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Solicitud de Patente de Invención titulada **"SINTESIS ELECTROLICA DE ACIDO PERACETICO"**

Solicitante **STERIS INC** -

Yo, **MARGARITA CASTELLANOS ABONDANO**, mayor de edad, identificada y domiciliada como aparece al pié de mi firma, atentamente me permito presentar a usted los dibujos formales y los siguientes documentos

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- Dibujos formales de las figuras 1 a 7

En consecuencia atentamente solicito a Ud se anexe dichos documentos y dibujos formales al expediente correspondiente a esta solicitud de Patente de Invención

Del Señor Jefe,

Margarita Castellanos Abondano

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PROVISIONAL APPLICATION UNDER 37 CFR 1 53 (c)
TRANSMITTAL LETTER

TO THE ASSISTANT COMMISSIONER OF PATENTS
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Entitled

ELECTROLYTIC SYNTHESIS OF PERACETIC ACID

Enclosed are

30 Pages of **SPECIFICATION**

1 Pages of **ABSTRACT**

1 Pages of **CLAIMS**

5 Sheets of **DRAWINGS** (FIGURES 1-5)

X A **CHECK** in the amount of $150 00 for the Provisional Application Filing Fee is enclosed

X The Commissioner is hereby authorized to charge any additional fees which may be required at any time during the prosecution of this application without specific authorization, or to credit any overpayment, to Deposit Account No 06-0308

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government

X No

___ Yes, the name of the U S Government agency and the Government contract number are

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Provisional Application for

ELECTROLYTIC SYNTHESIS OF PERACETIC ACID

Background of the Invention

The present invention relates to the sterilization and disinfection arts It finds particular application in conjunction with electrochemically produced solutions containing oxidizing agents, such as peracetic acid, for sterilization or disinfection of medical and pharmaceutical equipment, food products, and the like, and will be described with particular reference thereto It should be appreciated, however, that the invention is also applicable to other sterilization, disinfection, and sanitization methods, including treatment of water, food service equipment, and the like and has applications in bleaching and in chemical synthesis

Oxidizing agents, such as peracetic acid, and hydrogen peroxide, are useful disinfectants and sterilants for a variety of applications Peracetic acid has a number of uses, including disinfection of waste and sterilization of medical equipment, packaging containers, food processing equipment, and the like Peracetic acid poses few disposal problems because it decomposes to compounds which are readily degraded in sewage treatment plants It has a broad spectrum of activity against microorganisms, and is effective even at low temperatures

Conventionally, to form peracetic acid, concentrated peracetic acid or peracetic acid precursors are mixed with water and other chemicals to form a dilute solution Items to be decontaminated, either by sterilization or disinfection, are then immersed in the solution for a sufficient period to effect the required

level of decontamination The decontaminated items are then typically rinsed before use To ensure effective sterilization or disinfection within a preselected period of time, the concentration of peracetic acid is preferably at or above a minimum effective level, typically around 1000-2000 ppm for sterilization of medical instruments When the peracetic acid concentration is at or above the minimum effective level for sterilization, complete sterilization is expected in the time period for contact Lower levels of peracetic acid are effective as disinfectants Concentrations as low as 2-10 ppm, or less, have been shown to be effective for disinfection, which requires only the destruction of pathogenic microorganisms

In facilities where items are being sterilized or disinfected at frequent intervals throughout the day, the same batch of peracetic acid solution is often used repeatedly However, peracetic acid tends to decompose over time For example, a bath which is above the minimum effective peracetic acid concentration for sterilization at the beginning of a day, frequently drops below the effective concentration without further additions of the concentrated peracetic acid or precursors Elevated ambient temperatures, the quantity of items sterilized or disinfected, and the degree of contamination of the items, all contribute to reducing the useful life of the bath In addition, storage conditions sometimes lead to degradation of the peracetic acid precursors before use

Moreover, the precursors are often hazardous materials which sometimes pose shipment and storage problems Because of the risks of storage and also the fact that they degrade over time, it is preferable to maintain a limited supply of the precursors and reorder them at frequent intervals

Recently, the cleaning and decontamination properties of solutions formed by the electrolysis of water under special conditions have been explored Electrolysis devices are known which receive a supply of water, such as

tap water, commonly doped with a salt, and perform electrolysis on the water During electrolysis, an anolyte solution is produced from the doped water at an anode and a catholyte solution is produced at a cathode Examples of such water electrolysis units are as described in U S Patent Nos 5,635,040, 5,628,888, 5,427,667, 5,334,383, 5,507,932, 5,560,816, and 5,622,848, whose disclosures are incorporated herein by reference

To create these anolyte and catholyte solutions, tap water, often with an added electrically or ionically conducting agent such as halogen salts including the salts sodium chloride and potassium chloride, is passed through an electrolysis unit or module which has at least one anodic chamber and at least one cathodic chamber, generally separated from each other by a partially-permeable barrier An anode contacts the water flowing in the anodic chamber, while a cathode contacts the water flowing in the cathodic chamber The anode and cathode are connected to a source of electrical potential to expose the water to an electrical field The barrier may allow the transfer of selected electron carrying species between the anode and the cathode but limits fluid movement between the anodic and cathodic chambers The salt and minerals naturally present in and/or added to the water undergo oxidation in the anodic chamber and reduction in the cathodic chamber

An anolyte resulting at the anode and a catholyte resulting at the cathode can be withdrawn from the electrolysis unit The anolyte and catholyte may be used individually or as a combination The anolyte has been found to have anti-microbial properties, including anti-viral properties The catholyte has been found to have cleaning properties

However, electrochemically activated water is not without shortcomings Electrochemically activated water has a high surface energy which does not readily allow for penetration of the electrochemically activated water into creviced areas of medical instruments. Thus, complete kill

may not be achieved Further problems have arisen on metal surfaces coming into contact with the electrochemically activated water, including the surfaces of the decontamination equipment and metal medical devices The electrochemically activated water is corrosive to certain metals Stainless steel, used to produce many medical devices, is particularly susceptible to corrosion by electrochemically activated water

Other chemicals are also amenable to electrochemical conversion Khomutov, et al ("Study of the Kinetics of Anodic Processes in Potassium Acetate," Izv. Vyssh. Uchebn. Zaved., Khim. Teknol. 31(11) pp 71-74 (1988)) discloses a study of the conversion of acetate solutions to peracetic acid and acetyl peroxide in the temperature range of -10°C to 20°C using a three-electrode cell The anode and cathode regions of the cell were separated by a barrier of porous glass Anodes of platinum, gold or carbon, at a high potential, typically 2-3 2 V relative to a silver/silver chloride reference electrode, were used in the study Potassium acetate concentrations were initially 2-10 mol/L From conductivity and viscosity measurements, Khomutov, et al estimated that peracetic acid solutions were generated at the anode with concentrations of active oxygen of 0 1 gram equivalents/L However, no direct measurements of peracetic acid concentration in the bulk solution were made Moreover, the pH range of 8.2-10.4 disclosed by Khomutov, et al is undesirable for many practical decontamination solutions To reduce corrosion of the metal components of the instruments to be decontaminated, a pH of close to neutral is desirable.

The present invention provides a new and improved system for generation of peracetic acid and other oxidizing agents which overcomes the above referenced problems and others

Summary of the Invention

Peracetic acid is manufactured in situ, as needed, for a variety of applications, including decontamination of medical devices and treatment of food products In a preferred embodiment, hydrogen peroxide is generated in an electrolysis unit by reduction of oxygen and then reacted with an acetyl donor, such as acetyl salicylic acid, to form peracetic acid

One advantage of the present invention is that it enables peracetic acid solutions to be prepared in situ, as required

Another advantage of the present invention is that it enables antimicrobial solutions to be provided on line or in batch form, on demand

Another advantage of the present invention is that storage and shipment of hazardous sterilants is avoided

Another advantage of the present invention arising from the generation of peracetic acid on demand is that decomposition of peracetic acid during storage is avoided

Another advantage of the present invention is that it enables the concentration of peracetic acid in a microbial decontamination system to be maintained during repeated use of the system

Still further advantages of the present invention will become apparent to those of ordinary skill in the art upon reading and understanding the following detailed description of the preferred embodiments

Brief Description of the Drawings

The invention may take form in various components and arrangements of components, and in various steps and arrangements of steps The drawings are only for purposes of illustrating a preferred embodiment and are not to be construed as limiting the invention

FIGURE 1 is a schematic diagram of a preferred embodiment of an electrolysis unit for generation of sterilizing and disinfecting solutions of the present invention,

FIGURE 2 is a first alternative embodiment of an electrolysis unit according to the present invention,

FIGURE 3 is a plumbing diagram of a sterilization or disinfection system including the electrolysis unit of FIGURE 1, a reagent cup receiving well and a reagent cup,

FIGURE 4 is second alternative embodiment of an electrolysis unit according to the present invention, and

FIGURE 5 is a third alternative embodiment of an electrolysis unit according to the present invention

Detailed Description of the Preferred Embodiments

With reference to FIGURE 1, an electrolysis unit or cell 10 generates oxidizing species for use as liquid sterilants and disinfectants, such as peracetic acid and hydrogen peroxide The unit 10 includes two electrode chambers, namely an anodic chamber 12 and a cathodic chamber 14 An electrode is disposed in each of the chambers Specifically, an anode 16 forms a wall of the anodic chamber and a cathode 18 forms a wall of the cathodic chamber Other walls of the two chambers may be formed from materials which are compatible with peracetic acid, such as insulated steel, plexiglass, or other suitable material An ion selective barrier or membrane 20 connects the anodic and cathodic chambers 12,14 and controls the flow of dissolved species between them In the embodiment of FIGURE 1, the barrier 20 is a cation exchange membrane, which permits the migration of protons (H^+ ions) between the chambers but limits migration of anions between the two chambers The membrane also acts as a separation or barrier between electrolytes in the two chambers Particularly preferred cation exchange membranes are sulfonic acid based membranes Two such proton permeable membranes, NAFION™ 117 and NAFION™ 450, are

available from DuPont and Aldrich. Alternatively, filter paper, such as Fisher brand P-5 filter paper, is used for the membrane 20

Optionally, the membrane is supported on a liquid permeable support wall 22, such as a porous ceramic The support wall provides a physical support for the membrane and permits the movement of liquids and ions to and from the membrane

As will readily be appreciated, the cell 10 may be formed in a variety of configurations For example, the cell 10' is optionally cylindrical, with the membrane 20' and support wall 22' forming a concentric cylinder, which separates the anodic chamber 12' from the cathodic chamber 14', as shown in FIGURE 2 (where like parts are identified with a prime (') The anode 16' is in the form of a rod which is positioned in the center of the anodic chamber The cathode 18' forms an exterior wall of the unit

With reference once more to FIGURE 1, source of electric potential 24 applies a negative potential to the cathode The potential is selected to be high enough for the reduction of oxygen at the cathode A potential of around 3-10 volts, relative to Ag/AgCl in 3M NaCl, is preferred for this purpose, with a particularly preferred potential of around 5V Current densities of from 1 $\mu A/cm^2$ to about 1 A/cm^2 are suitable In one embodiment, the potential is sufficient to provide a current flow of about 5 amps through a cathode of 50 cm^2 in area (100 mA/cm^2)

The cathode chamber 14 is fed with a supply of oxygen, or an oxygen containing gas, such as air As shown in FIGURE 1, the cathode 18 is a gas diffusion electrode which permits the flow of air or other oxygen containing gas therethrough A compressor 28 supplies air under slight pressure to an air chamber 29 adjacent an exterior surface 30 of the gas diffusion electrode 18 The air passes through pores 32 (shown schematically) in the cathode 18 to an air-liquid interface between the air and liquid entering the cathode from the cathode chamber

Excess air is released from the air chamber 29 through an air outlet 34 The outlet preferably includes a regulator valve 35 which controls the outflow of air through the air outlet This allows the pressure of the air in the air chamber to be adjusted to a suitable level for maintaining an air liquid interface within the cathode

An alkaline electrolyte 36 is supplied to the cathode chamber 14 through an inlet 38 The electrolyte carries charge through the cathode chamber An acidic electrolyte 40, is fed to the anode chamber through an inlet 42 While the two electrolytes 36, 40 will be described herein as "acid" and "alkaline", it is to be understood that these terms are used relatively, to indicate a pH differential between the two electrolytes Thus, the "alkaline" electrolyte may be slightly acid or the "acid" electrolyte slightly alkaline In all cases, however, the alkaline electrolyte in the cathode chamber 14 is always at a higher pH (i e , is more basic) than the acid electrolyte in the anode chamber 12 and conversely, the acid electrolyte is always at a lower pH (i e , more acidic) than the alkaline electrolyte In the embodiment of FIGURE 1, the alkaline electrolyte preferably has a pH of about 5-14, more preferably, a pH of 7-9, and most preferably 7-7 5, while the acid electrolyte has a pH of from 0 to about 4, more preferably 0-3, the difference between the alkaline and acid pH preferably providing a pH gradient of about 5-7, or more The acidic electrolyte is thus one which is of sufficient strength to maintain a pH gradient across the membrane A sulphuric acid solution at a concentration of about 5% is a suitable acidic electrolyte and has a pH of about 0 2

The alkaline electrolyte includes a solution of electrolyte ions in water The ions carry charge through the electrolyte Suitable charge carrying ions are provided by salts of alkali and alkaline earth metals, such as phosphates, carbonates, silicates (such as zeolites), and sulfates of sodium and potassium, and other electrolyte

materials which also function as buffers Bases, particularly, hydroxides, such as sodium hydroxide, may also be used Phosphates and silicates are particularly preferred electrolytes as these also provide buffering

A buffering system is preferably added (where not already present as the charge carrying ion) to the alkaline electrolyte to maintain the alkaline electrolyte at an appropriate pH for generating hydrogen peroxide An alkaline electrolyte with a slightly alkaline pH is preferred, preferably around 7-9, most preferably around pH 7-8, although a pH as low as about pH 5 can be used. A combination of buffers and or other compounds may be used to adjust the pH The buffering system may also provide the ionic conductance of the electrolyte, as discussed above

As shown in FIGURE 1, the alkaline electrolyte is delivered to the cathodic chamber inlet from a reservoir or holding tank 50 by a pump 52, gravity feed, or other convenient means The buffering system is preferably added to the alkaline electrolyte reservoir 50 Alternatively, it is metered directly into the chamber, if desired Obviously, the pump 52 and reservoir 50 could be eliminated if a batch process was desired A second pump 54 circulates the sulphuric acid solution through the anode chamber via the anode chamber inlet from a reservoir 56 of sulphuric acid solution Peristaltic pumps are suitable pumps 52, 54 .

