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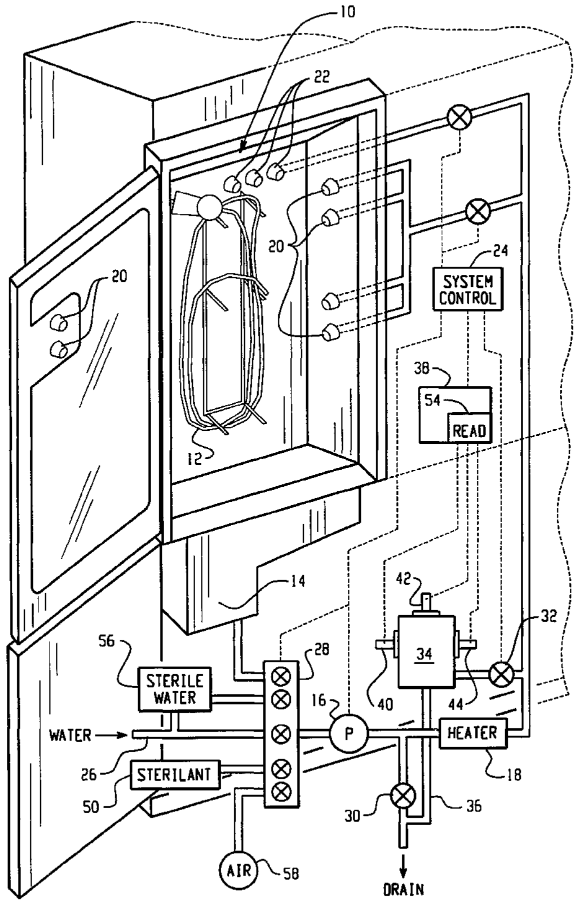
(54) Title: DURABLE CARBON ELECTRODE



(57) Abstract: A disinfection/sterilization system includes a chamber (10) through which an antimicrobial solution is circulated. An electrochemical analysis system taps off samples of the circulated antimicrobial solution for analysis. The analysis system includes a chamber (34) that receives the sample. A working electrode (40), a reference electrode (42), and a counter electrode (44) are in electrical contact with the sample. The working electrode includes a brittle material, such as a carbon rod (60), which is surrounded by a compressible polymeric sleeve (64) and a metal sleeve (66). One end of the metal sleeve is swaged (68) to form a fluid tight compression seal with the insulating sleeve and the carbon rod. An electrically conductive thermal extension joint between the carbon rod and an electrically conductive rod (70) includes a bore (72) in the conductive rod in which the carbon rod is slidably received and a compressed spring (74) in the bore. Preferably, an opposite end of the metal sleeve is swaged (78) to form a fluid tight seal with an insulating sleeve (76) and the electrically conductive rod (70).

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DURABLE CARBON ELECTRODE

Background of the Invention

The present invention relates to the electrode arts. It finds particular application in conjunction with carbon electrodes for use in peracetic acid detectors and will be described with particular reference thereto. It is to be appreciated, however, that the invention will also find application in conjunction with carbon-based electrodes for use in other electrochemical applications.

Heretofore, in liquid-based sterilization systems, a premeasured dose of a strong oxidant antimicrobial agent, such as peracetic acid or reagents which react to form peracetic acid, hydrogen peroxide, chlorides, hydrochlorite, and other strong oxidants, was added to each disinfection or sterilization cycle. Strong oxidants react with the microbes and other contaminants that may be in the system. In medical applications, a blood residue is a strong oxygen scavenger. To determine whether an appropriate concentration of the antimicrobial agent was maintained in solutions for an appropriate time, biological and chemical indicators were typically placed with the items to be disinfected or sterilized in the processor. After the cycle, these indicators were incubated and examined, colorimetrically or otherwise, to provide a pass/fail indication of the success of the disinfection or sterilization cycle. While it would be advantageous to provide a real time parametric monitoring of the antimicrobial agent concentration, cost effective, accurate, reliable, and long-lived monitors have proved elusive.

One particular promising electrochemical sensor includes three electrodes, a working electrode, a counter electrode, and a reference electrode which are emersed in this liquid to be monitored. Electrically conducting counter electrodes and silver/silver chloride reference electrodes which can be immersed in the strong oxidants for extended periods are available at a relatively reasonable cost. However, the ideal carbon working electrode tends to be fragile, expensive, and has a relatively short working life.

