Innovation, Sciences et Développement économique Canada
Office de la Propriété Intellectuelle du Canada

Innovation, Science and Economic Development Canada
Canadian Intellectual Property Office

CA 2841761 C 2018/03/20

(11)(21) 2 841 761

(12) BREVET CANADIEN
CANADIAN PATENT

(13) C

(86) Date de dépôt PCT/PCT Filing Date: 2012/01/26
(87) Date publication PCT/PCT Publication Date: 2012/08/02
(45) Date de délivrance/Issue Date: 2018/03/20
(85) Entrée phase nationale/National Entry: 2014/01/14
(86) N° demande PCT/PCT Application No.: US 2012/022699
(87) N° publication PCT/PCT Publication No.: 2012/103317
(30) Priorité/Priority: 2011/01/28 (US61/437,583)

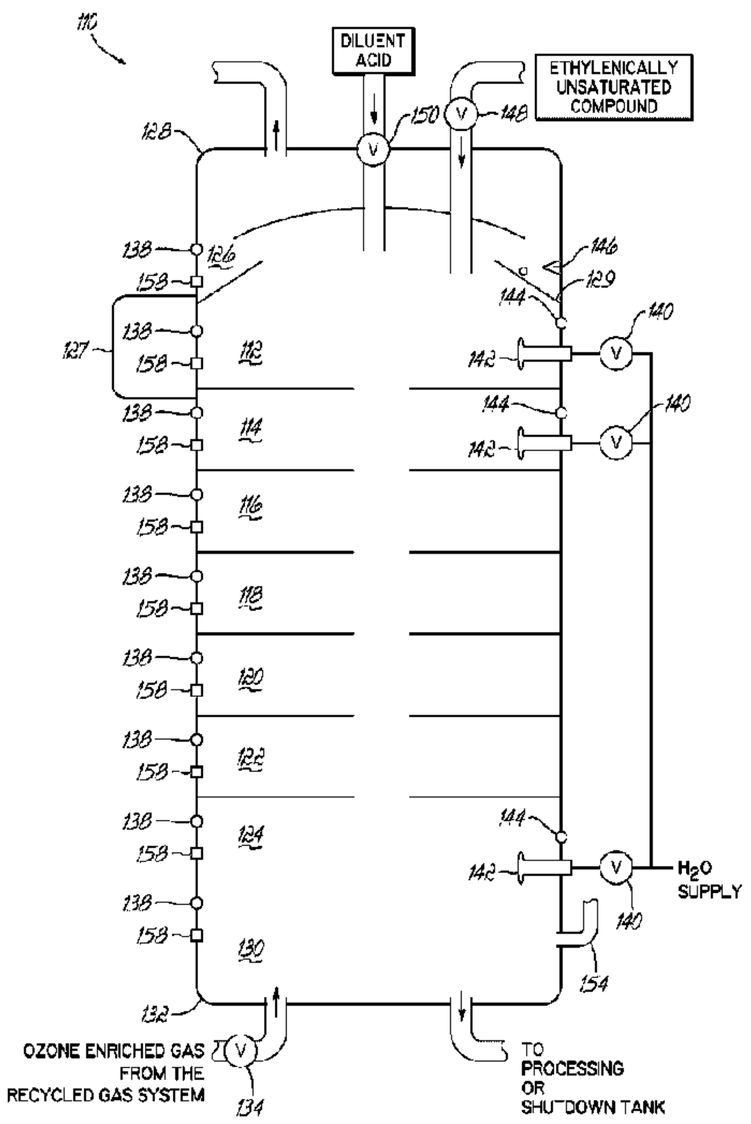
(51) Cl.Int./Int.Cl. *C07B 41/00* (2006.01), *C07C 51/34* (2006.01), *C07C 53/126* (2006.01), *C07C 55/02* (2006.01), *C07C 55/10* (2006.01), *C07C 55/14* (2006.01), *C07C 55/18* (2006.01), *C07C 55/20* (2006.01), *C07C 55/21* (2006.01)

(72) Inventeur/Inventor:
WALKER, THOMAS CHAD, US

(73) Propriétaire/Owner:
EMERY OLEOCHEMICALS LLC, US

(74) Agent: GOWLING WLG (CANADA) LLP

(54) Titre : SYSTEME DE SECURITE POUR COLONNE D'ABSORPTION A OZONE
(54) Title: SAFETY SYSTEM FOR AN OZONE ABSORBER COLUMN



(57) Abrégé/Abstract:
Described herein are improved processes and systems for producing a carboxylic acid mixture that includes a saturated mono carboxylic acid and a saturated dicarboxylic acid. The process includes generating an ozone enriched gas in an ozone generator and introducing the ozone enriched gas to an absorber column. There, the ozone enriched gas is contacted with an unsaturated carboxylic acid feed in the absorber to form an ozonide. The ozonide is contacted with an oxygen enriched gas in a reactor to obtain the carboxylic acid mixture. The carboxylic acid mixture is optionally purified. The process further includes a safety system that monitors operating conditions of the absorber column and responds to those conditions which are able to disrupt the formation of ozonide or generate other materials capable of causing a safety risk should the operating conditions exceed a threshold value.

ABSTRACT

Described herein are improved processes and systems for producing a carboxylic acid mixture that includes a saturated mono carboxylic acid and a saturated dicarboxylic acid. The process includes generating an ozone enriched gas in an ozone generator and introducing the ozone enriched gas to an absorber column. There, the ozone enriched gas is contacted with an unsaturated carboxylic acid feed in the absorber to form an ozonide. The ozonide is contacted with an oxygen enriched gas in a reactor to obtain the carboxylic acid mixture. The carboxylic acid mixture is optionally purified. The process further includes a safety system that monitors operating conditions of the absorber column and responds to those conditions which are able to disrupt the formation of ozonide or generate other materials capable of causing a safety risk should the operating conditions exceed a threshold value.