The sulphuric acid is essentially unconsumed in the electrolysis process In the embodiment of FIGURE 1, it is circulated through the anode chamber Specifically, the acid electrolyte is withdrawn from the anode chamber through an outlet 58, transported to the reservoir 56 through a return line 60 and then returned to the chamber 12 In this way, a constant flow of the acid electrolyte over the anode is maintained

Alternatively, the acid electrolyte solution is not circulated in to and out of the anode chamber during

the electrolysis, but remains in the anode chamber Optionally, it is stirred in the anode chamber to maintain a flow of electrolyte over the anode The acid electrolyte may be reused for extended periods of continuous peracetic acid generation or for the production of several batches of peracetic acid It may be introduced into the anode chamber or the acid electrolyte reservoir in the form of a sealed ampule, such as a plastic cup, which is opened by an opener member 61 in the reservoir when a lid to the reservoir is closed Optionally, water is added to the reservoir to dilute a concentrated acid solution to form the acid electrolyte

The oxygen is converted at the cathode 18 to peroxide ions, generally as follows

$$O_2 + 2H_2O \rightarrow 2HO_2^- + 2H^+$$

or

$$O_2 + 2OH^- \rightarrow 2HO_2^-$$

or

$$O_2 + H_2O + 2e^- \rightarrow HO_2 + OH^-$$

The peroxide ions react with hydrogen ions or water to form hydrogen peroxide

$$HO_2^- + H^+ \rightarrow H_2O_2$$

or

$$HO_2^- + H_2O \rightleftharpoons H_2O_2 + OH^-$$

Protons (H^+) generated at the anode 16 are able to pass through the membrane 20 into the cathode chamber to take part in the reaction Optionally, a air outlet 62 for the cathode chamber allows air which has bubbled through the electrolyte to leave the chamber The air outlet may include a check valve or pressure relief valve 64 which opens when the chamber pressure reaches a preselected minimum pressure

Temperatures of from about freezing to the boiling point of the electrolyte solution, more preferably about 10-50 °C, most preferably, about 15-30 °C, are suitable for the reactions taking place within the electrolysis unit **10** Accordingly, it is not necessary to heat the anode or cathode chambers

Solutions formed from the electrolyte solutions in the anodic and cathodic chambers during electrolysis are referred to as the anolyte and catholyte, respectively In the embodiment of FIGURE 1, for example, the catholyte comprises a solution of hydrogen peroxide and peroxide ions with phosphate buffers Other oxidizing species may also be present, such as peracetic acid, as will be discussed in greater detail below

The anode **16** and cathode **18** preferably have large surface areas The anode is preferably a dimensionally stable anode (DSA) Suitable materials for the anode include, but are not limited to, carbon (including graphite), platinum, iridium oxide, lead dioxide, palladium oxide, and ruthenium oxide In the case of iridium oxide, lead dioxide, palladium oxide, or ruthenium oxide, the oxide is preferably disposed on a substrate, such as a titanium wire mesh or other noble metal substrate, which supports the oxide and provides the anode with a large surface area Other suitable anode materials include precious metal-doped carbon and other metals, such as precious metal-doped stainless steel, platinum, or palladium Other materials include silver, molybdenum, platinum, or cobalt on a nickel support material, which is in the form of a film sheet, screen, block, foam, or the like

The cathode **18** is formed from any suitable electron acceptor, such as platinum, titanium, gold, or carbon (including graphite) Carbon, such as graphite, is particularly preferred for generation of hydrogen peroxide A suitable gas diffusion electrode includes a substrate, formed from a sheet of a conductive material, such as brass

or nickel, which has a mesh structure with fine pores for admitting the oxygen A layer of carbon is coated on the substrate The mesh provides support for the active material (carbon) of the cathode Cathodes comprising packed carbon beds, carbon frit, and open-celled porous or sintered materials coated with carbon may also be used. Other suitable cathode materials include platinum black (high and low hydrophobicity), platinum/ruthenium on carbon black (high and low hydrophobicity), platinum on carbon black (high and low hydrophobicity), and carbon black

The pressure of the entering air keeps liquids from passing through the gas diffusion cathode into the air chamber 29 Similarly, the pressure of the liquid within the cathode chamber tends to keep gas from passing through the cathode into the cathode chamber. The liquid gas interface within the cathode is where the oxygen reduction reaction takes place

Optionally the anodic chamber is fluidly connected with a reference electrode 66, such as a silver/silver chloride, to ensure that the selected applied potential is being maintained The reference electrode is preferably positioned as close to the anode as possible to eliminate the effects of solution resistance on the measured potential

The catholyte contains hydrogen peroxide and peroxide ions generated in the cathode chamber A peracetic acid precursor, such as an acetyl donor, is reacted with the hydrogen peroxide and peroxide ions in the catholyte solution to form peracetic acid The catholyte may also include a variety of other both short lived and longer living oxidizing species which react directly with the peracetic acid precursor or which participate in reaction paths which lead to the formation of peracetic acid from the precursor.

Suitable acetyl donors include, but are not limited to acetyl salicylic acid, TAED, acetic acid, sodium acetate, potassium acetate, acetic acid, and acetaldehyde

The maximum amount of peracetic acid which may be generated is limited, to some extent, by the solubility of the acetyl donor Thus, highly soluble acetyl donors are preferred A particularly preferred acetyl donor is acetyl salicylic acid

The peracetic acid precursor may be added before, during, or after formation of hydrogen peroxide In one embodiment, shown in FIGURE 1, the peracetic acid precursor is added after formation of hydrogen peroxide and other species Specifically, the catholyte containing hydrogen peroxide is withdrawn from the chamber through an outlet 72 to a holding chamber 74 A pump 76, connected with the outlet 72 is optionally used to pump the catholyte, or, sufficient pressure is supplied by pump 52 to carry the catholyte out of the chamber.

The peracetic acid precursor may be supplied in solid form or as a solution In the embodiment of FIGURE 1, the peracetic acid precursor is supplied to the holding chamber 74 from a reservoir 78 as a solution of the peracetic acid precursor in water

The preferred concentration of the precursor is dependent on the solubility of the precursor and on the desired concentration of the peracetic acid In a preferred embodiment, the precursor is metered into the holding tank in a proportional amount, i e , in a sufficient quantity that all or substantially all of the precursor combines with hydrogen peroxide and or peroxide ions In the case of acetyl salicylic acid as the peracetic acid precursor, the acetyl salicylic acid reacts with the peroxide in a ratio of from about 5 to about 7 1, i e , up to 7 moles of peracetic acid are generated from approximately 7 moles of acetyl salicylic acid and 1 mole of hydrogen peroxide

Optionally, a sensor 80, positioned within the holding tank, or positioned in a fluid line 82 connecting the chamber outlet with the holding tank, or at another suitable location, detects the hydrogen peroxide

concentration The sensor preferably signals a control system 84 which regulates the addition of the peracetic acid precursor by opening and closing a valve 86, between the reservoir 78 and the holding tank 74, or other suitable regulator

A catholyte solution with a hydrogen peroxide concentration of around 2300-2500 ppm is readily obtained in under 5 minutes from the start of applying the potential Adding a peracetic acid precursor to the catholyte in a proportional amount has been found to generate solutions of peracetic acid at concentrations suitable for sterilization or disinfection For example, adding 5g/L of acetyl salicylic acid to 1 L of a 2500 ppm hydrogen peroxide solution yields a peracetic acid solution of about 1800 ppm The resulting peracetic acid solution may be diluted, as desired, to an appropriate concentration for the application in which it is to be used Optionally, less than a proportional amount of the peracetic acid precursor is used so that the resulting antimicrobial solution contains both peracetic acid and hydrogen peroxide as antimicrobial agents

In an alternative embodiment, the peracetic acid precursor, in either liquid or solid form, is mixed with the alkaline electrolyte, prior to electrolysis of oxygen, to form a solution of the alkaline electrolyte and the precursor Specifically, the peracetic acid precursor is mixed with the alkaline electrolyte in the reservoir 50, or supplied directly to the chamber through a second inlet 88 Because acetyl donors, such a acetyl salicylic acid, tend to hydrolyze at high pH (typically above about pH 9) it is preferable, in this embodiment, to buffer the alkaline electrolyte to a pH of below 9, more preferably, around pH 7-8 Where the acetyl donor is combined with the alkaline electrolyte in the reservoir 50 and stored for a period of time, for example, it is desirable to buffer the combined solution within the reservoir to a pH of about 7-8 If the acetyl donor is supplied to the cathode chamber 14

separately from the alkaline electrolyte, the buffering agent may be added directly to the chamber 14 or supplied to the chamber mixed with the peracetic acid precursor In this embodiment, the peracetic acid precursor is available to react with the hydrogen peroxide/peroxide ions as soon as the peroxide is generated However, the reaction may continue to occur even after the catholyte has left the cathode chamber

Other additives may be added to the catholyte solution, either before, during, or after generation of peracetic acid to provide a suitable antimicrobial solution when mixed with the peracetic acid and other oxidants present These additives may include buffers, corrosion inhibitors, surfactants, sequestrants, chelating agents, and the like They may be added in liquid or solid form

Phosphates of alkali metals are suitable buffers for all of the embodiments described herein One preferred buffering system includes a combination of monosodium phosphate, disodium phosphate and tripolyphosphates Such a buffering system also provides anticorrosion properties Another preferred buffering system includes one or more potassium phosphates Other suitable buffers include silicates, such as zeolites The buffering system also serves to buffer the resulting peracetic acid solution to a suitable pH for effective antimicrobial decontamination, as will be described in further detail herein

Corrosion inhibiting and surface energy reducing additives are optionally introduced into the catholyte solution or to the resulting peracetic acid solution, either by adding them to the alkaline electrolyte prior to electrolysis, or during or subsequent thereto Other additives, including, but not limited to, detergents, chelators and sequestering agents, may also be added to the solution, either in combination with the other additives, or separately

The corrosion inhibitory agents are selected in accordance with the nature of the materials in the items

being cleaned and/or decontaminated with the oxidizing species Corrosion inhibitors which protect against corrosion of aluminum and steel, including stainless steel, include phosphates, sulfates, chromates, dichromates, borates, molybdates, vanadates, and tungstates Some additional aluminum corrosion inhibitors include 8-hydroxyquinoline and ortho-phenylphenol

More specifically, phosphates are preferred for inhibiting stainless steel corrosion Preferred phosphates include, but are not limited to, monosodium phosphate (MSP), disodium phosphate (DSP), sodium tripolyphosphate (TSP), sodium hexametaphosphate (HMP), and sodium sulfate either alone or in combination Preferred borates include sodium metaborate ($NaBO_2$)

Copper and brass corrosion inhibitors include triazoles, azoles, benzoates, tolyltriazoles, dimercapto-thiadiazoles, and other five-membered ring compounds Particularly preferred copper and brass corrosion inhibitors include sodium salts of benzotriazole and tolyltriazole which are preferred due to their stability in the presence of strong oxidizing compounds Mercaptobenzothiazole can also be utilized but is apt to be oxidized or destabilized by strong oxidizers Salicylic acid is an example of an acceptable benzoate corrosion inhibitor

In hard water, phosphate buffers and corrosion inhibitors tend to cause calcium and magnesium salts present in the hard water to precipitate and coat the instruments being decontaminated and/or cleaned and also leaves deposits on parts of the electrolysis system In such cases, a sequestering agent appropriate to prevent precipitation such as sodium hexametaphosphate (HMP), or trisodium nitrilotriacetic acid (NTA Na_3) is preferably provided Because sodium hexametaphosphate is also a corrosion inhibitor, it serves a dual purpose, both as a corrosion inhibitor and as a sequestering agent Other sequestering agents include sodium polyacrylates Of

course, if soft or deionized water is utilized, the sequestering agent may be eliminated However, to ensure universal applicability with any water that might be utilized, the presence of a sequestering agent is preferred

A surface energy reducing agent (surfactant) is optionally added to the peracetic acid solution to increase penetration into crevices of items being treated This is particularly important when cleaning and decontaminating complex medical instruments which may contain microbial contaminants in crevices, joints, and lumens Surface energy reducing agents usable in accordance with the present invention include various wetting agents Such wetting agents include anionic, cationic, nonionic, amphoteric, and/or zwitterionic surfactants Specific classes of wetting agents which are useful include anionic and nonionic surfactants or combinations thereof Examples of nonionic wetting agents usable in the present invention include surfactants such as fatty alcohol polyglycol ethers, nonylphenoxypoly (ethyleneoxy) ethanol, and ethoxylated polyoxypropylene Specific examples include Genapol UD-50™, Igepal™, Fluowet™, and Pegol™ The wetting agents set forth above may be used alone or in combination with each other

Amounts of corrosion inhibitor and wetting agents to be added to the peracetic acid solution will vary depending upon the type of agent being added and whether or not one or more agents are added

The inorganic corrosion inhibitors are preferably present in amounts ranging from about 0 01% to 20 0% weight per volume (w/v) Organic corrosion inhibitors are preferably present in amounts ranging from about 0 01% to 5 0% w/v Phosphates are effective at concentrations in the range of about 0 01% to about 11 0% w/v.

The wetting agents are preferably present in amounts ranging from about 0.0001% to about 5 0% w/v More

preferably, the wetting agent is present in amounts ranging from about 0 0001% to about 0.5% w/v

The electrolysis unit 10 thus described is suited to batch or continuous supply processes Dilute solutions of the oxidizing species generated, such as peracetic acid, are advantageously used as antimicrobial solutions for sterilization or disinfection, although the peracetic acid, and or other oxidizing species generated, is optionally used for other purposes In one preferred embodiment, the unit is used for generating batches of peracetic acid solution which can be used immediately, for disinfecting or sterilizing items, or stored for later use

In another embodiment, the unit 10 is used to produce a stream of peracetic acid solution The peracetic acid is carried from the holding tank 74, where present, or directly from the cathode chamber, to a decontamination system 90 or other site at which items to be microbially decontaminated are immersed in, sprayed with, or otherwise contacted with the peracetic acid solution The peracetic acid solution may also be supplied to a vaporizer where it is converted to vapor phase peracetic acid

This embodiment is suited to a variety of purposes, such as decontamination of equipment, including food processing, medical, and pharmaceutical equipment, for disinfecting packaging such as food containers, for removing Listeria and other bacteria from processed food, such as hot dogs and other cooked and uncooked meats and meat products, and for sterilizing waste and water Other applications for the peracetic acid and other oxidants generated include bleaching and as a raw material for a variety of chemical synthesis processes

Another embodiment includes the recirculation of a sterilant or disinfectant solution containing the peracetic acid generated A return line 92 carries the used peracetic acid solution from the site 90 at which items are sterilized or disinfected, to the cathode chamber 14 of the electrolysis unit 10 The peracetic acid

concentration of the solution is increased and then the solution is returned to the site The solution is preferably recirculated in this way until the desired peracetic acid concentration is achieved Once the desired concentration is achieved, the recirculation may be continued intermittently to maintain the desired peracetic acid concentration Alternatively, the solution is recirculated continuously and the potential applied or the compressor operated intermittently to maintain the concentration

A sensor 94 optionally detects the peracetic acid concentration FIGURE 1 shows the sensor positioned in the return line 92 between the site 90 at which sterilization takes place and the electrolysis unit, although other suitable locations in the circulation path are also contemplated The sensor signals the control system, which causes additional peracetic acid to be generated when the concentration is below a predetermined level Other sensors for detecting hydrogen peroxide or other oxidizing species may also be employed The sensors may be disposable or reusable

With reference to FIGURE 3, one embodiment of a system for recirculating a peracetic acid decontaminant solution through a decontamination system includes the electrolysis unit 10 and a microbial decontamination apparatus A, which is configured to sit on a counter top or other convenient work surface. While the system is described herein with particular reference to peracetic acid, it should be appreciated that the solution may also contain other oxidizing species which act as antimicrobial agents, such as hydrogen peroxide

A door or lid 100 of the apparatus A is manually openable to provide access to a tray 102 which defines a receiving region 104 for receiving items to be microbially decontaminated In the illustrated embodiment, the tray 102 is configured to receive devices, such as endoscopes or other long, coilable items Other trays with item

receiving regions of different configurations for receiving the items themselves or item holding containers are also contemplated A well 106 preferably receives a unit dose of reagents for forming a sterilant, disinfectant, or other microbial decontaminating solution In one preferred embodiment, the dose of reagents includes a peracetic acid precursor, preferably in a solid form, such as acetyl salicylic acid and a measured amount of concentrated alkaline electrolyte sufficient to provide the desired concentration on addition of water Alternatively, the peracetic acid precursor, which may be liquid or solid, is added to the electrolysis unit from a vessel (not shown), or introduced by other suitable means

A reagent containing package C, which contains the dose of reagents, is inserted into the well 106. Optionally, the peracetic acid precursor is contained separately from the other reagents within the cup In one preferred embodiment, the cup has a first compartment 110, which holds the peracetic acid precursor and a second compartment 112, which holds the alkaline electrolyte solution Other reagents, such as buffers, corrosion inhibitors, may be contained in one or other of the two compartments 110, 112, or separately added to the well, for example, in a third compartment

In another embodiment, the peracetic acid precursor, buffers, and other additives are all contained in a single compartment cup C.