Prior art carbon electrodes typically include a short rod of a glassy carbon, a brittle fragile material. A glassy carbon rod is commonly interfaced electrically with a metallic electrode rod using a graphite or other conductive grease. The peripheral surface of the carbon rod, the electrode rod, and the grease interface is surrounded by a plastic sheath, e.g., a heat shrunk or pressed in plastic sheath. The face of the glassy carbon electrode is polished, typically hand polished. Unfortunately, the plastic sleeve does not adhere tightly to the carbon electrode under all normal operating conditions. For example, when mounted in a sealed fitting, the plastic sleeve tends to separate from the carbon allowing the monitored solution to pass in between. This provides both an escape path for the conductive graphite grease and an entrance path for the analyzed solution to degrade the grease and the metal electrode. Further, fluid access along the side of the electrode changes the effective surface area of the electrode, hence its output characteristics. When the plastic ceases to hold the carbon electrode firmly against the grease and the metal electrode, the electrical signal transmission characteristics are altered and eventually totally interrupted.

Glassy carbon electrodes are used in other electrochemical analysis and other processes. In some, the carbon itself is used as the electrode. In others, the carbon is used as a carrier for a different electrode material, such as mercury. In these other applications, particularly applications in which the carbon electrode is handled manually, the delicacy of the electrodes, their cost, and short working life are again significant drawbacks.

In accordance with the present invention, a new and improved carbon electrode, parametric sensing system, and disinfectant/sterilizer with improved parametric monitoring are provided.

Summary of the Invention

In accordance with one aspect to the present invention, an electrode including an electrically conductive rod is provided. The electrode is characterized by a compressible insulating sleeve surrounding the rod. The compressible insulating sleeve compresses at a pressure lower than a compressive pressure at which the electrically conductive rod fractures. A ductile sleeve surrounds the insulating sleeve, the ductile sleeve being compressed, thereby compressing the insulating sleeve and providing a fluid-tight seal between the ductile sleeve, the insulating sleeve, and the electrically conductive rod.

In accordance with another aspect of the present invention, an electrochemical analyzer is provided. The analyzer includes an electrochemical cell which defines a fluid receiving reservoir. A plurality of measuring electrodes are disposed in the reservoir to be in contact with solutions received in the reservoir. An analysis control system is connected with the electrodes for applying at least one of voltages and currents thereto and reading at least one of voltages and currents therefrom which are indicative of a concentration of a target composition. At least one of the electrodes is an electrode as previously described.

In accordance with another aspect of the present invention, a disinfectant/sterilant system is provided. The system includes a chamber, which receives an item to be disinfected or sterilized. A pump pumps an antimicrobial solution into the chamber. A reservoir is fluidly connected with the chamber. A plurality of measuring electrodes are disposed in the reservoir to be in electrical contact with solutions received in the reservoir. At least one of the electrodes is an electrode as previously described. An analysis control system is connected with the electrodes for applying voltages thereto and reading currents therefrom which are indicative of the concentration of an oxidizing species in the antimicrobial solution.

In accordance with another aspect of the present invention, an electrochemical method is provided. The method includes surrounding a carbon rod with an insulating sleeve, surrounding the insulating sleeve with a ductile metal sleeve, and compressing the ductile metal sleeve forming a fluid-tight compression seal with the insulating sleeve and the carbon rod. The method further includes making an electrical connection with the carbon rod which compensates for thermal expansion of metallic sleeve and connecting the electrical connection with an electrochemical analysis system. The method further includes contacting at least a portion of the carbon rod with a fluid to be analyzed.

One advantage of the present invention is that it facilitates parametric monitoring of peroxyacetic acid concentration.

Another advantage of the present invention resides in a more durable and longer lived carbon electrode.

Another advantage of the present invention resides in its cost efficiency.

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.

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 diagrammatic illustration of a liquid disinfection/sterilization system utilizing an electrochemical sensor with a carbon electrode in accordance with the present invention;

Figure 2 is an enlarged, cross-sectional view of an electrochemical sensor in accordance with the present invention; and

Figure 3 is a cross-sectional view of a carbon electrode in accordance with the present invention.