SAFETY SYSTEM FOR AN OZONE ABSORBER COLUMN

FIELD OF THE INVENTION

[0001] The present invention relates to a safety system for use with an absorber column for ozonizing ethylenically unsaturated compounds.

BACKGROUND OF THE INVENTION

[0002] Azelaic acid and pelargonic acid can be produced in commercial quantities via an oxidative cleavage of an alkenyl unit (i.e., double bond between two carbon atoms) in oleic acid. For example, azelaic acid has been prepared from oleic acid by oxidation with chromium sulfate. However, because stoichiometric use of chromium reagents is undesirable, a more efficient oxidation approach utilizing ozone was developed.

[0003] The basic process is best understood by referring to the description in the accompanying FIG. 1, which is a diagrammatic flow chart indicating the pieces of equipment used and their relationship in the ozonolysis process. This process involves reacting an ethylenically unsaturated compound, such as oleic acid, with ozone in an absorber 13 to form an ozonide. The ozone is provided by a continuous closed system 12 that recirculates and recycles the ozone enriched gas. The ozonide is transferred from the absorber 13 to one or more reaction chambers 37 where it is decomposed in the presence of additional oxygen and optional ozone to form a mixture of compounds including monobasic and dibasic acids, the mixture being referred to as mixed oxidation products (MOP). Monobasic acids and dibasic acids are then separated and individually processed in a series of stills 40 and 52, condensers 43 and 55, extractor 64, and evaporators 70 to remove compounds and undesired fractions among the range of molecular weights of monobasic and dibasic acids produced. The separated and purified monobasic and dibasic acids are then stored in storage tanks 46, 76.

[0004] Ozonized gas is fed into the absorber 13 by a continuous closed system 12 through which the gas circulates. In the closed system 12, the gas is recycled, i.e., the gas is reconditioned for reuse multiple times in the absorber 13. The closed system 12 reconditions the gas by removing organic compounds and water from the gas, restoring the desired oxygen concentration, and generating the desired concentration of ozone.

The closed system 12 maintains the desired predetermined oxygen concentration by bleeding off a small portion of the spent gas and replacing the bled off portion with fresh oxygen gas from an oxygen supply 16. The gas is then passed through a dehydrator 19 before being transferred to an ozone generator 22 which utilizes electricity to generate ozone. From the ozone generator 22, the ozone and oxygen mixture passes to the absorber 13 in which its ozone content is absorbed by the oleic acid as further explained below. From the absorber 13, the oxygen gas, now substantially devoid of ozone, passes to an electrostatic precipitator 25, which removes any contaminating fine mist organic compounds that may have been picked up in the absorber 13. The decontaminated oxygen gas then passes from the electrostatic precipitator 25 through a compression pump 28 to a cooler 31 and then returns back to the dehydrator 19, in which substantially all remaining moisture is removed to complete a pass through the closed system 12. Between the cooler 31 and dehydrator 19, a portion of the gas may be bled from the closed system 12 through a valve 34 to one or more reaction chambers 37 for reaction with the ozonide to form the MOP.

[0005] While the process and apparatus described above generates organic acids such as azelaic and pelargonic acids, from longer chain unsaturated organic acids such as oleic acid, deficiencies exist with respect to personnel safety, system efficiencies and equipment longevity. In particular, the reaction of the ozone with ethylenically unsaturated compounds such as oleic acid to form ozonides is exothermic. Moreover, ozonides are very unstable and can spontaneously decompose and explode if the temperature is not carefully maintained below certain thresholds, such as by controlling the concentration of the reactants and the temperature of the absorber column with coolers. Temperatures within the absorber column can rise above safe operating values due to, for example, a failure in the cooling system or a change in the flow rate or concentration of one or both of the ozone containing gas and the unsaturated fatty acid. To decrease the risk of an accidental explosion of the ozonide, a system that considers the operating temperatures, the flow rates, pressures, and volumes for the reactants and products, and then responds to those parameters in the event they fall outside acceptable ranges is needed to improve the safety of the process.

SUMMARY

[0006] Described herein is an improved process for producing a carboxylic acid mixture comprising a saturated mono carboxylic acid and a saturated dicarboxylic acid.

The process includes generating an ozone enriched gas in an ozone generator and introducing the ozone enriched gas to an absorber column. There, the ozone enriched gas is contacted with an unsaturated carboxylic acid feed in the absorber to form an ozonide. The ozonide is contacted with an oxygen enriched gas in a reactor to obtain the carboxylic acid mixture. The carboxylic acid mixture is optionally purified. The process further includes a safety system that monitors operating conditions of the absorber column and responds to those conditions which are able to disrupt the formation of ozonide or generate other materials capable of causing a safety risk should the operating conditions exceed a threshold value.

BRIEF DESCRIPTION OF THE DRAWINGS

[0007] FIG. 1 is a schematic representation of a prior art oleic acid ozonolysis system.

[0008] FIG. 2 is a schematic representation of the safety system in an exemplary absorber column.

DETAILED DESCRIPTION OF THE INVENTION

[0009] The gas recycling system is used to circulate and recondition depleted gas from an absorber column in which an ozonizing reaction is conducted. The recycling gas system is useful to recondition the depleted gas from the absorber and as shown in Fig. 2, provides a significant improvement in efficiency and safety over the recycling gas system shown in Fig. 1.