Once the items are loaded into the tray and the reagent carrying package C is inserted into the well, the lid 100 is closed and latched A cup opener, such as a cutter 114 is disposed at the bottom of the well 106 The cutter 114 cuts the base of the cup, or otherwise creates an opening in the cup, allowing the circulating water to dissolve or entrain the dose of reagents

In a preferred embodiment, the cutter 114 opens the compartment 112, containing the concentrated alkaline electrolyte, buffers, corrosion inhibitors, surfactants,

and the like These are circulated through the system to provide corrosion inhibition and buffer the circulating solution to the desired pH The potential is applied across the cell 10 and generation of hydrogen peroxide and peroxide ions begins Then, the cutter 114 opens the compartment 110 containing the acetyl salicylic acid which circulates through the system and reacts with the hydrogen peroxide and peroxide ions The water and reagents are circulated through the electrolysis unit until a selected concentration of peracetic acid is reached

Optionally, a fill valve 120 passes water through a microbe removing filter 122 in flow paths of a fluid circulating system The microbe removing filter 122 blocks the passage of all particles of around 0 2μ or larger The incoming water, which has passed through the filter, is directed through a spray or distribution nozzle 124 and fills the item receiving region 104 in the tray 102 As additional water is received, it flows into the well 106 dissolving solid reagents, or entraining liquid reagents, in the cup C, forming a solution Filling is continued until all air is forced through an air system 126 and an entire interior volume is filled with the water After the fill valve 120 is closed, a pump 128 circulates the fluid through the item receiving region 104 of the tray, the well 106, the electrolysis unit 10, and, optionally, a heater 130 The pump also forces the anti-microbial solution through the filter 122 to a check valve 132 decontaminating the filter Further, the pump forces the anti-microbial solution through another microbe filter 134 in the air system 126 to a check valve 136 The circulation is continued until sterilization or disinfection is achieved

A peracetic acid concentration sensor 138 optionally senses the concentration of peracetic acid in the decontamination apparatus A In a preferred embodiment, the concentration sensor controls the application of the potential across the anode 16 and cathode 18 or switches the potential off for a period of

time while the peracetic acid remains above a minimum preselected concentration When the peracetic acid concentration drops to the minimum preselected concentration, or below, the voltage is applied to bring the peracetic acid concentration up to the desired level In an alternate embodiment, the concentration sensor controls the valves which direct flow, through and around the electrolysis unit 10 to control concentrations in the decontamination apparatus

When decontamination is complete, a drain valve 140 is opened, allowing the solution to drain from the system Optionally, the drain valve is fluidly connected to the electrolysis unit for carrying the used peracetic acid solution back to the unit for destruction of oxidizing species Air is drawn through the microbe filter 134 such that sterile air replaces the fluid within the system Thereafter, the drain valve is closed and the fill valve 130 opened again to fill the system with a sterile rinse fluid

With reference now to FIGURE 4, another embodiment of an electrolysis unit 210 is shown The unit is similar to that of FIGURE 1, in that it includes two electrode chambers, namely an anodic chamber 212 and a cathodic chamber 214 An anode 216 forms a wall of the anodic chamber and a gas diffusion cathode 218 forms a wall of the cathodic chamber A barrier or membrane 220 connects the anodic and cathodic chambers 212, 214 and controls the flow of dissolved species between them In this embodiment, however, the membrane is an anion exchange membrane, rather than a proton permeable membrane The membrane permits the migration of anions, including OH^- and HO_2^-, between the chambers 212, 214, but limits mixing of other species, such as cations, between the two chambers Suitable anion exchange membranes include ammonium ion membranes obtainable from Tokayama Neosepta, Asahi Glass, Morgane, Pall, Sybron, Electrosynthesis Co , and others, such as AMH and Asahi Glass Model AMP, both obtainable from

Electrosynthesis Co Optionally, the membrane is supported on a liquid permeable support wall 222, such as a porous ceramic

A source of electric potential 224 applies a potential across the anode 216 and cathode 218 A compressor 226 supplies air or pure oxygen to the gas diffusion electrode 218. The air enters the gas diffusion cathode 218 where it reacts with an alkaline electrolyte at the liquid gas interface within the cathode As in the case of the electrolysis unit of FIGURE 1, the applied potential causes the generation of peroxide ions (HO_2^-) at the cathode 218 in the alkaline electrolyte 236 In this embodiment, the alkaline electrolyte includes an alkali, such as sodium or potassium hydroxide, which is supplied to the cathode chamber through an inlet 238 A 0 1M to about 1M sodium hydroxide solution at a pH of 12-14 may be used, although more dilute solutions may also be used

An acidic electrolyte 240 is fed to the anode chamber through an inlet 242 In this embodiment, the acidic electrolyte preferably contains a buffering system, such as described for the embodiment of FIGURE 1, which buffers the pH of the acid electrolyte to a pH of around 5-9, more preferably, to a pH of about 7-8 An acid, such as sulphuric acid, may also be added to the acid electrolyte to adjust the pH, if desired As with the embodiment of FIGURE 1, the pH gradient between the cathode and anode chambers is preferably about 4-9, more preferably, about 5-7

The sodium hydroxide solution (alkaline electrolyte) is delivered to the cathodic chamber inlet 238 from a reservoir 250 by a pump 252, or other convenient means A second pump 254 pumps the acid electrolyte to the anode chamber inlet 242 from a reservoir 256

Suitable temperatures, electric charge, and other operating conditions for the electrolysis unit of FIGURE 4 are as for the embodiment of FIGURE 1, where not expressly stated otherwise

The peroxide ions formed by reduction of oxygen at the cathode migrate through the anion exchange membrane **220** to the anode chamber **212** In this embodiment, a peracetic acid precursor is supplied to the anode chamber (rather than the cathode chamber) where it reacts with the peroxide ions which have passed through the membrane **220**. The peracetic acid precursor may be any of those previously listed and may be added directly to the anode chamber from a precursor reservoir **258** or mixed with the acid electrolyte in the reservoir **256**

In this embodiment, it is the sodium hydroxide, alkaline electrolyte which is essentially unconsumed in the electrolysis process It is withdrawn from the anode chamber **212**, through an outlet **260**, and recirculated to the reservoir **256** through a return line **260** In this way, a constant flow of the alkaline electrolyte over the cathode is maintained The antimicrobial solution containing the peracetic acid formed in the anode chamber is withdrawn from the anode chamber through an outlet **264** and carried by a fluid line to a site **266** at which items are to be decontaminated The decontaminant solution may be recirculated through the anode chamber one or more times to increase the peracetic acid concentration Further additions of the acetyl donor to the recirculating solution may be made from the reservoir **258**, as desired As for the embodiment of FIGURE 1, additives, such as buffers, corrosion inhibitors, surfactants, and the like, may be added, but in this embodiment they are added to the acid electrolyte The additives may be added either prior to, during, or after generation of peracetic acid, in either liquid or solid form In a preferred embodiment, the buffers are added to the reservoir **256** and buffer the acid electrolyte to a pH of about 5-9, more preferably, to a pH of about 7-8 In another embodiment, the buffers, etc, are added directly to the chamber, or mixed with the peracetic acid precursor in reservoir **258**

As will readily be appreciated, the electrolysis unit 210 may be employed in a similar decontamination system to that shown in FIGURE 3, although in this case, the solution circulating through the fluid flow paths, the tray 102, and the well 106, passes through the anode chamber 212 of the electrolysis unit 210, rather than the cathode chamber 214 A multi-compartment cup C may also be used in this embodiment, with the first compartment 112 holding a peracetic acid precursor and the second compartment 110 holding the acidic electrolyte Buffers, corrosion inhibitors, surfactants, sequestering agents, and other additives are disposed in one or other of the compartments 110, 112 or separately added to the well 106

With reference now to FIGURE 5, another embodiment of an electrolysis unit 310 is shown The unit includes a chamber 312 An anode 316 forms a first wall of the chamber and a gas diffusion cathode 318 forms a second wall of the cathodic chamber Suitable anodes and cathodes are as previously described As will be appreciated, no membrane is needed in this embodiment as only one electrolyte chamber 312 is employed

A source of electric potential 324 applies a potential across the anode 316 and cathode 318 A compressor 328 supplies air or pure oxygen to the gas diffusion electrode 318 The air enters the electrode and is reduced at the air liquid interface As in the case of the electrolysis unit of FIGURE 1, the applied potential causes the generation of peroxide ions (HO_2^-) at the cathode 318 in an electrolyte 336 The peroxide and hydrogen peroxide formed react with a peracetic acid precursor to form peracetic acid The peracetic acid precursor is preferably one of those previously listed

In this embodiment, the electrolyte includes a charge carrying species, such as a phosphate, carbonate, or silicate, which may also act as a buffer Preferred buffers include those previously described Acids or bases may also be added to adjust the pH A peracetic acid

precursor, preferably selected from those previously listed, such as acetyl salicylic acid, is added to the electrolyte prior to, during, or after hydrogen peroxide generation

The electrolyte containing the acetyl salicylic acid, or other peracetic acid precursor, preferably has a pH of between about 2 and about 14, more preferably from about 5-9, and most preferably about 7-8 At high pH, generation of peracetic acid occurs rapidly, but the peracetic acid tends to break down fairly rapidly also Additionally, where acetyl salicylic acid is the peracetic acid precursor, it tends to decompose rapidly at at pH over about 9, rendering it ineffective as an acetyl donor. At low pH, peracetic acid generation is much slower, but decomposition of peracetic acid is also slower Thus, the preferred pH is partly dependent on the desired rate of peracetic acid generation, and also on the desired lifetime of the peracetic acid generated, and on the deterioration time for the acetyl donor at the selected pH For some applications, it may be desirable to generate the peracetic acid quickly and then buffer the resultant solution with a buffer, once outside the electrolysis unit

The electrolyte is supplied to the chamber through an inlet 338 from a reservoir 350 by a pump 352, or other convenient means Suitable temperatures, electric charge, and other operating conditions for the electrolysis unit of FIGURE 5 are as for the embodiment of FIGURE 1, where not expressly stated otherwise

The peroxide ions formed by reduction of oxygen at the cathode 318 react with the peracetic acid precursor The peracetic acid precursor may be added directly to the anode chamber from a separate precursor reservoir 353 or mixed with the electrolyte in the reservoir 350

In a preferred embodiment, a two compartment cup C holds buffers, and other additives, such as corrosion inhibitors, sequestrants, and surfactants in a first compartment 354 and a peracetic acid precursor, such as

acetyl salicylic acid, in a second compartment 356 A cup cutter 358, or other suitable cup-opening member, sequentially opens the first and second compartments, releasing the buffers and other additives, then the peracetic acid precursor, into the reservoir A supply of water delivers fresh water to the reservoir through a water inlet line 360 The water mixes with the peracetic acid precursor and other additives to form a solution which is pumped by the pump to the chamber 312 Alternatively, corrosion inhibitors, and other additives are added to the electrolyte after formation of peracetic acid

The antimicrobial solution containing the peracetic acid formed in the chamber is withdrawn from the chamber through an outlet 364 and carried by a fluid line 365 to a site 366 at which items are to be decontaminated The decontaminant solution may be recirculated through the chamber one or more times to increase the peracetic acid concentration As shown in FIGURE 5, a return line 368 carries the used peracetic acid solution back to the chamber 312 from the site 366 Further additions of the acetyl donor to the recirculating solution may be made from the reservoir 350, by opening a valve 370 in the inlet line between the reservoir and the chamber 312, as desired Alternatively, additional precursor may be added from the separate reservoir 353, such as by pumping a metered amount of an aqueous solution of the precursor from the separate reservoir to the chamber

In another embodiment, at least a portion of the circulating peracetic acid is recirculated through the reservoir 350 This helps to ensure that all of the materials in the cup C are washed out of the reservoir 350 and carried to the chamber 312

In another embodiment, the buffers, etc., are added directly to the chamber, or added to the peracetic acid in the supply line 365

Peracetic acid concentration is measured by a sensor 372 placed in contact with the peracetic acid

flowing through the fluid lines The sensor signals a control system 378 which operates valve 370 to add more peracetic acid precursor from reservoir 350 (or from the separate reservoir 353), as needed, to bring the peracetic acid concentration up to a desired level

As will readily be appreciated, the electrolysis unit 310 may be employed in a similar decontamination system to that shown in FIGURE 3, although in this case, the solution circulating through the fluid flow paths, the tray 102, and the well 106, passes through the single chamber 312 of the electrolysis unit 310, rather than through an anode or a cathode chamber

In several applications, it is desirable to maintain the peracetic acid at or slightly above a minimum level for extended periods For example, in the spray treatment of food containers or food stuffs, such as precooked hot dogs, or in other treatment processes where a conveyor system is used to transport items to be sterilized through the antimicrobial spray, it is desirable for the sprayed antimicrobial solution to be recirculated and reused for extended periods, such as hours or even days Even in relatively short term antimicrobial processes, which are effected in an hour or less, such as the sterilization of medical instruments, the presence of bodily fluids or other contaminants may degrade the peracetic acid such that it is desirable to replenish the peracetic acid concentration during the process The peracetic acid solution can be maintained at the preselected level in a number of ways

In one method, the acetyl salicylic acid, or other precursor, is added to the appropriate electrolyte (the acid electrolyte in the embodiment of FIGURE 4, the alkaline electrolyte in the embodiment of FIGURE 1, the single electrolyte in the embodiment of FIGURE 5) in excess Hydrogen peroxide is generated in a sufficient amount to react with a portion of the acetyl salicylic acid and bring the peracetic acid concentration up to the

desired level Then the potential (and/or the supply of oxygen) is switched off When the peracetic acid becomes depleted, the potential is applied (or supply of oxygen switched on) and more peroxide is generated This combines with some of the residual acetyl salicylic acid to form peracetic acid

In a second method, additional acetyl salicylic acid is added to the recirculating peracetic acid solution as needed In this method, the potential may be applied to the electrolysis unit continuously.