Detailed Description of the Preferred Embodiments

With reference to Figure 1, a liquid disinfection/sterilization system includes chamber **10** in which an item, such as an endoscope **12** is mounted for sterilization or disinfection. The chamber **10** defines a sump **14** at the bottom where fluid which has been sprayed on the surface of the endoscope or passed through its lumens collects. A pump **16** pumps the liquids from the sump through a heater **18** to nozzles **20** and inlet ports **22** of the chamber. The nozzles **20** spray fluid over the surface of the endoscope and the fluid outlet ports **22** are connected with interior lumens of the endoscope. A system controller **24** controls system components to affect rinsing, cleaning, and sterilization or disinfection operations. Preferably, the system controller controls a water supply **26** supply water through a manifold valve **28** to the pump to rinse debris from the endoscope. After the initial rinse cycle, a drain valve **30** drains the rinse water from the system. During a wash cycle, the system is again filled with water. Detergent is released from an ampule received in the sump **14** into the water and is again circulated by the pump. At the end of the cleaning cycle, the drain valve again drains the system. Next, the manifold connects the water system to the pump again for another rinse cycle.

After the rinse, water is drained and the system refilled. The ampule releases treatment agents including anticorrosives, buffers, and surfactants into the water. During the circulation of the treatment agents, a sample of the treatment solution is selectively drawn by a valve **32** through an electrochemical analysis chamber **34** to fill an internal reservoir. Preferably, the reservoir is overfilled such that a small amount of fluid goes down a drain line **36** to assure accurate filling. When these agents are received in the analysis chamber, an analysis control system **38** cycles electrical potential to a working electrode **40**, a silver/silver chloride or other suitable reference electrode **42**, and a non-reactive counter electrode **44**. Alternatively, the counter electrode may be omitted. In this case, the reference electrode serves also as the counter electrode, with some loss of stability often resulting.

Alternating negative pulses of amplitude of about -1.5 to -2.5 volts and positive pulses of about +1.5 to +3.5 volts vs Ag/AgCl are applied to the working electrode **40** for periods of 1-10 seconds to condition the face of the electrode. In one embodiment, the negative pulses are about -2.0 volts for a duration of about 2 seconds and the positive pulses are about +2.5 volts for a duration of about 4 seconds, although other pulse sequences may be equally effective, depending on the electrode materials used and the cleanliness of the electrode surface. About 10-50 pulses may be used, e.g., 15 pulses of 2-4 seconds duration.

After a preselected circulation time, the control circuit **38** causes the release of an antimicrobial agent either in the sump **14** or from a reservoir **50**. The antimicrobial agent is added to the solution and circulated over and through the endoscope for a period of time controlled by the analysis controller **38**. Once the antimicrobial agent, peroxyacetic acid in the preferred embodiment has been added to the solution, the control system **24** repeatedly causes the valve **32** to open, flushing and refilling the electrochemical analysis chamber reservoir with the antimicrobial solution. Other peracids, or mixtures of peracids are also useful antimicrobial agents. Once the electrochemical analysis reservoir has been filled, it is brought to a preselected temperature by a heater **52**. Once at temperature, the electrodes are cycled through an about +1.5 to +3.5 volt and an about -1.5 to -2.5 volt vs Ag/AgCl preparation cycle prior to commencing an electrochemical measurement.

In a preferred measurement procedure, a polarizing voltage is maintained between the reference and working electrodes of a 3-electrode system. A feedback loop maintains the specified voltage between the reference and working electrodes and applies a voltage to the counter electrode, such that current flows between the working electrode and the counter electrode and little or no current flows from the reference electrode. The potential of the counter electrode is typically of greater magnitude than the potential at the reference electrode. Operational amplifiers connected between the reference and counter electrode permit current flow through the sample solution only between the working and counter electrodes. This allows precise control of the applied potential while preventing current from flowing through the reference electrode. The current flowing between the working and counter electrodes is detected by a current monitor or reader **54** in the analysis control system **38** which converts the current into an indication of peracetic acid concentration (or other antimicrobial being sensed). In the preferred embodiment, the reader **54** is preprogrammed with a look-up table which correlates current to concentration. At a given temperature, fixed by the heater **52**, the measured current is dependent on the concentration of peroxyacetic acid concentration as well as a concentration of other oxidizing species, e.g., hydrogen peroxide. The contributions of these components are dependent on the selected read voltage. In the preferred embodiments, for specific detection of peracetic acid in the presence of hydrogen peroxide, the read voltage is selected in a range of -0.5 to -1.6 volts, relative to the Ag/AgCl reference electrode, the exact voltage being dependent on the choice of electrode material. At the preferred read potential, the measurement is relatively insensitive to hydrogen peroxide concentration while being sensitive to peroxyacetic acid concentration. Near the preferred read voltage, the contribution from hydrogen peroxide concentration is comparable with the contribution from background noise. Hence, the concentration determined by the reader **54** is an accurate measurement of peroxyacetic acid concentration.