[0010] This invention is directed to a safety system that includes sensors, switches, valves, and rupture disks located in the vicinity of an ozone absorber column used in the process of adding ozone across a double bond of an unsaturated organic compound, such as oleic acid. The ozonation reaction forms an ozonide of the unsaturated organic compound at the unsaturated bond. The ozonide leaves the absorber column and is oxidized in a series of oxidation reactions to yield a mixture of a dibasic acid, such as azaleic acid, and a monobasic acid, such as pelargonic acid. The desired monobasic and dibasic acids may be purified from by products and each other in a series of distillation and crystallization steps. The reaction product, i.e., the ozonide, is an extremely unstable species, and can create an explosion risk if the ozonide temperature in the absorber column exceeds prescribed limits. To minimize the hazard to personnel and equipment in the vicinity of the absorber column and to

decrease the down-time associated with having to completely shut down and restart the process, the safety system includes a plurality of valve and switch assemblies arrayed at various locations contiguous to the column to interrupt reactant and product flows, as well as to introduce a water quench into the column under certain conditions. In addition, the safety system includes mechanisms to safely relieve excessive pressure in the absorber column that could result in an explosion.

[0011] The conditions that are monitored in the absorber column 110 include the operating temperatures within the absorber column 110, the flow of reactants through the column 110 (e.g., ethylenically unsaturated compound, such as oleic acid, ozone carried by an oxygen gas (ozone enriched gas), or both), the flow of the product (e.g., ozonide) out of the column, and the operating pressure in the absorber column 110. Each condition is typically monitored independently of the other conditions. If any one of these conditions is observed to meet or exceed the threshold values described in further detail below, then an aspect of the safety system is activated, shutting down the column and decreasing the risk of an explosion.

[0012] The absorber column 110 has separate stages 112, 114, 116, 118, 120, 122, and 124 that optimize the reaction conditions for the reactants, e.g. an unsaturated fatty acid such as oleic acid and ozone, to produce an ozonide. The exemplary absorber column 110 described herein has seven such stages and further includes a trough 126 at the top 128 of the absorber column 110 and a pot 130 at the bottom 132. The unsaturated fatty acid enters the trough 126 at the top 128 of the absorber column 110 and flows down the column 110 through the separate stages 112, 114, 116, 118, 120, 122, 124. The ozone enriched gas has an ozone concentration in the range between about 0.5% to about 12% ozone, enters near the bottom 132 of the absorber column 110 and travels up the absorber column 110 through the separate stages 112, 114, 116, 118, 120, 122, 124, counter-current to the flow of the unsaturated fatty acid. While the exemplary absorber column 110 has seven stages 112, 114, 116, 118, 120, 122, 124 it is understood that absorber columns with greater than or less than seven stages are within the scope of the invention. Accordingly, the number of components for the safety system such as the number of sensors, switches, valves, and rupture disks may similarly be varied and are within the scope of the invention.

[0013] Due to the instability of the mixture of ozone enriched gas and the ethylenically unsaturated compounds, as well as their reaction products (i.e., the

ozonide and its intermediates and by products), the temperature in the reaction column 110 must not be allowed to exceed a critical temperature above which the risk of explosion becomes too great. With this safety concern in mind, the first element of the safety system described herein includes a sensor that monitors the temperature within the column and a switch linked to the sensor stops the supply of the ozone enriched gas to the absorber column 110 in the event that the sensor detects a temperature at any of the stages 112, 114, 116, 118, 120, 122, 124 of the absorber column 110 that is above a first threshold temperature but is less than the critical temperature. The switch causes the ozone generators to shut down by switching off supply of electricity to the generators thereby stopping the production of ozone in the generators. The switch also causes the ozone enriched gas valve 134, which supplies the absorber column 110 with ozone enriched gas, to close. In one embodiment, the first threshold temperature value is between about 100°F and about 140°F as measured at any of the stages 112, 114, 116, 118, 120, 122, 124 in the absorber column 110, in the trough 126, or in the pot 130. Alternatively, the first threshold temperature value is between about 110°F and about 130°F. As another alternative, the threshold temperature value is between about 120°F and about 125°F.

[0014] In the exemplary absorber column 110, the first threshold temperature is monitored and the response is controlled by first temperature monitor 138 located at each stage 112, 114, 116, 118, 120, 122, 124 of the column 110. The first temperature monitor 138 includes a sensor to measure the temperature and a switch to effect the response as needed. The sensor and switch may be included in a single device or may be in discrete devices that are in communication with each other. The first temperature monitors 138 are set to detect if the temperature in any one of the stages 112, 114, 116, 118, 122, 124 of the column 110, or in the trough 126 or pot 130 reaches or exceeds the first threshold temperature. If the first threshold temperature is reached or exceeded as measured by the first temperature monitor 138, this monitor 138 will send a signal to automatically shut down the ozone generators (not shown) by switching off the supply of electricity to the ozone generator and also to close the ozone enriched gas valve 134, thereby preventing a runaway exothermic reaction that could result in an explosion. The first temperature monitor 138 closes an electrical contact in the switch portion of the monitor 138 when the sensor portion detects a temperature in the column 110 that exceeds the first threshold temperature. The ozone enriched gas valve 134 closes automatically in response to the signal from the first temperature monitor 138. The ozone enriched gas valve 134 may be opened and closed by a control valve, such as an

air pressure control valve as is known to those of ordinary skill in the art, that is linked to the first temperature monitor 138.