In a third method, decomposition products of peracetic acid (such as acetic acid) act as peracetic acid precursors which are reconverted to peracetic acid in the electrolysis unit by reaction with hydrogen peroxide or peroxide ions

While not intended to limit the invention, the following examples are illustrative of the methods of preparing the antimicrobial solutions containing one or more oxidizing agents

EXAMPLES

EXAMPLE 1

Anodic Generation of Peracetic Acid

An alkaline electrolyte for the catholyte was prepared by dissolving 50 g/L Na_2HPO_4 and 5 g/L acetyl salicylic acid in 2 liters of deionized water An acid electrolyte for the anolyte was prepared by diluting 100 mL of concentrated sulphuric acid in 2 liters of deionized water to form a 5% H_2SO_4 solution An electrolysis unit of the type shown in FIGURE 1 was used The cathode was a gas diffusion electrode formed from brass coated with carbon A DSA anode was used The membrane was a Nafion 450 cation exchange membrane Peristaltic pumps were used to pump the two electrolytes from reservoirs to the respective cathode and anode chambers at a flow rate of about 15 mL/min A current of 5 amps (~5 volts) was applied to the cell A compressor supplied air to the cathode The anolyte was

recirculated through the reservoir and anode chamber The catholyte was collected and analyzed with indicator strips at intervals over a period of 45 minutes

Within 30 seconds, a peracetic acid concentration of 10-20 ppm was obtained After 10 minutes, the peracetic acid concentration was about 1500 ppm and the hydrogen peroxide concentration was about 1000 ppm The concentrations of peracetic acid and hydrogen peroxide remained at these levels throughout the 45 minute test

EXAMPLE 2

Generation of Peracetic acid in a Single-Chamber Cell

An electrolyte was prepared by dissolving 50 g/L Na_2HPO_4 and 5 g/L acetyl salicylic acid in 2 liters of deionized water to form a solution having a pH of 7 7 An electrolysis unit of the type shown in FIGURE 5 was used The cathode was a gas diffusion electrode formed from brass coated with carbon A DSA anode was used A peristaltic pumps was used to pump the electrolyte from the reservoir to the chamber at a flow rate of about 17 mL/min A current of 5 amps (~5 volts) was applied to the cell A compressor supplied air to the cathode The electrolyte leaving the chamber was analyzed for peracetic acid with indicator strips at intervals

Within 10 minutes, the peracetic acid concentration was between 1000 and 2000 ppm and the hydrogen peroxide concentration was between 1000 and 2500ppm

The invention has been described with reference to the preferred embodiment Obviously, modifications and alterations will occur to others upon reading and understanding the preceding detailed description It is intended that the invention be construed as including all such modifications and alterations insofar as they come within the scope of the appended claims or the equivalents thereof

Having thus described the preferred embodiment, the invention is now claimed to be

1 A method for preparing an antimicrobial solution containing peracetic acid substantially as described herein

2 An apparatus for preparing an antimicrobial solution containing peracetic acid substantially as described herein.

ELECTROLYTIC SYNTHESIS OF PERACETIC ACID

Abstract of the Disclosure

An electrolysis unit (10, 210) has an ion selective barrier (20, 220) for separating an anodic chamber (12, 212) from a cathodic chamber (14, 214) Air is fed to a gas diffusion cathode (18, 218) to generate hydrogen peroxide and peroxide ions in an alkaline electrolyte 36 flowing through the cathode chamber A peracetic acid precursor, such as acetyl salicylic acid, reacts with the peroxide to form peracetic acid By selecting a proton permeable membrane or an anion exchange membrane for the barrier, the peracetic acid may be formed in either the alkaline electrolyte or in an acid electrolyte flowing through the anode chamber, respectively In one preferred embodiment, a solution containing the peracetic acid is transported to a site where articles, such as medical instruments, are to be decontaminated The oxidizing species are generated as required, avoiding the need to store hazardous decontaminants

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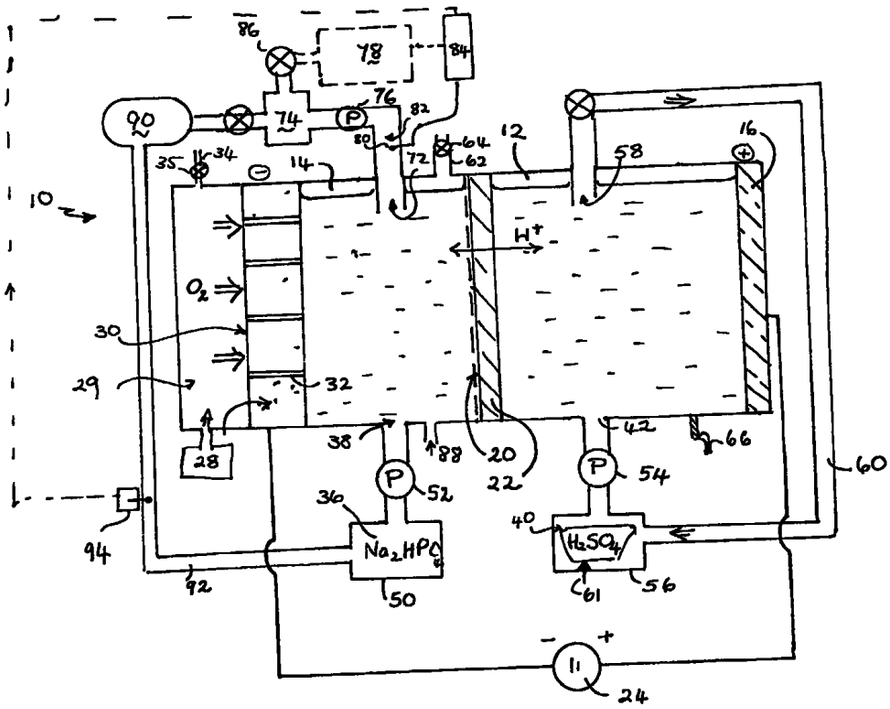
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FIG 1

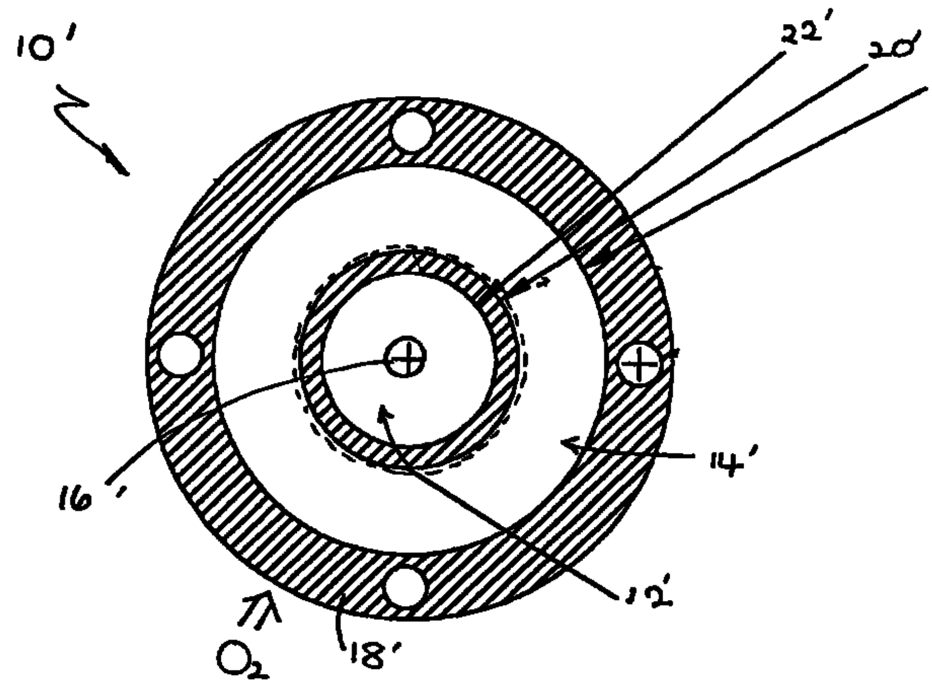


FIG 2

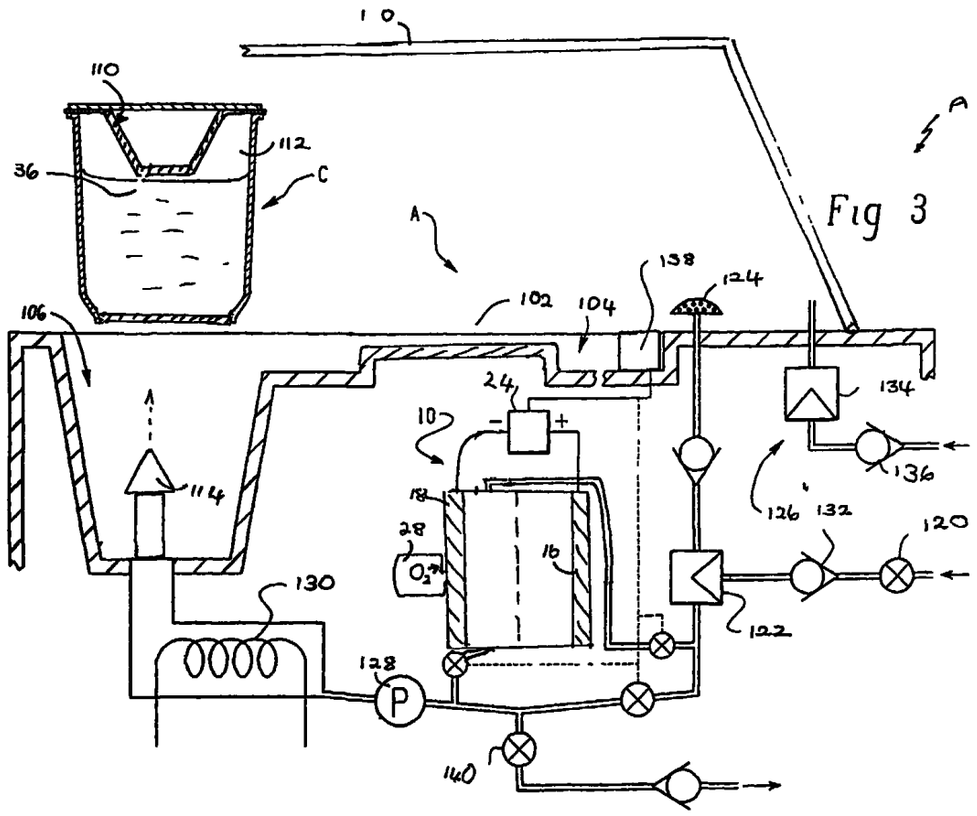
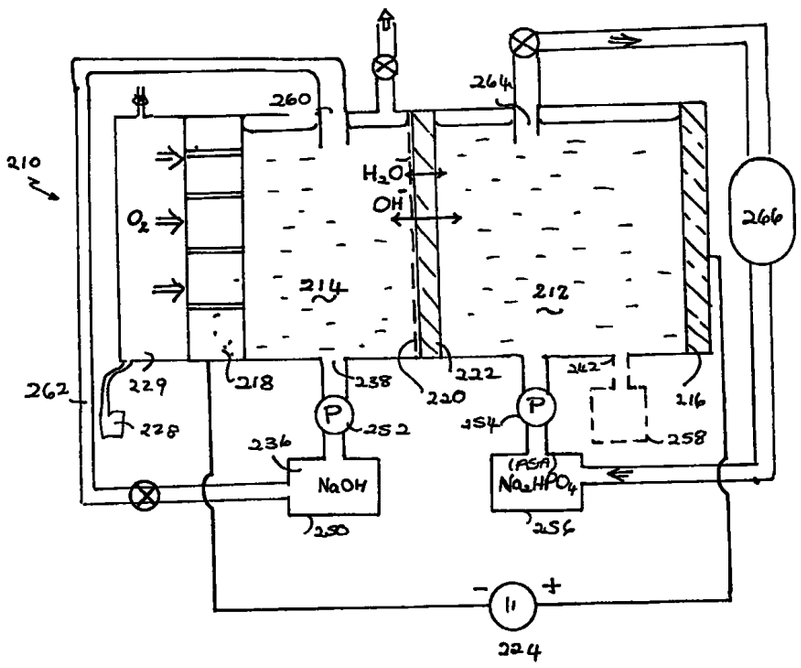


Fig 3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O_2
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228
218
238
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216
236
252
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NaOH
(ASA)
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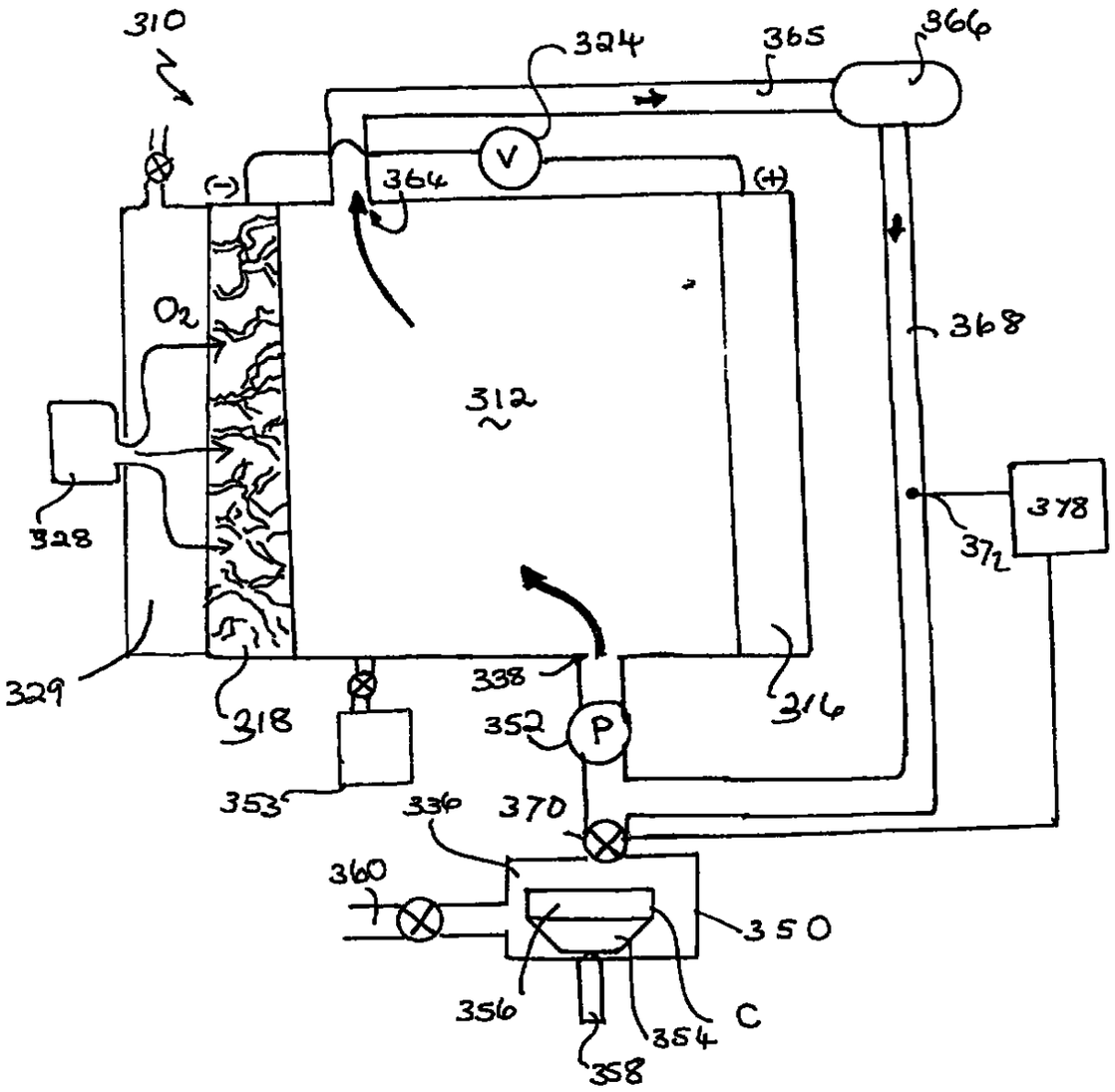
250
256
224