After the antimicrobial solution is drained, sterile water from a sterile water source **56** rinses any antimicrobial solution, buffer, or anticorrosive residue from the item **14**.

After one or more sterile rinses, a sterile air source **58** blows excess rinse water out of the lumens. Optionally, the sterile air can blow excess liquid off the surface of the endoscope.

Looking now to details of the preferred working electrode and with reference to Figure 3, the working electrode **40** includes a rod **60**, formed from an electrically conductive material, such as carbon, and having an exposed portion, such as a face **62** of preselected area. Carbon is an effective electroactive for peracetic acid and is highly selective for peracetic acid in the presence of hydrogen peroxide. Carbon electrodes are also relatively resistant to peracetic acid, giving them a longer useful life. Crystalline forms of carbon, such as graphite, provide effective working electrodes for measurements in the diffusion limited region. One preferred form of crystalline carbon is pyrolytic graphite (obtainable from Advanced Ceramics Corp), a highly crystalline form, manufactured by decomposition of hydrocarbon gas at high temperatures in a vacuum furnace. Another form of crystalline carbon is sold under the tradename Isomolded graphite by Industrial Sales, Melrose Park, Ill. Amorphous forms of carbon, such as glassy carbon, also provide effective working electrode materials for measurements in the diffusion limited region. Other forms of amorphous carbon may also be used, such as mixtures of carbon powder in oil, carbon black, and the like. The present invention is particularly suited to use with brittle conductive materials, such as glassy carbon, which is very brittle and relatively easily crushed. For ease of reference, the conductive rod **60** will be described as a carbon rod, although the use of other conductive materials is also contemplated.

A preferred read voltage for detection of peracetic acid with glassy and iso carbon electrodes is from -0.9 volts to -1.1 volts. For pyrolytic graphite, a preferred read voltage is from -1.1 to -1.3 volts.

The carbon rod **60** is surrounded by a close-fitting polymeric sleeve **64** which is compressible at a compressive force lower than that which fractures the carbon rod and which does not flow significantly, e.g., Teflon™. A second sleeve **66**, which is inert to the material being analyzed, surrounds the polymeric sleeve. The second sleeve is preferably sufficiently ductile to allow compression of the sleeve into a desired shape but retains sufficient rigidity during use to inhibit bending and damage to the brittle rod. Metals provide good second sleeves **66**. For the analysis of strong oxidants, stainless steel is preferred. At one end, the stainless steel sleeve **66** is compressed or swaged **68** into a tight, compressive frictional fit with the Teflon™ polymer, hence the carbon rod. The carbon rod is electrically interconnected with an electrical fitting **70** of preselected geometry, in the preferred embodiment, a rod.

Due to the brittle nature of some forms of carbon, particularly glassy carbon rods, and the differences in thermal expansion characteristics between stainless steel and typical electrode materials such as brass, a reliable interconnection between a carbon rod and the conductive rod **70** is not trivial. In the preferred embodiment, the electrically conductive rod **70** has a bore **72** which matches the exterior diameter of the carbon rod **60**. The carbon rod is slidably received in the bore providing an electrical interconnection therebetween. Optionally, this interface is lubricated with an electrically conductive grease. A metallic spring **74** is disposed in the bore and compressed between a seat at the bottom of the bore and an end surface of the carbon rod **60**. This spring pressure assures electrical interconnection between the carbon rod and the electrically conductive rod.

For complete water tightness and improved durability, another Teflon or other polymeric plug **76** is disposed between the electrical connecting rod **70** and an opposite end of the stainless steel tube **66**. This end of the tube is also compressed, e.g., swaged **78**, into a compressive watertight seal. Optionally, a mounting flange **80**, of appropriate shape and configuration for the analysis chamber or other appliance to which the electrode is to be mounted is welded, brazed, or otherwise connected with the stainless steel tube **66**.

After the electrode is assembled, the face **62** of the carbon electrode is polished and a protective overcap is slipped over the swaged end **68** of the stainless steel tube to prevent damage prior to installation.