[0015] The safety system also includes a second temperature monitor 144 that is independent of the first temperature monitor 138. The second temperature monitor 144 detects the temperature of certain stages of the absorber column, such as stages 112, 122, or 124 in the exemplary column, and if the temperature exceeds a second threshold temperature that is greater than the first threshold temperature but less than the critical temperature, all ozone generators will shut down, the ozone enriched gas valve 134 supplying the absorber column 110 will close as described above, and water valves 140 will open to that corresponding stage wherein the second threshold temperature was exceeded, injecting water through a nozzle 142 to cool the absorber column 110 and to quench the reaction in that stage. In one embodiment, the second threshold temperature value is between about 110°F and about 150°F. Alternatively, the second threshold temperature value is between about 120°F and about 140°F. As another alternative, the second threshold temperature value is between about 125°F and about 135°F.

[0016] When the temperature in the monitored stages reaches the second threshold temperature, the risk of a runaway reaction in that stage which can result in explosion is substantially increased. Typically, when the risk of a runaway reaction in a reactor such as an absorber column is too high, i.e., reaches the second threshold, the response is to shut off the supply of reactants to the reactor and to cool the reactor such as with the external application of a coolant. For most runaway or near runaway chemical reactions, cooling agents are not directly added to the reactant because of the risk that the cooling agent could either react with the reactants or rapidly convert to a gas (or steam, as is the case with water) thereby rapidly increasing the pressure in the vessel and increasing the risk of an explosion. However, in the reaction occurring in the absorber column, it was surprisingly observed that the direct addition of water quenches the reaction and decreases the likelihood of a runaway reaction. Accordingly, in some embodiments of the invention, water is injected into the absorber column with the reactants when the temperature in a stage of the absorber column reaches or exceeds a second threshold.

[0017] Water is injected at a rate to quench the reaction and continues to be injected until the temperature reaches the first threshold temperature or below, at

which point water injection is stopped by closing the water valves 140. Typically, the operator of the absorber column 110 will stop the injection of the water by closing the water valves 140. However, automatic systems, such as a computer controlled system (not shown), can also be used. The material in the column 110, i.e., water, reactants, and products, may then be sent to a catch tank (not shown) and processed for recycling back through the system to complete the reaction.

[0018] In addition to the first and second temperature monitors 138, 144 described above, the safety system also monitors and responds to changes in the flow of reactants in the absorber column. One of the reactants is the ozone enriched gas that flows from the recycled gas system to the absorber column. The ozone enriched gas flow safety component is activated if either or both of the pressure or volume of the ozone enriched gas drops below their respective threshold values. An exemplary ozone enriched gas pressure threshold is in the range between about 0 psig to about 15 psig, or, alternatively, in the range between about 5 psig and about 10 psig. If the ozone enriched gas pressure drops to the ozone enriched gas pressure threshold or below, then the ozone generators will shut down and close the ozone enriched gas valve 134 feeding the absorber column 110. The ozone enriched gas pressure is measured by pressure sensors located upstream from the ozone generators. Any gas pressure sensor as known to those skilled in the art may be used. A similar result will follow if the ozone enriched gas flow falls below the ozone enriched gas volume threshold. The ozone enriched gas flow is measured at the inlet of the ozone generator with a flow meter that has a low level trip, i.e., is tripped when the flow drops below the threshold value. Any gas flow meter as known to those skilled in the art may be used.

[0019] In addition to the safety features described above, the safety system described herein also monitors and responds to the flow of liquid reactant through the column. As the liquid reactant and ozone enriched gas pass through the column, some liquid reactant will become entrained as liquid droplets in the ozone enriched gas. The amount of liquid reactant entrained in the ozone enriched gas will increase if the liquid reactant begins backing up in any of the lower stages of the column. Therefore, increased levels of entrained liquid reactant may be used as an indicator of the flow of liquid reactants through the column.

[0020] The trough 126 is part of a system at the top of the column that removes the entrained liquid reactant from the ozone enriched gas. The trough 126 collects the

liquid reactant that has been removed from the ozone enriched gas. The collected liquid reactant is then drained to a lower stage of the column. The drain 127 has a limited capacity to remove collected liquid reactant in the trough 126. Generally, the drain 127 may remove the volume of liquid reactant collected from the ozone enriched gas under normal operating conditions without the liquid reactant pooling in the trough. However, if the volume of liquid reactant entrained in the ozone enriched gas increases, such as will occur if the liquid reactant becomes backed up on a lower stage of the column, then the drain 127 will be unable to remove the increased collected volume of liquid reactant, which will begin to pool in the trough 126. In one embodiment, the safety system monitors the volume of liquid reactant in the trough 126.

[0021] In one embodiment, the volume of liquid reactant in the trough 126 is monitored. If the depth of liquid reactant in the trough 126 meets or exceeds a threshold value, referred to herein as the liquid reactant threshold, a sensor 146 will detect the increased depth of the liquid reactant and generate a signal to shut down the ozone generators as described above, close the ozone enriched gas valve 134, close the ethylenically unsaturated compound valve 148 supplying the column 110, and also close the diluent feed valve 150 used to control the viscosity of the mixture of the ozone enriched gas with the liquid reactant. The liquid reactant threshold can vary depending on the characteristics of the column 110, such as the volume of the trough, size of the drain, etc. In another embodiment, the liquid reactant threshold is a depth of liquid reactant in the trough 126 that is greater than the normal operating depth of liquid reactant in the trough. The liquid reactant threshold can be an increase of at least about 5% of the normal operating depth of the liquid reactant in the trough, depending on the characteristics of the column. The change in depth correlates to a change in the volume of liquid reactant in the trough of at least about 5% over the normal operating volume of the liquid reactant in the trough, depending on the characteristics of the column.