FIG. 4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324
365
366
V
364
(-)
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368
312
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378
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FIG 5

130 44
134

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A TODOS AQUELLOS A QUIENES LA PRESENTE VINIERE

Departamento de Comercio de los Estados Unidos
Oficina de Patentes y Marcas de los Estados Unidos

Agosto 14 del 2000

Por la presente se certifica que lo que obra anexo a ésta es copia fiel de los registros en la Oficina de Patentes y Marcas de los Estados Unidos de aquellos escritos de la solicitud de patente más adelante identificada que reunió los requisitos para que se le concediera una fecha de presentación sobre la base de 35 USC 111

Número de Solicitud. 60/147,327
Fecha de Presentación Agosto 5 de 1999

Por autoridad del
Comisionado de Patentes y Marcas

N WILLIAMS
Funcionario certificante

(Hay un sello-oblea)

131

Numero de Etiqueta del apoderado MED 2 1151 P

SOLICITUD PROVICIONAL BAJO CFR 37 1 53 ©

IMPRESION TRANSMITIDA

POR EL COMISARIO ASSISTENTE DE PATENTES EN WASHINGOTN, D C 20231

Se transmite adjunto la presentacion de solicitud de Patente Provisional de

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Título
SINTESIS ELECTROLITICA DEL ACIDO PERACETICO

Adjunto se encuentra

30	Paginas de la descripción
1	Página del resumen
1	Página de las reivindicaciones
5	Hojas de los dibujos (Figuras 1-5)
x	Un cheque con la cantidad de $150 oo por la tasa de presentación de solicitud provisional se adjunta
X	El comisario está autorizado con la presente para solicitar cualquier tasa adicional que puede requerirse en cualquier momento durante la prosecución de esta solicitud sin especificar autorización o para acreditar cualquier sobre pago , para depositar en la cuenta No 06-0308

La invencion se realizó por un agencia del gobierno de los Estados Unidos o bajo un contrato con una agencia del gobierno de los Estados Unidos

X NO

_ Si, El nombre de la agencia del gobierno de los Estados Unidos y número de contrato son

_____

_____

Todas las llamadas se dirigen a Thomas E Kocovsky, Jr, al teléfono (216) 861-5582

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REIVINDICACIONES

Habiendo así descripto la modalidad preferida, la invención está ahora reivindicada por

1 Un método para preparar una solución antimicrobiana que contiene ácido peracético sustancialmente como se ha descrito

2 Un aparato para preparar una solución antimicrobiana que contiene ácido peracetico sustancialmente como se ha descrito

134

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Señor

Jefe de la Division de Nuevas Creaciones

Superintendencia de Industria y Comercio

Ministerio de Desarrollo Económico

E S D

Re **PODER** conferido por la sociedad **STERIS INC.**

Yo, **XIMENA CASTELLANOS ABONDANO,** *mayor de edad, identificada y domiciliada* como aparece al pié de mi firma, atentamente manifiesto a Uds que sustituyo en la Dra **MARGARITA CASTELLANOS ABONDANO,** el poder que me confirió la sociedad **STERIS INC., en Mentor, Ohio, ESTADOS UNIDOS DE AMERICA, de Agosto 31 del 2.000** Debidamente legalizado por el Ministerio de Relaciones Exteriores el dia **11** de **Octubre** del **2.000** bajo el No **354036** , con las mismas facultades que me fueron conferidas, para tramitar la solicitud de **Patente de Invención titulada :** **" SINTESIS ELECTROLICA DE ACIDO PERACETICO ".**

Del Señor Jefe,

Acepto

Ximena Castellanos Abondano
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T P A No **58 236** de Del Cons Sup de la Judicatura
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Santafé de Bogotá,

DILIGENCIA DE AUTENTICACION DE FIRMA
CON PRESENTACION PERSONAL
(Art 1ro del decreto 22 82 de 1989)

Ante el Notario Cuarenta y uno (41) del Círculo de esta ciudad compareció el Doctor Ximena Castellanos Abondano

quien Exhibió la cédula de ciudadanía número 39773330 expedida en Usaquen y la tarjeta profesional numero 58236 del CSJ. con el presente documento dirigido a Superindustria y declaró que la firma que aparece en él es suya

FIRMA AUTOGRAFA DEL DECLARANTE

Santa Fé de Bogotá D C 11 OCT 2000

AUTORIZO LA ANTERIOR

REPUBLICA DE COLOMBIA Círculo de Santafé de Bogotá D C NOTARIA 41 Carrera 12 No 93 26 AGUSTIN MORALES RIVERA

DILIGENCIA DE AUTENTICACION DE FIRMA
CON PRESENTACION PERSONAL
(Art 1ro del decreto 22 82 de 1989)

Ante el Notario Cuarenta y uno (41) del Círculo de esta ciudad compareció el Doctor Margarita Castellanos Abondano

quien Exhibió la cédula de ciudadanía número 39779684 Usaquen expedida en 39779684 y la tarjeta profesional numero 70819 del CSJ con el presente documento dirigido a Superintendencia y declaró que la firma que aparece en él es suya

Margarita Castellanos

FIRMA AUTOGRAFA DEL DECLARANTE

Santa Fé de Bogotá D C 11 OCT 2000

AUTORIZO LA ANTERIOR

REPUBLICA DE COLOMBIA Círculo de Santafé de Bogotá D C NOTARIA 41 Carrera 12 No 93 26 AGUSTIN MORALES RIVERA

COLOMBIA 4766

MED 2 115J CO

PODER

Yo (nosotros) David C Dvorak, en representación de STERIS INC ,
domiciliado (s) en 43425 Business Park Drive Temecula, CA 92590, Estados Unidos de A
Nombro (amos) a Ximena Castellanos A y/o Margarita Castellanos A

De Santa Fé de Bogotá Colombia apoderados verdaderos (s) legales con poder especial amplio y suficiente para recabar de las oficinas y autoridades colombianas administrativas judiciales y de policia en cualquier instancia o recurso la obtención de patentes de invención registros de marcas modelos y dibujos industriales depósitos de nombres comerciales y enseñas registros de derechos de autor y contratos sobre los mismos así como sus prorrogas renovaciones anotaciones de traspaso cambios de nombre y/o domicilio elaborar tramitar y registrar contratos de licencia y licencias de cualesquiera clases intervenir como demandante o como demandado en cualquier instancia y recurso y ante cualquier juez corporacion funcionario publico o autoridad en acciones judiciales administrativas y de policia tales como oposiciones al registro de marcas acciones de caducidad cancelación y nulidad acciones reivindicatorias acciones derivadas de la obtención o explotación de licencias acciones de competencia desleal demandas penales acciones de indemnizacion de perjuicios reclamos y observaciones a solicitudes de patentes modelos y dibujos industriales en el ejercicio de medidas cautelares y en otras acciones o medidas similares

A este efecto lo (s) faculto (amos) para elevar solicitudes formular descripciones enmiendas protestas declaraciones oposiciones cancelaciones nulidades apelaciones y reclamos pagar impuestos cuotas y gravámenes prescritos por la ley renunciar o desistir a derechos concedidos o en curso de concesión recibir toda clase de documentos y valores dando el descargo respectivo y en general para dar ante dichas autoridades todos los pasos necesarios al efecto indicado y tomar todas las medidas que creyeren conducentes al resguardo de mis (nuestros) intereses con todas las demás facultades legales necesarias

Este poder comprende la facultad de ratificar o confirmar en mi (nuestro) nombre lo actuado así como la facultad de desistir transigir comprometer, y la de sustituirlo en todo o en parte y revocar sustituciones Así mismo nombro (amos) a **Ximena Castellanos A. y/o Margarita Castellanos A.**

mis (nuestros)
representantes con facultades de recibir notificaciones y para designar apoderados judiciales o extrajudiciales

Otorgado y firmado en Mentor, Ohio, EE. UU.A

hoy 31 de Agosto de 49 2000

Firma

Nombre
Cargo

POWER OF ATTORNLY

I (We) David C Dvorak, Senior Vice President, Corporate Sec & Gen Counsel of STERIS INC ,
domiciled in 43425 Business Park Drive
Temecula, CA 92590, USA
Hereby appoint
Ximena Castellanos A y/o Margarita Castellanos A
of Santa Fé de Bogota Colombia my(our) true and lawful attorneys with special ample and sufficient power to obtain from the Colombian administrative judicial and police offices and authorities in any instance privileges of patents of invention registration of trademarks industrial models and drawings registration of commercial names and emblems registration of copyright and contracts related thereto as well as extensions renewals and recordal of assignments and changes of name and domicile related to the above to prepare procecute and register licenses of any kind to act as plaintiff or defendant in any instance or recourse an before any judge corporation public official or authority in judicial administrative and police actions such as oppositions to the registration of trademarks caducity cancellation and nullity actions claim actions derived from the obtaining or explotation of licenses actions of unlair competition penal demands actions of indemnities for damages claims or observations on patent applications models and industrial designs in the excercise of preventive measures and in other actions or similar proceedings

To that effect they hereby empowered to file applications formulate descriptions amendments protests , declarations oppositions cancellations nullities appeals and claims to pay taxes quotas and assessments prescribed by law renounce or waive the rights granted or in the process of concesion to receive all kinds of documents and valuables giving the respective release of receipt and in general to take before said authorities the necessary steps to such effect and any measures that they may deem pertinent to the safeguard of my (our) interest(s) with all the other necessary legal powers

This power includes the facuity to ratify or confirm desist settle and compromise on my (our) behalf and to substitute this power in whole or in part and to revoke substitutions At the same time I(we) hereby appoint
Ximena Castellanos A y/o Margarita Castellanos A.
my(our)
representative(s) in
with powers to receive notifications and to designate judicial and extrajudicial attorneys

Granted and signed in Mentor, Ohio USA

this 31st day August of 49 2000

Signature

Name David C Dvorak
Title Senior Vice President, Corporate Secretary & General Counsel STERIS INC

Esta copia coincide con el original que tuvo a la vista Santa Fé de Bogotá
18 OCT 2000
AGUSTIN MORALES RIVEIRA
NOTARIO CINCUENTA Y UNO

136

The State of Ohio, Lake County } ss

I, Lynne L Mazeika, Clerk of the Court of Common Pleas a Court of Record of Lake County, aforesaid,

Do Hereby Certify That Robert J. Bertolo

before whom the annexed acknowledgment, oath, affidavit, was taken, was at the date thereof a NOTARY PUBLIC, in and for said County, duly authorized by the laws of Ohio to take the same, also to make acknowledgments, affidavits and proofs, of deeds or conveyances for land tenements or hereditaments situated and lying in said State of Ohio, and further that I am well acquainted with his handwriting and believe his signature thereto is genuine, and that the annexed instrument is executed according to the laws of the State of Ohio

Commission expires March 1, 2001

In Testimony Whereof, I hereunto subscribe my name and affix the seal of said Court, at Painesville, Ohio, this 5th day of September A.D. 1990

A 90-91492

Lynne L. Mazeika, CLERK OF COURTS

Lynne L. Mazeika Clerk

BARRETT BROTHERS SPRINGFIELD, OHIO 1 800-322 7711

Esta copia coincide con el original que se ha visto Santa

18 OCT 2000

United States of America

DEPARTMENT OF STATE

To all to whom these presents shall come, Greetings:

I Certify That the document hereunto annexed is under the Seal of the Secretary of State of the State(s) of Ohio, and that such Seal(s) is/are entitled to full faith and credit *

In testimony whereof, I, Strobe Talbott, Acting Secretary of State , have hereunto caused the seal of the Department of State to be affixed and my name subscribed by the Assistant Authentication Officer, of the said Department, at the city of Washington, in the District of Columbia, this sixteenth day of September, 2000

Acting Secretary of State

By

Assistant Authentication Officer,
Department of State

Issued pursuant to CHXIV Stats of Sept 15 1789 1 Stat 68-69 22 USC 2657 22 USC 2651a 5 USC 301 28 USE 1733 et seq 8 USC 1443(f), RULE 44 Federal Rules of Civil Procedure.