Although the counter electrode is illustrated as a discrete electrode, it is to be appreciated that the stainless steel tube insulated from the analysis chamber **34** and can be used as the counter electrode **44**. The tube **66** can also be made of or plated with other advantageous electrode materials, such as platinum, gold, and the like.

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

1. An electrode **(40)** comprising an electrically conductive rod **(60)**, the electrode characterized by:

a compressible insulating sleeve **(64)** surrounding the rod, the compressible insulating sleeve compressing at a pressure lower than a compressive pressure at which the electrically conductive rod fractures;

a ductile sleeve **(66)** surrounding the insulating sleeve, the ductile sleeve being compressed, thereby compressing the insulating sleeve and providing a fluid-tight seal between the ductile sleeve, the insulating sleeve, and the electrically conductive rod.

2. The electrode of claim 1, further characterized by:

the electrically conductive rod **(60)** being formed from carbon.

3. The electrode of claim 2, further characterized by:

the carbon being selected from the group consisting of amorphous carbon and crystalline carbon.

4. The electrode of claim 2, further characterized by:

the carbon being selected from the group consisting of an amorphous glassy carbon and pyrolytic graphite crystalline carbon.

5. The electrode of any one of preceding claims 1-4, further characterized by:

the insulating sleeve **(64)** being formed from an inert polymer.

6. The electrode of any one of preceding claims 1-5, further characterized by:

the ductile sleeve **(66)** being formed from stainless steel.

7. The electrode of any one of preceding claims 1-6, further characterized by:

an electrically conductive element **(70)** electrically connected with the electrically conductive rod and supported by a second end of the ductile sleeve **(66)**.

8. The electrode of claim 7, further characterized by:

an electrically conductive biasing member **(74)** disposed between the electrically conductive element **(70)** and the electrically conductive rod **(60)**.

9. The electrode of either one of preceding claims 7 and 8, further characterized by:

a bore **(72)** defined in the electrically conductive element **(70)** which is sized and shaped to receive the electrically conductive rod **(60)** slidably therein.

10. The electrode of claim 9, further characterized by:

an electrically conductive spring **(74)** being disposed in the bore **(72)** in a compressed state to make mechanical and electrical contact with the electrically conductive element **(70)** and the electrically conductive rod **(60)**.

11. The electrode of any one of preceding claims 7-10, further characterized by:

a second end of the ductile sleeve **(66)** being compressed against an insulator sleeve **(76)** to support and form a fluid-tight seal with the electrically conductive element **(70)**.

12. An electrochemical analyzer comprising an electrochemical cell which defines a fluid receiving reservoir **(34)**, a plurality of measuring electrodes **(40, 42, 44)** disposed in the reservoir to be in contact with solutions received in the reservoir, and an analysis control system **(38)** connected with the electrodes for applying at least one of voltages and currents thereto and reading at least one of voltages and currents therefrom which are indicative of a concentration of a target composition, characterized by:

at least one of the electrodes being the electrode **(40)** of any one of preceding claims 1-11.

13. A disinfectant/sterilant system comprising a chamber **(10)** which receives an item to be disinfected or sterilized, a pump **(16)** for pumping an antimicrobial solution into the chamber, the system characterized by:

a reservoir **(34)** fluidly connected with the chamber;

a plurality of measuring electrodes **(40, 42, 44)** disposed in the reservoir to be in electrical contact with solutions received in the reservoir, at least one of the electrodes comprising the electrode **(40)** of any one of preceding claims 1-11; and

an analysis control system **(38)** connected with the electrodes **(40, 42, 44)** for applying voltages thereto and reading currents therefrom which are indicative of the concentration of an oxidizing species in the antimicrobial solution.

14. An electrochemical method characterized by:

surrounding a carbon rod **(60)** with an insulating sleeve **(64)**;

surrounding a portion of the insulating sleeve with a ductile metal sleeve **(66)**;

compressing a portion of the ductile metal sleeve forming a fluid-tight compression seal with the insulating sleeve and the carbon rod;

making an electrical connection with the carbon rod which compensates for thermal expansion of metallic sleeve;

connecting the electrical connection with an electrochemical analysis system **(38)**;

contacting at least a portion **(62)** of the carbon rod with a fluid to be analyzed.