[0022] In an exemplary embodiment, the sensor 146 begins indicating an increasing depth of liquid reactant as soon as the liquid reactant touches the sensor 146, which can be located about 0.25 inches (about 0.6 cm) to about 5 inches (about 12.7 cm) above the bottom 129 of the trough 126. In this embodiment, the threshold is triggered when the sensor detects a change in the depth of the liquid reactant in the range from about 0.25 inches (about 0.6 cm) to about 1.5 inches (about 3.8 cm) above the normal operating depth of the liquid reactant, or about a 5% to about a 600% change in the volume of liquid reactant in the trough collected during normal operating

conditions.

[0023] Any sensor 146 that is able to detect changes in the volume of the liquid reactant may be used, such as a float mechanism, as are known to those of ordinary skill in the art. The term "liquid reactant" is understood to include the mixture of ethylenically unsaturated compound, such as oleic acid, and its reaction products and intermediaries as well as a diluent. Such a diluent includes, but is not limited to, saturated short chain acids such as acetic acid, butanoic acid, caproic acid, heptanoic acid, caprylic acid, pelargonic acid, and capric acid; esters such as ethyl acetate and butyl acetate; and alkanes such as hexane, octane, and decane. However, the use of pelargonic acid is recommended because, as an end product of the process, it does not interfere with the operation of the circulating oxygen system and requires no separate distillation. In other words, since pelargonic acid is one of the end products of the process, it is an ideal diluent.

[0024] Similarly, the safety system also monitors and responds to the flow of the product out of the pot. The term "product" is understood to include the ozonide, unreacted liquid reactants, and diluent. This aspect is monitored as part of the safety system to prevent overfilling the pot 130 and possibly backing up in the column 110 of the product, which can result in an explosion. The flow of product out of the pot 130 may be determined by monitoring the liquid level of the product in the pot 130. The liquid level of the product in the pot may be measured with a differential pressure sensor 154 that measures the head of the liquid, or with a float switch similar to the float switch 146 described above. If the level of product meets or exceeds a threshold level in the pot 130, the safety system will shut down the ozone generators, close the ozone enriched gas valve 134 supplying the absorber column 110 with ozone enriched gas, close the unsaturated carboxylic acid valve 148 supplying the column 110, and also close the diluent feed valve 150. In an exemplary embodiment, sensor 154 in Fig. 2 is used to detect if the level of product in the pot reaches a product threshold level of about 1/3 of the height of the pot 130 or about a height of 22 inches from the bottom of the pot having a diameter at its widest point of about 34 inches. It is understood that the product threshold level can vary depending on the characteristics of the column 110.

[0025] Another component of the safety system described herein are mechanisms for relieving excessive pressure that could result in an explosion in the absorber column. To this end, the safety system also includes rupture disks 158 fitted throughout

the absorber column 110 that are designed to burst if the pressure anywhere in the column 110 exceeds a first threshold operating pressure that is at or below maximum safe operating pressure for the absorber column 110. In one embodiment, the first threshold pressure is in the range between about 20 psig to 60 psig, or more preferably in the range between about 35 psig to 45 psig. The rupture disks can be formed as known in the art from a disk of material, such as a metal, and designed to burst at a specified pressure. When the rupture disks burst, the vented gases and product are discharged into a catch tank. In addition, piping that is associated with the absorber column may also be fitted with rupture disks.

[0026] The mechanism for relieving excessive pressure may also include at least one relief valve (not shown) on the dryer (not shown) upstream of the ozone generators that is set to open if the pressure within the dryer exceeds a second threshold operating pressure. This prevents the pressure within the ozone enriched gas supply from being too great upon entry into the column 110. If the ozone enriched gas pressure is greater than the second threshold operating pressure, then the mixture of the reactants, i.e., ozone and oleic acid, could result in a runaway reaction and explosion. In the exemplary system described herein, the dryer pressure relief valve is set to open if the pressure at the dryer exceeds the second threshold pressure in the range between about 10 psig and about 25 psig, or more preferably between about 15 psig and 20 psig.

[0027] The safety system described herein is useful with an absorber column that generates ozonides of unsaturated acids in an ozonolysis system. As mentioned above, the safety system is particularly suited for use with an ozonolysis system that breaks down oleic acid into pelargonic acid and azelaic acid. However, the safety system may be useful with ozonolysis systems used to break down other unsaturated acids into component carbon chains which form monobasic and dibasic acids via the ozonolysis reaction. The unsaturated acids may generally have between 8 and 30 carbon atoms and one or more unsaturated carbon to carbon bonds. The monobasic and dibasic acid products that result from the ozonolysis reaction are determined by the location of the one or more unsaturated carbon to carbon bonds in the unsaturated acid. The unsaturated acids may be isolated from biological sources, such as plants, animals, or microorganisms. Alternatively, the unsaturated acids may be isolated from petroleum sources and synthetic sources. Exemplary mono unsaturated acids and their respective potential oxidation products are included in the Table below.

Carbons	Exemplary Unsaturated Fatty Acid	Exemplary Monobasic Product	Exemplary Dibasic Product
10	Obtusilic acid	Caproic acid	Succinic acid
10	Caproleic acid	Formic acid	Azelaic acid
11	Undecenoic acid	Formic acid	Sebacic acid
12	Lauric acid	Propionic acid	Azelaic acid
14	Myristoleic acid	Valeric acid	Azelaic acid
16	Palmitoleic acid	Heptanoic acid	Azelaic acid
18	Petroselinic acid	Lauric acid	Adipic acid
18	Oleic acid	Pelargonic acid	Azelaic acid
18	Vaccenic acid	Heptanoic acid	Hendecanedioic acid
18	Octadecenoic acid	Caproic acid	Dodecanedioic acid
20	Gadoleic acid	Undecanoic acid	Azelaic acid
22	Cetoleic acid	Undecanoic acid	Hendecanedioic acid
22	Erucic acid	Pelargonic acid	Brassylic acid
24	Selacholeic acid	Pelargonic acid	Pentadecanedioic acid
26	Hexacosenoic acid	Pelargonic acid	Heptadecanedioic acid
30	Tricosenoic acid	Pelargonic acid	Heneicosanedioic acid