*For the contents of the annexed document,the Department assumes no responsibility

This certificate is not valid if it is removed or altered in any way whatsoever

Esta copia coincide con el original que tuve a la vista Santa Fe de Bogotá

18 OCT 2000

ACUSTA
NOTA

133 $2

United States of America

State of Ohio

Office of the Secretary of State

I ***J KENNETH BLACKWELL,*** *Secretary of State do hereby certify that I am the duly elected qualified and acting Secretary of State of the State of Ohio and I further certify that* LYNNE L MAZEIKA *whose signature and official seal are affixed to the attestation hereto attached, was at the date hereof, the duly elected, commissioned and qualified* CLERK OF THE COURT OF COMMON PLEAS OF LAKE COUNTY, OHIO, *and that she is the proper official to make said attestation, which is in due form, and that her acts are entitled to full faith and credit*

IN TESTIMONY WHEREOF I have hereunto subscribed my name and affixed the official Seal of the Secretary of State of Ohio at Columbus Ohio this 11th *day of* September 2000

DORA PUQUE DE PRIJAL

J Kenneth Blackwell

J Kenneth Blackwell

Secretary of State

SEC4000 (Rev. 1/99)

Esta copia coincide con el original que tuve a la vista Santa Fe de Bogotá

18 OCT 2000

24030

Consulado General de Colombia

500 NORTH MICHIGAN AVENUE SUITE 2040
CHICAGO ILLINOIS 60611

TEL (312)923 1196 FAX(312)923 1197

No 1458

Chicago, septiembre 21 de 2000

El suscrito Cónsul General de Colombia, **CERTIFICA**, que el señor(a) **J KENNETH BLACKWELL**, quien como **SECRETARIA DEL ESTADO DE OHIO**, autenticó la firma del signatario del poder precedente, ejercía en la fecha las funciones indicadas y su firma corresponde a la registrada en este Consulado Que la compañía **STERIS INC** , es una sociedad constituida de acuerdo con las leyes de EE UU y cumple su objeto conforme a las mismas Que el(la) señor(a) **DAVID C DVORAK**, quien en su carácter de representante legal de la citada compañía confiere el poder precedente

La presente certificación se expide en cumplimiento del artículo 480 del Código de Comercio

El Cónsul General no asume responsabilidad alguna por el contenido del documento

PAGADO IMPUESTO DE TIMBRE Y DERECHOS CONSULARES
US$57 oo (Cincuenta y siete dólares)

CONSULADO GENERAL DE COLOMBIA ★ CHICAGO ★

JOSE FERNANDO GOMEZ MORA
Cónsul General de Colombia

MINISTERIO DE RELACIONES EXTERIORES
354036
CUYA FIRMA APARECE EN EL DOCUMENTO DESEMPEÑA LAS FUNCIONES INDICADAS

JEFE DE LEGALIZACIONES
SANTAFE DE BOGOTA D C
2000 OCT 11

NO SE ASUME RESPONSABILIDAD DEL CONTENIDO DEL TEXTO

Esta copia coincide con el original que tuve a la vista Santa fé de Bogotá
18 OCT
ACUSTIN NORALES PEREIRA
NOTARIO CUARENTA Y UNO

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140 X44

----------TRADUCCION OFICIAL No 2K-1005----------

de un documento escrito en INGLES, el cual para su identificación lleva el sello de la Traductora e Intérprete Oficial Res 2755/68 y 01144/97 de Minjusticia, al igual que la presente ----------

(Anexos a un Poder otorgado por STERIS INC)

ESTADO DE OHIO

CONDADO DE LAKE

Yo, Lynne L Mazeika, Escribano de la Corte Ordinaria, una Corte de Registro del Condado de Lakes antes mencionado, certifico por medio del presente que ROBERTA J BERTON, ante quien el instrumento, juramento o declaración anexo fue tomado, era en la fecha del mismo un Notario Publico en y para dicho Condado debidamente autorizado por las leyes de Ohio para tomar el mismo, y para tomar reconocimientos, declaraciones y pruebas de escrituras o traspasos de terrenos, tenencias o heredamientos situados y que queden en dicho Estado de Ohio, y además que conozco su escritura y verdaderamente creo que su firma en el mismo es auténtica, y que el instrumento anexo está otorgado de acuerdo con las leyes del Estado de Ohio-

La comisión vence el 11 de marzo de 2001

En testimonio de lo cual, he impuesto aquí mi firma y he fijado el sello de dicha Corte en la ciudad de Painesville, Ohio hoy día 5 de Septiembre de 2000

(fdo) Lynne L Mazeika, Escribano de las Cortes

Lynne L Mazeika, Escribano

No A 90-91492 (Sello en relieve)

Esta copia coincide con el original que tuve a la vista Santa fé de Bogotá

18 OCT 2000

AGUSTIN MORALES RIVEIRA
NOTARIO CUARENTA Y UNO

141

ESTADOS UNIDOS DE AMERICA

ESTADO DE OHIO

OFICINA DEL SECRETARIO DE ESTADO

Yo, J KENNETH BLACKWELL, Secretario de Estado certifico por medio del presente que soy el Secretario de estado del Estado de Ohio debidamente elegido, habilitado y en ejercicio, y certifico además que LYNNE L MAZEIKA cuya firma y sello oficial están impuestos en la certificacion adjunta al presente, era en la fecha de la misma el ESCRIBANO DE LA CORTE ORDINARIA DEL CONDADO DE LAKE, OHIO, debidamente elegido comisionado y habilitado, y que ella es la funcionaria competente para hacer dicha certificación, la cual está en debida forma y que sus actos como tal tienen derecho a plena fe y crédito

EN TESTIMONIO DE LO CUAL, he suscrito aquí mi nombre y he fijado el sello oficial del Secretario de estado de Ohio, en Columbus, Ohio, el dia 11 de Septiembre de 2000

(fdo) J Kenneth Blackwell

J KENNETH BLACKWELL

Secretario de Estado

(Sello en oblea ocre)

00046477-4

ESTADOS UNIDOS DE AMERICA

DEPARTAMENTO DE ESTADO

A TODOS A QUIENES LOS PRESENTES LLEGAREN, SALUDO

Esta copia coincide con el original que tuve a la vista Santa Fe de Bogotá

18 OCT 2000

142 S/6 1/46

Certifico que el documento anexo al presente está bajo el sello del Secretario de Estado del Estado de Ohio, y que tal sello merece plena fe y crédito *

En testimonio de lo cual, yo, Strobe Talbott, Secretario de Estado Encargado, he hecho fijar al presente el sello del Departamento de Estado y he hecho suscribir mi nombre por el Funcionario de Autenticación de dicho Departamento, en la ciudad de Washington, en el Distrito de Columbia, hoy día dieciseis de Septiembre de 2000

(fdo) Strobe Talbott

Secretario de Estado Encargado

Por (fdo) ilegible

Funcionario de Autenticación Asistente

Departamento de Estado

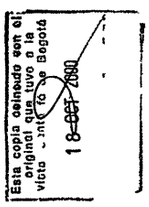
- El Depto no asume ninguna responsabilidad por el contenido de los documentos anexos
- Este certificado no es válido si se remueve o altera de otra forma

---

EN ESPAÑOL, la legalización de la firma de J Kenneth Blackwell por el Consulado de Colombia en Chicago, No 1458 de fecha Septiembre 21, 2000 y autenticación de la firma consular de José Fernando Gomez Mora por el Ministerio de Relaciones Exteriores de Colombia bajo el numero 354036 de Octubre 11, 2000

ES TRADUCCION FIEL Y COMPLETA, a la cual me remito para su confrontación Bogotá, Octubre 12, 2000

Esta copia coincide con el original que tuve a la vista Santa fe de Bogotá 18 OCT 2000 AGUSTIN ... NOTARIO ...



MINISTERIO DE RELACIONES EXTERIORES
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CUYA FIRMA APARECE EN EL DOCUMENTO DESEMPEÑA LAS FUNCIONES INDICADAS

NO SE ASUME RESPONSABILIDAD DEL CONTENIDO

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JEFE DE LEGALIZACIONES
SANTAFE DE BOGOTA D C

4766

United States of America

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DEPARTMENT OF STATE

To all to whom these presents shall come, Greetings:

I Certify That the document hereunto annexed is under the Seal of the Secretary of State of the State(s) of Ohio, and that such Seal(s) is/are entitled to full faith and credit *

In testimony whereof, I, Strobe Talbott, Acting Secretary of State , have hereunto caused the seal of the Department of State to be affixed and my name subscribed by the Assistant Authentication Officer, of the said Department, at the city of Washington, in the District of Columbia, this sixteenth day of September, 2000

DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
RES 02753/68 Y 01144/97
REP DE COLOMBIA

Acting Secretary of State

By _____
Assistant Authentication Officer,
Department of State

Issued pursuant to CHXIV State of Sept. 15 1789 1 Stat 68–69 22 USC 2657 22 USC 2651a, 5 USC 301 28 USE 1733 et seq 8 USC (443(f) RULE 44 Federal Rules of Civil Procedure.

*For the contents of the annexed document,the Department assumes no responsibility.

This certificate is not valid if it is removed or altered in any way whatsoever

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Consulado General de Colombia

500 NORTH MICHIGAN AVENUE SUITE 2040
CHICAGO ILLINOIS 60611

TEL. (312) 923 1196 FAX (312) 923 1197

MINISTERIO DE RELACIONES EXTERIORES
CUYA FIRMA APARECE EN EL DOCUMENTO DESEMPEÑA LAS FUNCIONES INDICADAS
540034

No 3147 Chicago, SEP 22 2000

El suscrito Cónsul General de Colombia, CERTIFICA que el señor Kenneth Blackwell _____, quien autoriza el presente documento, ejercía legalmente, en la fecha allí expresada, las funciones de Secretario de Estado de Ohio, y que la firma y sello que en el documento aparecen como suyos son los que usa y acostumbra en sus actos oficiales

El Cónsul no asume responsabilidad alguna por el contenido del documento

NO SE ASUME RESPONSABILIDAD DEL TEXTO
JEFE DE LEGALIZACIONES SANTAFE DE BOGOTA D.C.
00 OCT 11 4 45

DERECHOS DE TIMBRE Y DERECHOS CONSULARES
(VEINTIDOS DOLARES US$22 00)

JOSE FERNANDO GOMEZ MORA
Cónsul General de Colombia

CONSULADO GENERAL DE COLOMBIA

145 SB DA9

United States of America

State of Ohio

Office of the Secretary of State

I ***J KENNETH BLACKWELL,*** *Secretary of State do hereby certify that I am the duly elected qualified and acting Secretary of State of the State of Ohio, and I further certify that* LYNNE L MAZEIKA *whose signature and official seal are affixed to the attestation hereto attached, was at the date hereof, the duly elected, commissioned and qualified* CLERK OF THE COURT OF COMMON PLEAS OF LAKE COUNTY, OHIO, *and that she is the proper official to make said attestation, which is in due form, and that her acts are entitled to full faith and credit*

IN TESTIMONY WHEREOF I have hereunto subscribed my name and affixed the official Seal of the Secretary of State of Ohio at Columbus Ohio this 11th *day of* September 2000

J Kenneth Blackwell
Secretary of State

SEC4000 (Rev 1/99)

DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
RES 02755/98 Y 01144/97 REPUBLICA
REP DE COLOMBIA

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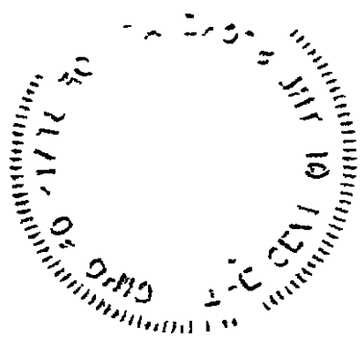
CONSULADO GENERAL DE



The State of Ohio, Lake County } ss

I, Lynne L Mazeika, Clerk of the Court of Common Pleas, a Court of Record of Lake County, aforesaid,

Do Hereby Certify That Roberto J. Bertoni

before whom the annexed acknowledgment, oath, affidavit, was taken, was at the date thereof a NOTARY PUBLIC, in and for said County, duly authorized by the laws of Ohio to take the same, also to make acknowledgments, affidavits and proofs, of deeds or conveyances for land tenements or hereditaments situated and lying in said State of Ohio, and further that I am well acquainted with his handwriting and believe his signature thereto is genuine, and that the annexed instrument is executed according to the laws of the State of Ohio

Commission expires March 11, 2001

In Testimony Whereof, I hereunto subscribe my name and affix the seal of said Court, at Painesville, Ohio, this 5th day of September A D 1998

Lynne L Mazeika, CLERK OF COURTS

Lynne L Mazeika Clerk

A 90-97 DORA DUQUE DE BERNAL
INTERPRETE OFICIAL
RES 02755/68 Y 01144/97 CHAJUSTICIA
REP DE COLOMBIA

AFFIDAVIT

Assignment

I, Roberta J Bertone, do hereby swear by virtue of my signature below, that the attached document is a true copy of the original International Convention Assignment (filed with the United States Patent and Trademark Office on August 2, 1999) on which I was the Notary Public and which evidences the assignment of all international patent rights in the invention titled ELECTROLYTIC SYNTHESIS OF PERACETIC ACID to STERIS INC

Dated September 1st, 2000

Roberta J Bertone
Notary Public

Notary Public State of Ohio
Recorded in Lake County
My Comm Expires 03-11-01

DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
RES 02755/58 Y 01144/97 MINJUSTICIA
REP DE COLOMBIA


CONSULADO GENERAL

143 #60
#52

Attorney Docket No MED 2 1151

ASSIGNMENT

Whereas we, Tom L Mark of 7851 Birchwood Drive, City of Chesterland, County of Geauga, and State of Ohio; Paul M Malchesky of 239 Barrington Ridge, City of Painesville Township, County of Lake, and State of Ohio, and Chung-Chiun Liu of 2919 E Overlook Road, City of Cleveland Heights, County of Cuyahoga, and State of Ohio, having invented certain new and useful improvements in

ELECTROLYTIC SYNTHESIS OF PERACETIC ACID

for which we are about to make application for Letters Patent of the United States, do hereby, in consideration of One Dollar ($1 00) and other good and valuable consideration, receipt of which is hereby acknowledged, sell, assign, and transfer unto STERIS INC a Delaware corporation, having a principal place of business at

43425 Business Park Drive
Temecula, CA 92590

the full and exclusive right, title, and interest in and to the said invention in the United States and its territorial possessions, and in all foreign countries with all rights under the International Convention including the right to claim priority, and the entire and exclusive right, title, and interest in and to any and all Letters Patent which may be granted therefor in the United States and its territorial possessions and in any and all foreign countries, and in and to any and all divisions, reissues, continuations, and extensions thereof

We therefore authorize and request the Patent Office officials in the United States and in any and all foreign countries to issue any and all Letters Patent when granted, solely to the said

STERIS INC.

for its sole use, and that of its successors, and assigns

DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
RES 02755/98 Y 01144/97 - MINJUSTICIA
REP DE COLOMBIA

Signed at Mentor, Ohio, on this 1st day of August, 2000

Tom L Mark

STATE OF Ohio)
) SS
COUNTY OF Lake)

Personally appeared before me this day, the above named Tom L Mark to me personally known, who acknowledged that he signed the foregoing instrument for the uses and purposes therein mentioned, and that the same is his free act and deed

In testimony whereof, I have hereunto set my hand and affixed my seal, this 1st day of August, 2000

Roberta J Bertone
Notary Public

ROBERTA J. BERTONE
Notary Public, State of Ohio
Recorded in Lake County
My Comm. Expires 05-11-01

(Seal)

DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
RES. 02755/68 Y 01144/97 - MINJUSTICIA
REP DE COLOMBIA

62
150 184

Signed at Mentor, Ohio, on this 1st day of August, 2000

Paul S Malchesky

STATE OF Ohio)
) SS
COUNTY OF Lake)

Personally appeared before me this day, the above named Paul S Malchesky to me personally known, who acknowledged that he signed the foregoing instrument for the uses and purposes therein mentioned, and that the same is his free act and deed

In testimony whereof, I have hereunto set my hand and affixed my seal, this 1st day of August, 2000

Roberta J. Bertone
Notary Public

(Seal)

ROBERTA J. BERTONE
Notary Public, State of Ohio
Recorded in Lake County
My Comm Expires 03-11-01

151 65 155

Signed at Mentor, Ohio on this 1st day of August, 2000

Chung-Chiun Liu

STATE OF Ohio)
) ss
COUNTY OF Lake)

Personally appeared before me this day the above named Chung-Chiun Liu to me personally known, who acknowledged that he signed the foregoing instrument for the uses and purposes therein mentioned, and that the same is his free act and deed