15. The method of claim 14, further characterized by:

prior to making an electrochemical measurement, applying alternating conditioning voltage pulses to the carbon rod.

16. The method of claim 15, further characterized by:

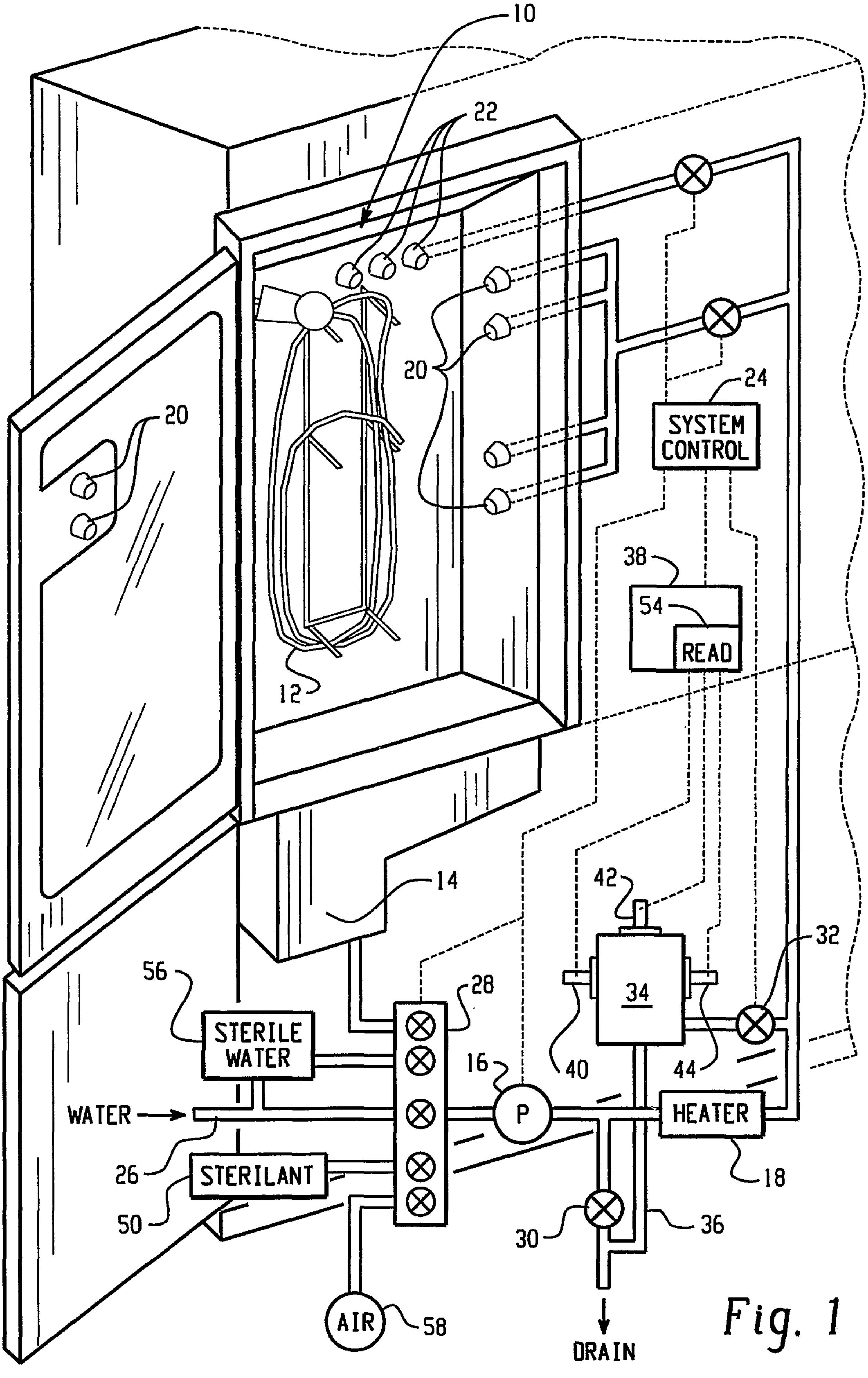
one of the alternating conditioning pulses having a voltage of about +1.5 to +2.8 volts relative to a silver/silver chloride reference electrode and a duration of 1-10 seconds and the other alternating conditioning pulse having a magnitude of -1.5 to -2.5 volts relative to a silver/silver chloride reference electrode and a duration of 1-10 seconds.