[0028] While the list above includes monounsaturated acids, it is understood that polyunsaturated acids could be utilized as well. The resulting monobasic acids and dibasic acids, and their respective derivatives, may be used for a number of different purposes such as in the preparation of lubricant base stocks, plasticizers, lacquers, herbicides, skin treatments, textile coning oils, flotation agents for mineral refining, fragrances, catalyst scavengers, corrosion inhibitors, metal cleaners, polymerization initiators, lithium complex greases, epoxy flexibilizers, thermosetting unsaturated polyester resins, polyamide hot melts, urethane elastomers, and elastomeric fibers, wire coatings and molding resins.

[0029] It will be appreciated that while the exemplary system described herein utilizes all of the described safety features, it is within the scope of the invention that some of the safety features may be omitted in some embodiments. These and other modifications, methods and apparatus will become readily apparent from this application without departing from the scope of the invention and applicant intends to be bound only by the claims appended hereto.

What is claimed:

1. A process for producing an ozonide from an ethylenically unsaturated fatty acid having between 8 and 30 carbon atoms and one or more unsaturated carbon to carbon double bonds, comprising the steps of

contacting an ozone enriched gas with an ethylenically unsaturated fatty acid in an absorber column having separate stages to form an ozonide; and

operating a safety system comprising:

monitoring an operating condition wherein the operating condition comprises at least an operating temperature, and optionally at least one of an operating pressure of ozone enriched gas, a pressure of ozone enriched gas, the flow of a liquid reactant, and the flow of a product in the absorber column and assigning a value to the monitored operating condition;

comparing the operating condition value with a threshold value for the at least one condition;

affecting the formation of the product in response to the operating condition reaching the threshold value, wherein the operating condition value is a temperature in the absorber column and the threshold value is a first threshold temperature between 100°F (37°C) and 140°F (60°C) and wherein the formation of product in response to the temperature reaching the first threshold value is affected by stopping the introduction of the ozone enriched gas to the absorber column and wherein the threshold value further comprises a second threshold temperature that is greater than the first threshold temperature, and the second threshold temperature is between 110°F (43°C) and 150°F (66°C), and wherein the formation of the product in response to the temperature reaching the second threshold value is affected by direct addition of water into the absorber column.

2. The process of claim 1 wherein the operating condition further comprises the pressure of ozone enriched gas and the threshold value is an ozone enriched gas pressure threshold having a value between 0 psig and 15 psig (103400 pascal).

3. The process of claim 1 or 2 wherein the operating condition further comprises the flow of the liquid reactant and the flow is monitored to detect the volume of the liquid

reactant in the trough of the column; and wherein the threshold value is an increase of the volume of liquid reactant by at least 5% in the trough of the absorber column.

4. The process of any one of claims 1 to 3 wherein the operating condition further comprises the flow of product in the absorber column and is monitored by measuring the level of product near the bottom of the column and the threshold value is an increase in the height of ozonide relative to the bottom of the absorber column.

5. The process of any one of claims 1 to 4 wherein the operating condition further comprises an operating pressure in the absorber column and monitoring the operating pressure in the absorber column and relieving the pressure within the absorber column if the operating pressure exceeds a threshold operating pressure; wherein the operating pressure within the absorber column is monitored with a rupture disk and the excess pressure in the absorber column is relieved by the bursting of the rupture disk.

6. The process of claim 5 wherein the threshold for relieving the pressure is between 20 psig (137900 pascal) and 60 psig (413700 pascal).

7. The process of any one of claims 1 to 6 wherein the operating condition further comprises monitoring the operating pressure of the ozone enriched gas prior to introduction into the absorber column and relieving the pressure of the ozone enriched gas prior to introduction into the absorber column if the operating pressure of the ozone enriched gas exceeds a threshold operating pressure.

8. The process of claim 7 wherein the pressure of the ozone enriched gas is relieved by opening a pressure relief valve and wherein the threshold for relieving the pressure is between 10 psig (68950 pascal) and 25 psig (172400 pascal).