In testimony whereof, I have hereunto set my hand and affixed my seal, this 1st day of August, 2000

Roberta J. Bertone
Notary Public

ROBERTA J. BERTONE
Notary Public, State of Ohio
Recorded in Lake County
My Comm. Expires 03-11-01

(Seal)

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DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
DES. 02755/68 Y 01144/97 - [illegible]
REP DE COLOMBIA

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----------TRADUCCION OFICIAL No 2K-1004----------

de un documento escrito en INGLES, el cual para su identificación lleva el sello de la Traductora e Intérprete Oficial, Res 2755/68 y 01144/97 de Minjusticia, al igual que la presente ----------

DECLARACION

Cesión

Yo, Roberta J Bertone, por medio del presente juro en virtud de mi firma más adelante que el documento adjunto es fiel copia de la Cesión de Convencion Internacional (presentada en la Oficina de Patentes y Marcas de Estados Unidos el 2 de Agosto de 1999) en la cual actué como Notario Público y la cual de prueba de la cesión de todos los derechos de patentes internacionales sobre la invencion titulada SINTESIS ELECTROLITICA DE ÁCIDO PERACETICO a favor de STERIS, INC

(fdo) Roberta J Bertone

Roberta J Bertone

Notario Publico

Fecha Septiembre 1, 2000

Notario Publico Estado de Ohio

Registrado en el Condado de Lake

Mi com Vence 03-11-01

CESION

Considerando que nosotros, Tom L Merk con domicilio en 7851 Birchwood Drive, Ciudad de Chesterland, Condado de Geauga y Estado de

DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
RES 02755/68 Y 11[illegible] MINJUSTICIA
B.P. DE COLOMBIA

Ohio, Paul S Malchesky con domicilio en 239 Barrington Ridge, Ciudad de Painesville Township, Condado de Lake y Estado de Ohio, y Chung-Chiun Liu con domicilio en 2919 E Overlook Road, Ciudad de Cleveland Heightsm Condado de Cuyahoga y Estado de Ohio, hemos inventado ciertas mejoras nuevas y utiles en relación con

SINSTESIS ELECTROLITICA DE ACIDO PERACETICO

Para los cuales estamos proximos a solicitar Patente en los Estados Unidos, por medio del presente, con compensación de suma de Un dólar ($1 oo) y otras consideraciones positivas y de valor, cuyo recibo se reconoce por medio del presente, vendemos, cedemos y traspasamos a favor de STERIX INC una sociedad de Delaware, con domicilio principal de negocios en

4325 Business Park Drive

Temecula, CA 92590

el derecho, título e interés, pleno y exclusivo en y sobre dicha invención en los Estados Unidos y sus posesiones territoriales y en todos los paises extranjeros con todos los derechos bajo la Convención Internacional incluyendo el derecho a reivindicar prioridad y el derecho, titulo e interés pleno y exclusivo en y sobre todas las Patentes que puedan otorgarse para la misma en los Estados Unidos y sus posesiones territoriales y en todos y cada uno de los paises extranjeros y en y sobre todas las divisiones, re-expediciones, continuaciones y extensiones de la misma

Por lo tanto, autorizamos y solicitamos a los funcionarios de la Oficina de Patentes en los Estados Unidos y en cualquiera y todos los paises extranjeros,

DORA DUQUE DE BERNAL

154 158

que expidan toda y cualquier patente cuando sea concedida, exclusivamente a dicha STERIS INC

para su uso exclusivo y el de sus sucesores y cesionarios

Firmado en Mentor, Ohio, hoy dia 1° de Agosto, 2000

(fdo) ilegible

Tom L Merk

ESTADO DE OHIO

CONDADO DE LAKE

Comparecio personalmente ante mi hoy el antes nombrado Tom L Mark a quien conozco personalmente, quien reconoció que él firmó el anterior instrumento para los usos y fines allí mencionados y que el mismo es su acto y escrito voluntario

En testimonio de lo cual, he impuesto aqui mi firma y he fijado mi sello hoy dia 1° de Agosto de 2000

(fdo) Roberta J Bertone

Notario Publico

Roberta J Bertone

Notario Publico, Estado de Ohio

Registrado en el Condado de Lake

Mi comisión vence 05-11-01

Firmado en Mentor, Ohio, hoy dia 1° de Agosto, 2000

(fdo) ilegible

DORA DUQUE DE PERSIVAL
TRADUTORA E INTERPRETE OFICIAL
RES 02755/08 F 011 [illegible] DE JUSTICIA
REP DE [illegible]

6PT
155 159

Paul S Malchesky

ESTADO DE OHIO

CONDADO DE LAKE

Compareció personalmente ante mi hoy el antes nombrado Paul S Malchesky a quien conozco personalmente, quien reconoció que él firmó el anterior instrumento para los usos y fines allí mencionados y que el mismo es su acto y escrito voluntario

En testimonio de lo cual, he impuesto aquí mi firma y he fijado mi sello hoy dia 1° de Agosto de 2000

(fdo) Roberta J Bertone

Notario Publico

Roberta J Bertone

Firmado en Mentor, Ohio, hoy día 1° de Agosto, 2000

(fdo) ilegible

Chung-Chiun Liu

ESTADO DE OHIO

CONDADO DE LAKE

Compareció personalmente ante mi hoy el antes nombrado Chung-Chiun Liu a quien conozco personalmente, quien reconoció que él firmó el anterior instrumento para los usos y fines allí mencionados y que el mismo es su acto y escrito voluntario

DORA DUQUE DE BERNAL
NOTARIO PUBLICO

En testimonio de lo cual, he impuesto aqui mi firma y he fijado mi sello hoy dia 1º de Agosto de 2000

(fdo) Roberta J Bertone

Notario Publico

Roberta J Bertone

ESTADO DE OHIO

CONDADO DE LAKE

Yo, Lynne L Mazeika, Escribano de la Corte Ordinaria, una Corte de Registro del Condado de Lakes antes mencionado, certifico por medio del presente que ROBERTA J BERTON, ante quien el instrumento, juramento o declaración anexo fue tomado, era en la fecha del mismo un Notario Publico en y para dicho Condado debidamente autorizado por las leyes de Ohio para tomar el mismo, y para tomar reconocimientos, declaraciones y pruebas de escrituras o traspasos de terrenos, tenencias o heredamientos situados y que queden en dicho Estado de Ohio, y además que conozco su escritura y verdaderamente creo que su firma en el mismo es auténtica, y que el instrumento anexo está otorgado de acuerdo con las leyes del Estado de Ohio-

La comisión vence el 11 de marzo de 2001

En testimonio de lo cual, he impuesto aquí mi firma y he fijado el sello de dicha Corte en la ciudad de Painesville, Ohio hoy dia 5 de Septiembre de 2000

(fdo) Lynne L Mazeika, Escribano de las Cortes

Lynne L Mazeika, Escribano

No A 90-91491 (Sello en relieve)

DORA DUQUE DE BERNAL
NOTARIO PUBLICO
RES. 027[illegible]
REP. DE COLOMBIA

ESTADOS UNIDOS DE AMERICA

ESTADO DE OHIO

OFICINA DEL SECRETARIO DE ESTADO

Yo, J KENNETH BLACKWELL, Secretario de Estado, certifico por medio del presente que soy el Secretario de estado del Estado de Ohio debidamente elegido, habilitado y en ejercicio, y certifico además que LYNNE L MAZEIKA cuya firma y sello oficial están impuestos en la certificación adjunta al presente, era en la fecha de la misma el ESCRIBANO DE LA CORTE ORDINARIA DEL CONDADO DE LAKE, OHIO, debidamente elegido, comisionado y habilitado, y que ella es la funcionaria competente para hacer dicha certificación, la cual está en debida forma, y que sus actos como tal tienen derecho a plena fe y crédito

EN TESTIMONIO DE LO CUAL, he suscrito aquí mi nombre y he fijado el sello oficial del Secretario de estado de Ohio, en Columbus, Ohio, el dia 11 de Septiembre de 2000

(fdo) J Kenneth Blackwell

J KENNETH BLACKWELL

Secretario de Estado

(Sello en oblea ocre)

00046477-3

ESTADOS UNIDOS DE AMERICA

DEPARTAMENTO DE ESTADO

A TODOS A QUIENES LOS PRESENTES LLEGAREN, SALUDO

Certifico que l documento anexo al presente esta bajo el sello del Secretario de Estado del Estado de Ohio, y que tal sello merece plena fe y crédito *

En testimonio de lo cual, yo, Strobe Talbott, Secretario de Estado Encargado, he hecho fijar al presente el sello del Departamento de Estado y he hecho suscribir mi nombre por el Funcionario de Autenticación de dicho Departamento, en la ciudad de Washington, en el Distrito de Columbia, hoy dia dieciseis de Septiembre de 2000

(fdo) Strobe Talbott

Secretario de Estado Encargado

Por (fdo) ilegible

Funcionario de Autenticación Asistente

Departamento de Estado

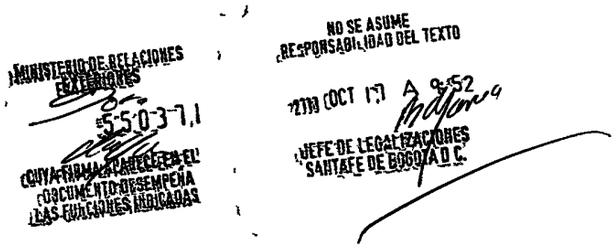
- El Depto no asume ninguna responsabilidad por el contenido de los documentos anexos
- Este certificado no es válido si se remueve o altera de otra forma

---

EN ESPAÑOL, la legalización de la firma de J Kenneth Blackwell por el Consulado de Colombia en Chicago, No 3147 de fecha Septiembre 22, 2000 y autenticación de la firma consular de José Fernando Gomez Mora por el Ministerio de Relaciones Exteriores de Colombia bajo el numero 354034 de Octubre 11, 2000

ES TRADUCCION FIEL Y COMPLETA, a la cual me remito para su confrontación

DORA DUQUE DE BERNAL
TRADUCTORA E INTERPRETE OFICIAL
RES. 02755/68 Y 01144/97 MINJUSTICIA
REP DE COLOMBIA


MINISTERIO DE RELACIONES EXTERIORES
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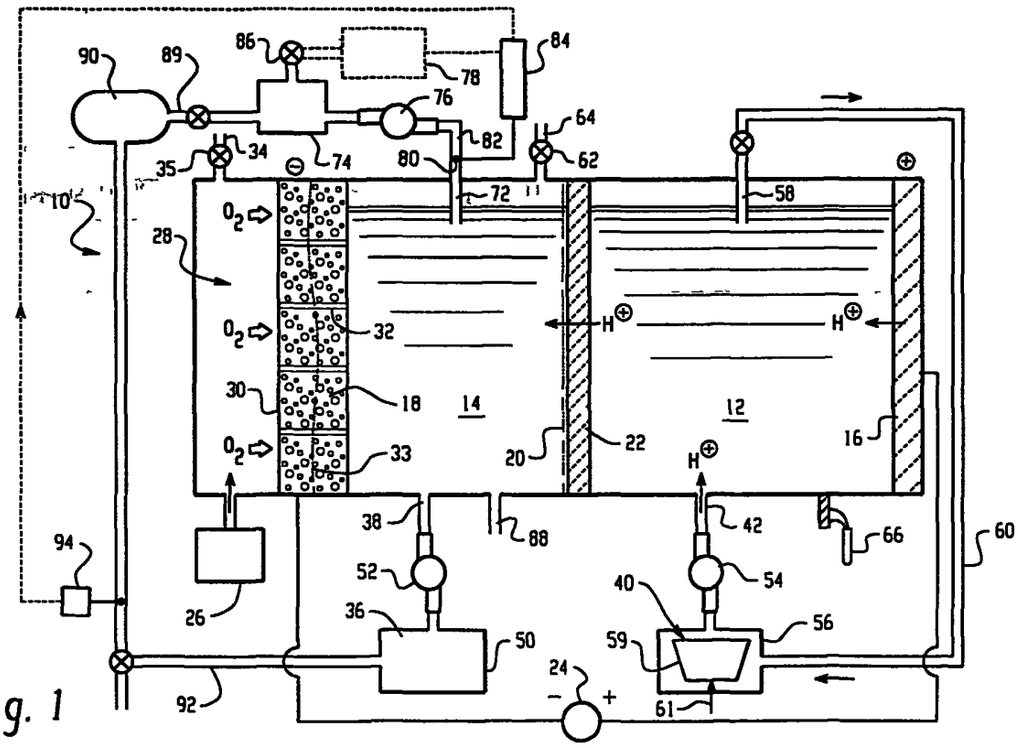
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86
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80
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72
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10
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O_2
O_2
O_2
32
H
H
30
18
14
12
16
33
20
22
H
38
88
42
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60
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54
36
50
56
59
26
24
92
61

