaring the gaseous waste stream at a temperature of greater than about 1650 °C.

35. The method of any one of claims 27-34, further comprising stabilizing the hydrogen mode flare with a flare stabilizer.

36. The method of any one of claims 27-35, further comprising increasing a hydrogen concentration of the gaseous waste to increase a heating value of the gaseous waste.

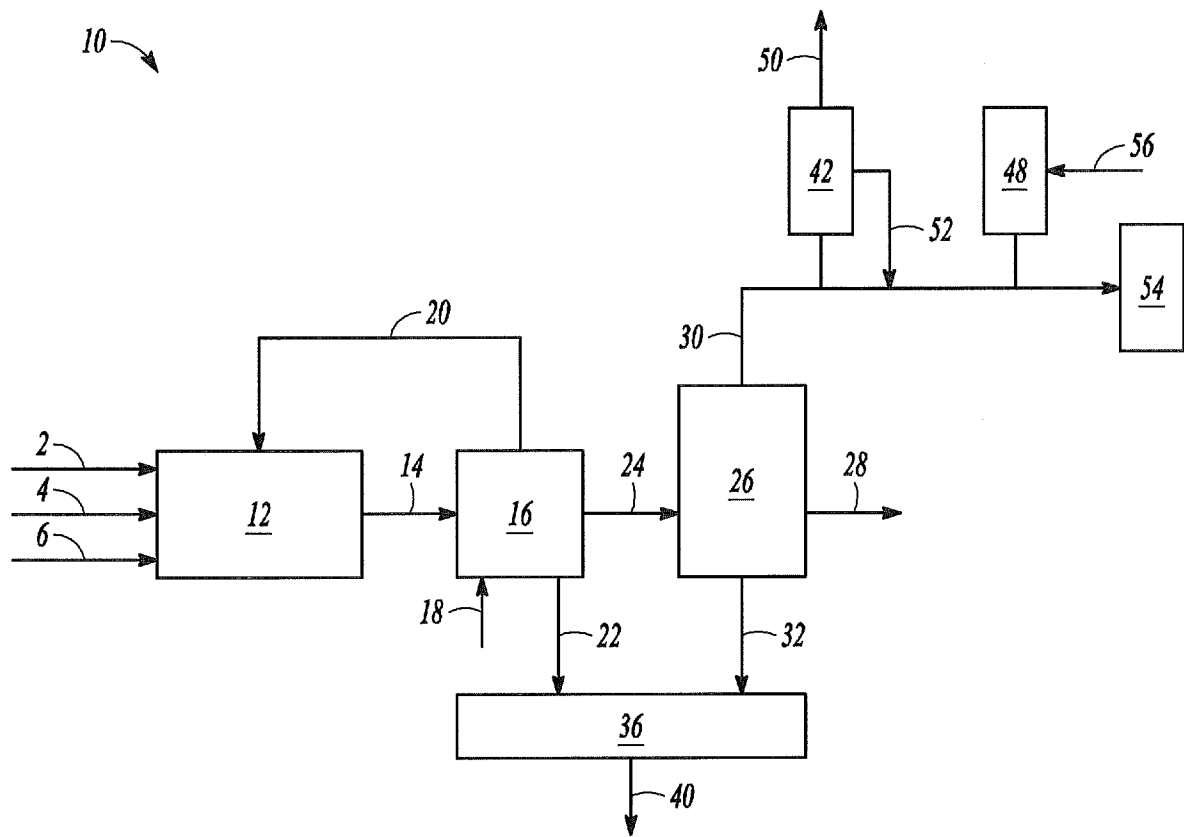
37. The method of any one of claims 27-36, further comprising supplementing the gaseous waste with a hydrogen supplement stream.

38. The method of claim 36, further comprising increasing an amount of the oxygen allowed to react.

39. The method of any one of claims 27-37, further comprising:
detecting a hydrogen concentration of the gaseous waste; and
increasing an amount of oxygen allowed to react when the hydrogen concentration is detected below a threshold value.

40. The method of claim 39, further comprising increasing an oxygen concentration of an air feed stream.

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**FIG. 1**

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2013/074609

A. CLASSIFICATION OF SUBJECT MATTER
 INV. C01C3/02 F23G7/06
 ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C01C F23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, CHEM ABS Data, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Pesce, L.D.: "Cyanides. Kirk-Othmer Encyclopedia of Chemical Technology", 12 March 2010 (2010-03-12), XP002723419, pages 1-29, DOI: 10.1002/0471238961.0325011416051903.a01.pu b2,	1-3, 5-11,14, 23-29,31
A	pages 4-7; figure 1	4,12,13, 15-22, 30,32-40
X	----- WO 2009/075692 A2 (INVISTA TECH SARL [US]; SLATEN COLIN S [US]) 18 June 2009 (2009-06-18)	1,27
A	page 30, line 11 - page 31, line 17; figure 10 ----- -/-	2-16, 28-40



Further documents are listed in the continuation of Box C.



See patent family annex.

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

17 April 2014

Date of mailing of the international search report

12/05/2014

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
 NL - 2280 HV Rijswijk
 Tel. (+31-70) 340-2040,
 Fax: (+31-70) 340-3016

Authorized officer

Werner, Håkan

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2013/074609

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	EP 1 172 137 A2 (ROHM & HAAS [US]) 16 January 2002 (2002-01-16) paragraphs [0010], [0017] - [0019], [0024] - [0032]; figure 1 -----	1,27 2-16, 28-40

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2013/074609

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EP 1172137 A2	16-01-2002	CN 1333083 A EP 1172137 A2 JP 2002119842 A KR 20020006610 A MX PA01007098 A US 2002071798 A1	30-01-2002 16-01-2002 23-04-2002 23-01-2002 13-07-2005 13-06-2002