9. The process of any one of claims 1 to 8 wherein the unsaturated fatty acid is oleic acid.

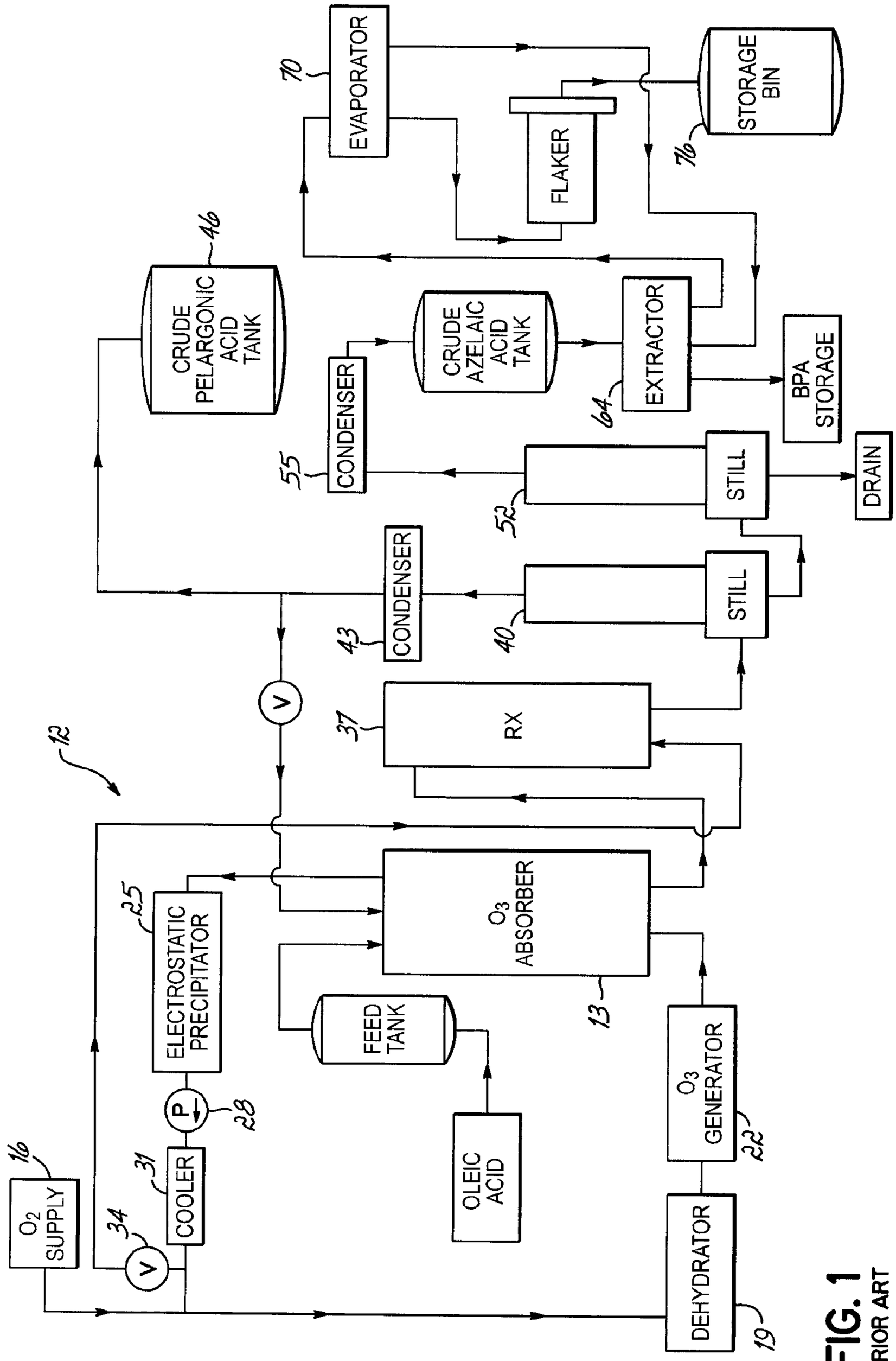


FIG. 1
PRIOR ART

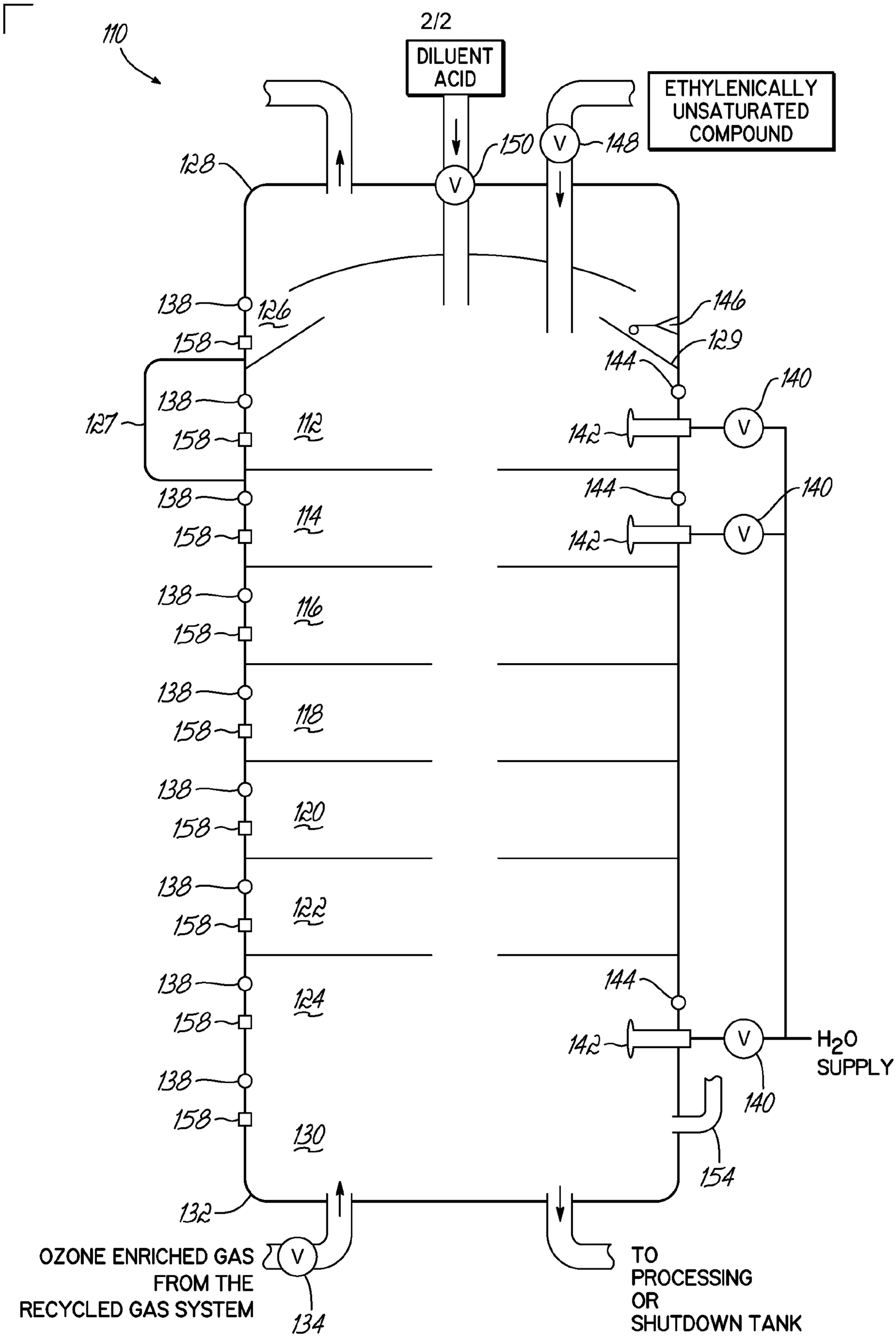
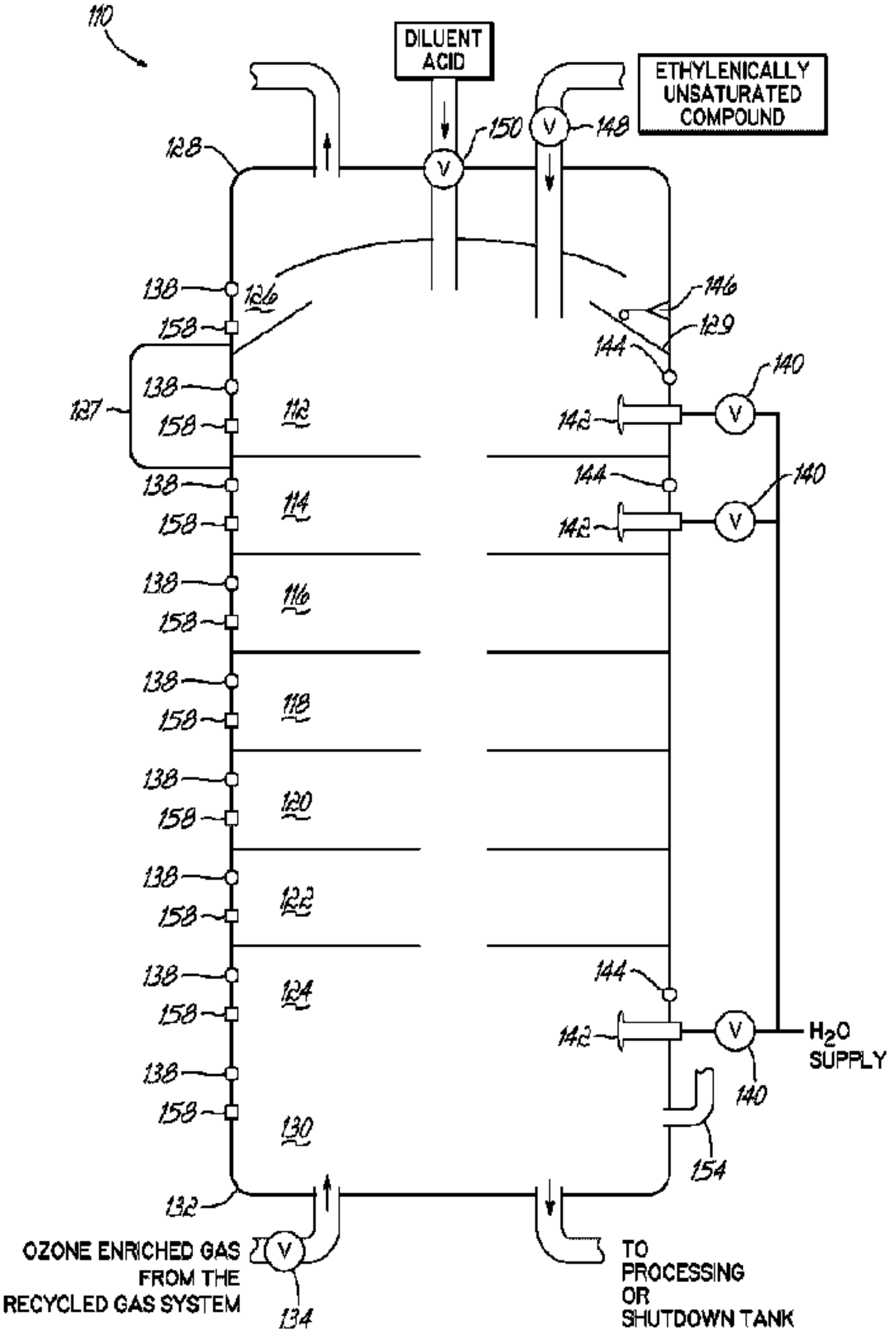

2/2
110
DILUENT ACID
ETHYLENICALLY UNSATURATED COMPOUND
150
148
V
128
138
126
158
146
129
144
140
127
112
142
114
116
118
120
122
124
130
132
134
H_2O SUPPLY
154
OZONE ENRICHED GAS FROM THE RECYCLED GAS SYSTEM
TO PROCESSING OR SHUTDOWN TANK

FIG. 2


110
DILUENT ACID
ETHYLENICALLY UNSATURATED COMPOUND
150
149
128
138
126
158
146
129
144
140
127
112
142
114
116
118
120
122
124
130
132
154
134
H_2O SUPPLY
OZONE ENRICHED GAS FROM THE RECYCLED GAS SYSTEM
TO PROCESSING OR SHUTDOWN TANK