Fig. 1

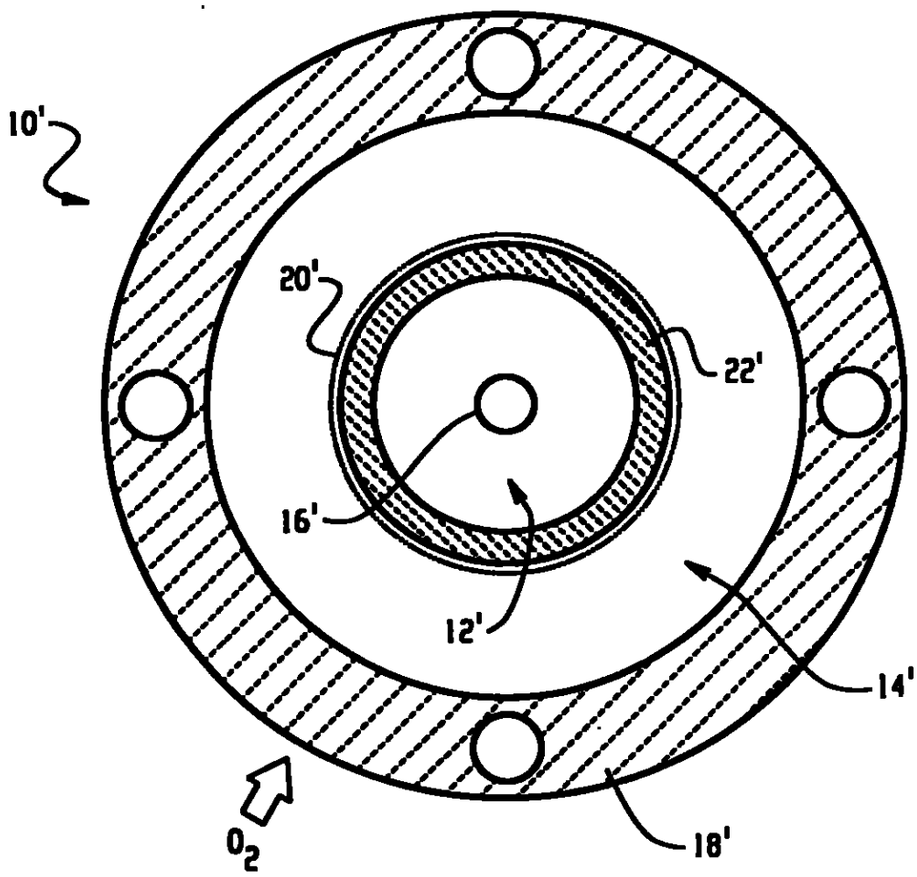
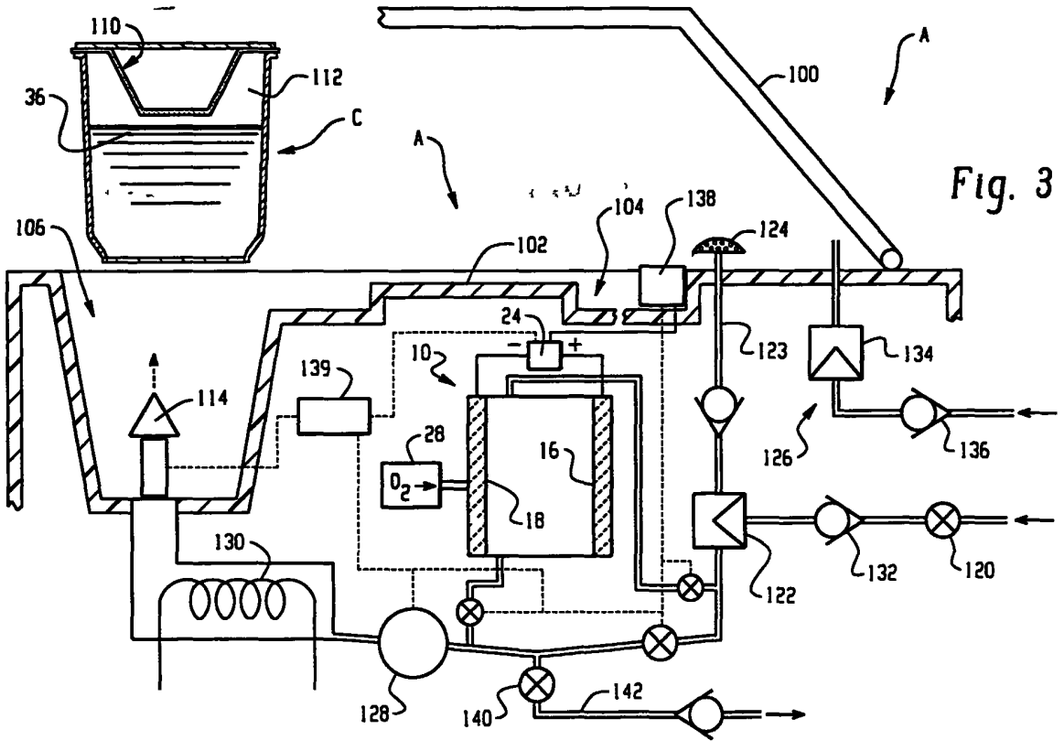


Fig. 2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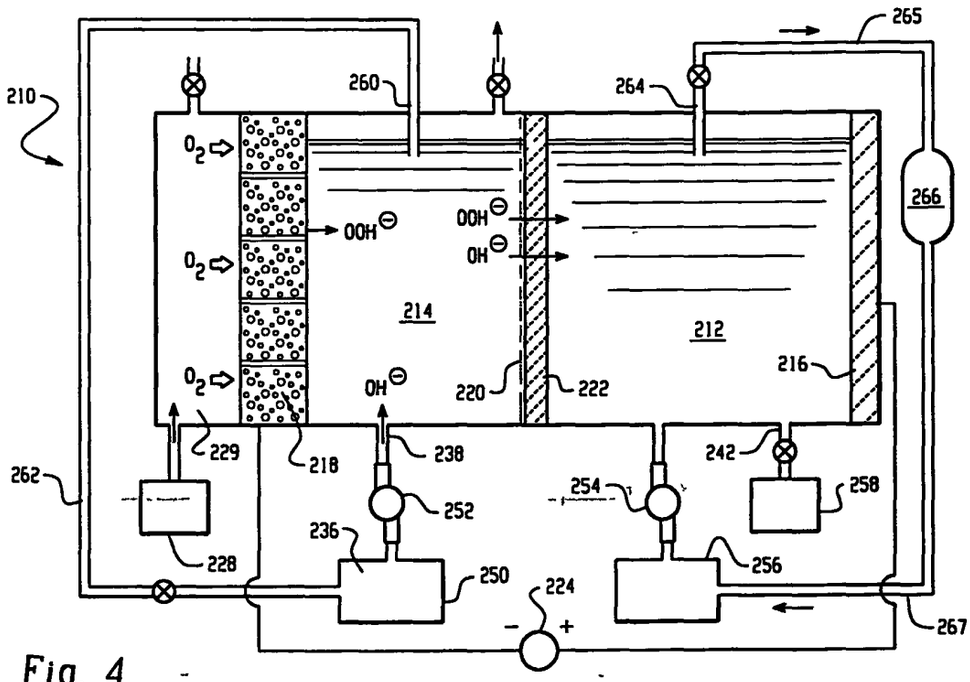
112
36
C
A
100
A
106
102
104
138
124
24
123
134
139
10
114
28
16
126
136
O_2
18
130
132
120
122
128
140
142

Fig. 3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265
266
O_2
O_2
O_2
OOH
OOH
OH
OH
214
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216
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222
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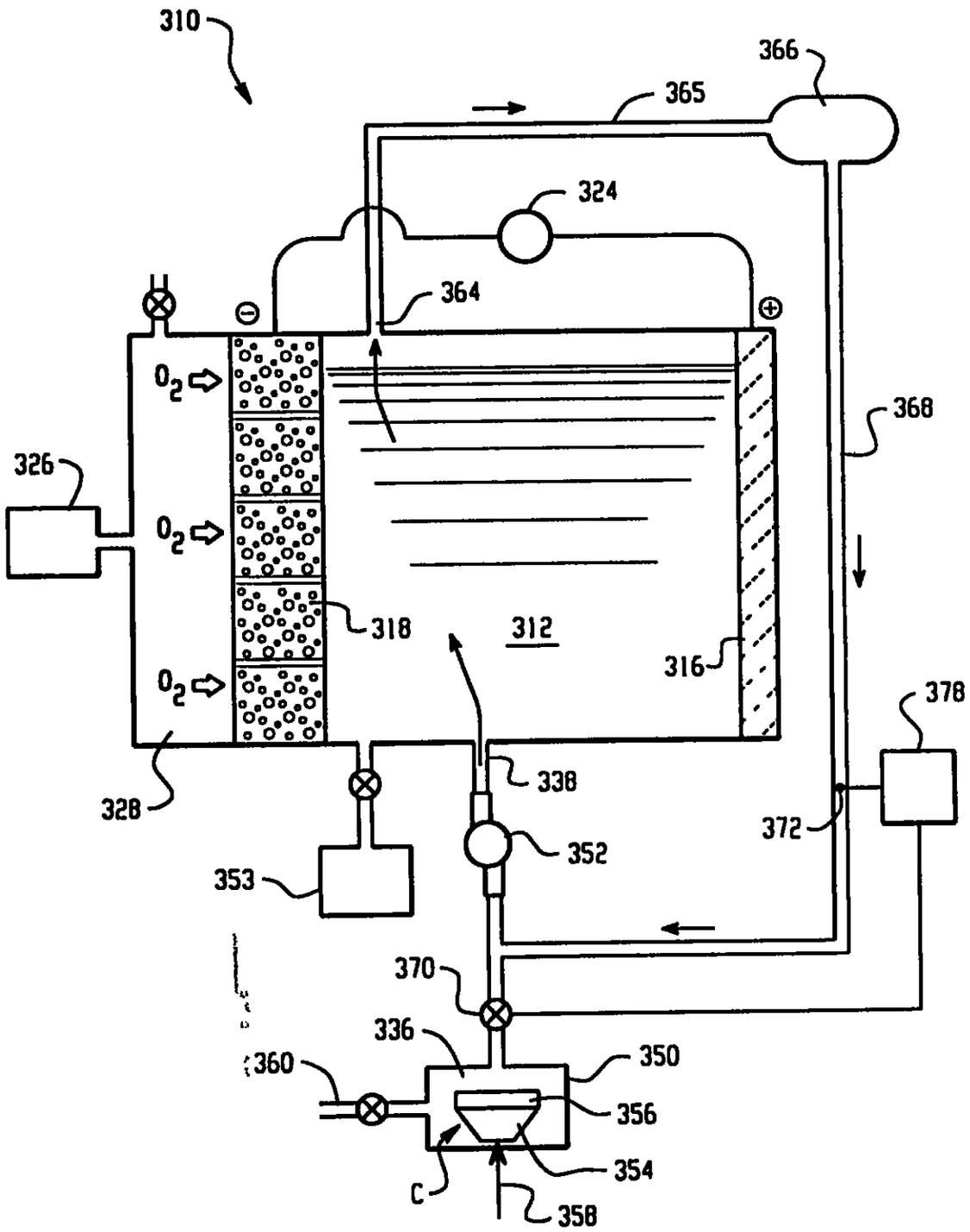
218
238
242
262
254
258
252
236
228
250
256
224
267

Fig. 4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366
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O_2
O_2
O_2
326
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353
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336
350
360
356
354
C
358

Fig. 5

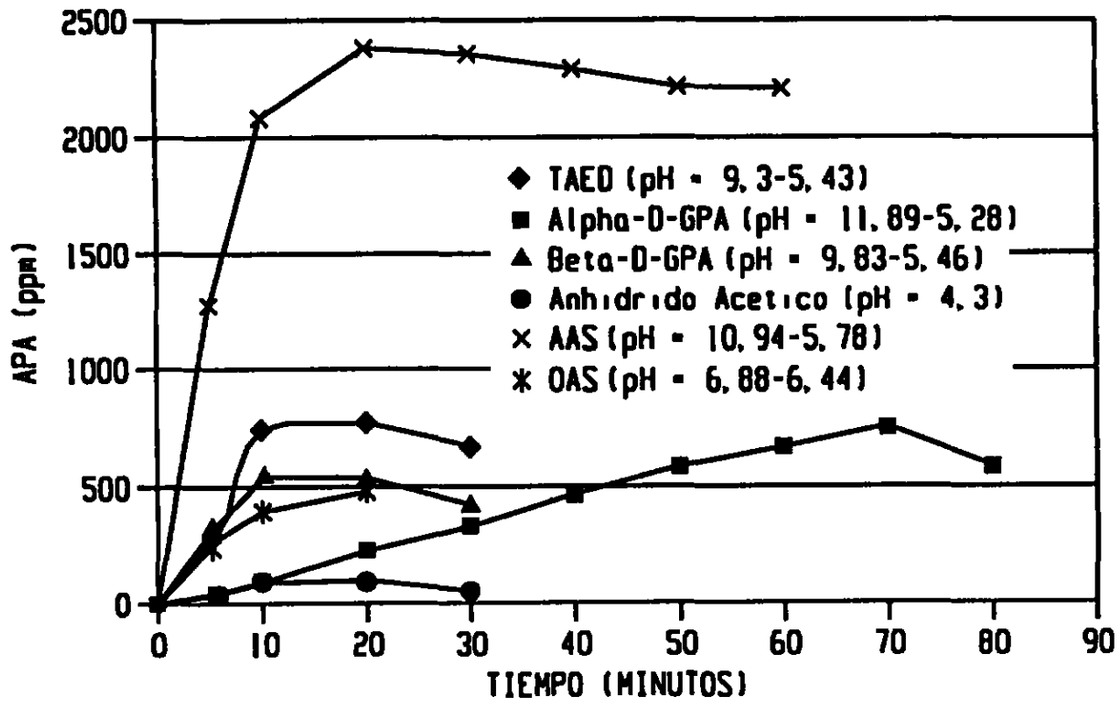
2500
2000
1500
1000
500
0
APA (ppm)
0
10
20
30
40
50
60
70
80
90
TIEMPO (MINUTOS)
◆ TAED (pH = 9.3-5.43)
■ Alpha-D-GPA (pH = 11.89-5.28)
▲ Beta-D-GPA (pH = 9.83-5.46)
● Anhidrido Acetico (pH = 4.3)
× AAS (pH = 10.94-5.78)
* OAS (pH = 6.88-6.44)

Fig. 6

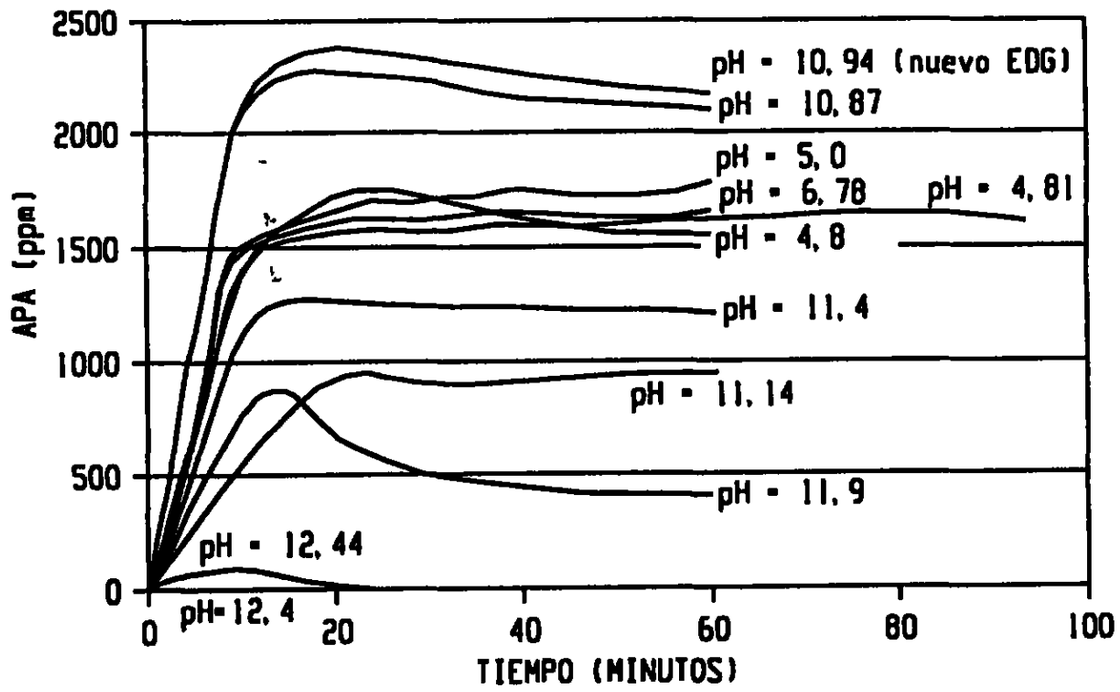
2500
2000
1500
1000
500
0
APA (ppm)
0
20
40
60
80
100
TIEMPO (MINUTOS)
pH = 10.94 (nuevo EDG)
pH = 10.87
pH = 5.0
pH = 6.78
pH = 4.81
pH = 4.8
pH = 11.4
pH = 11.14
pH = 11.9
pH = 12.44
pH=12.4

Fig. 7