17. The method of any one of claims 14-16, further characterized by:

the alternating conditioning pulses comprising a plurality of pulses.

18. The method of any one of claims 14-17, further characterized by:

the carbon rod being formed by one of amorphous glassy carbon and pyrolytic graphite.


10
22
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24
SYSTEM CONTROL
38
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READ
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34
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STERILE WATER
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WATER
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HEATER
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STERILANT
50
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36
AIR
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DRAIN

Fig. 1

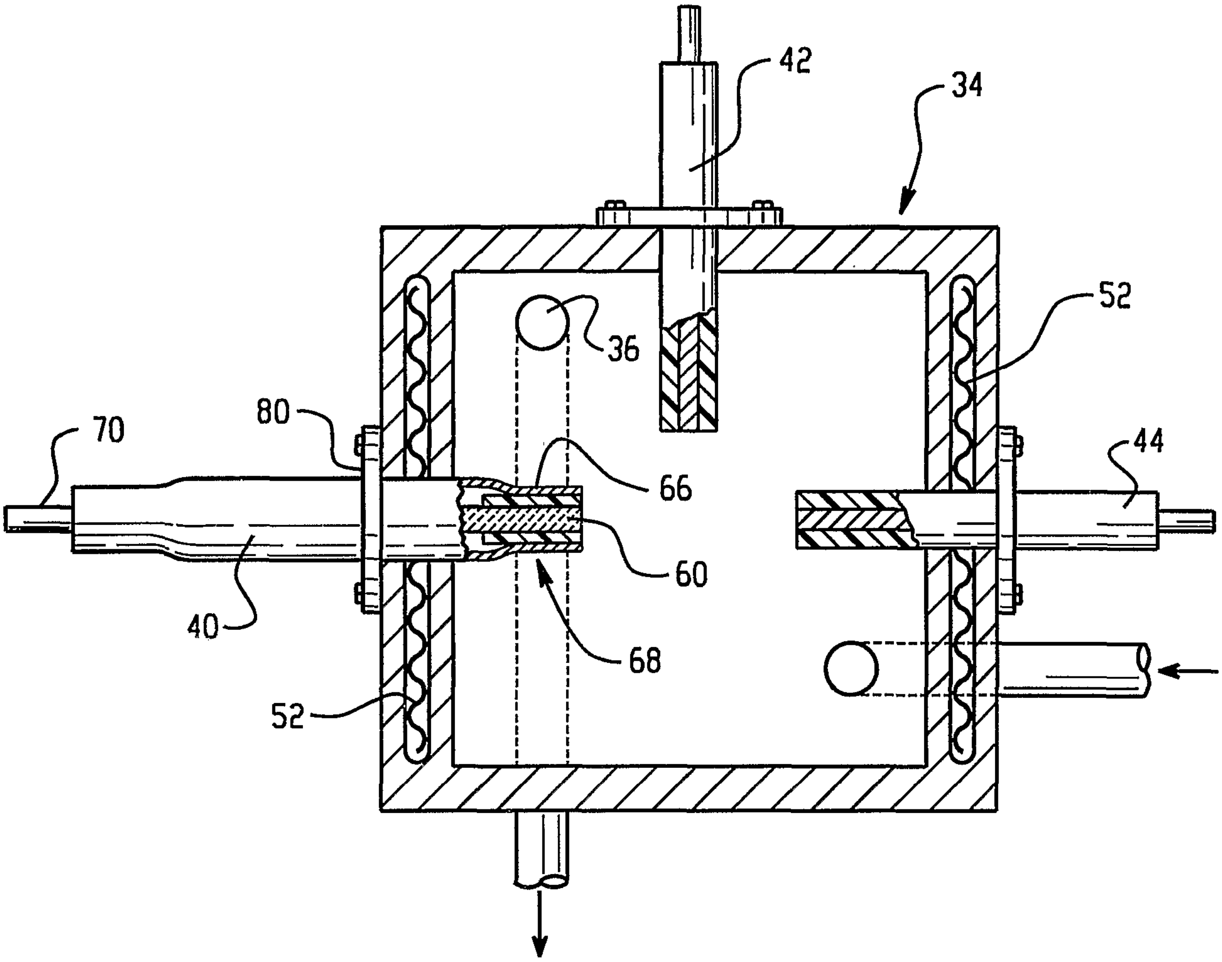

Fig. 2

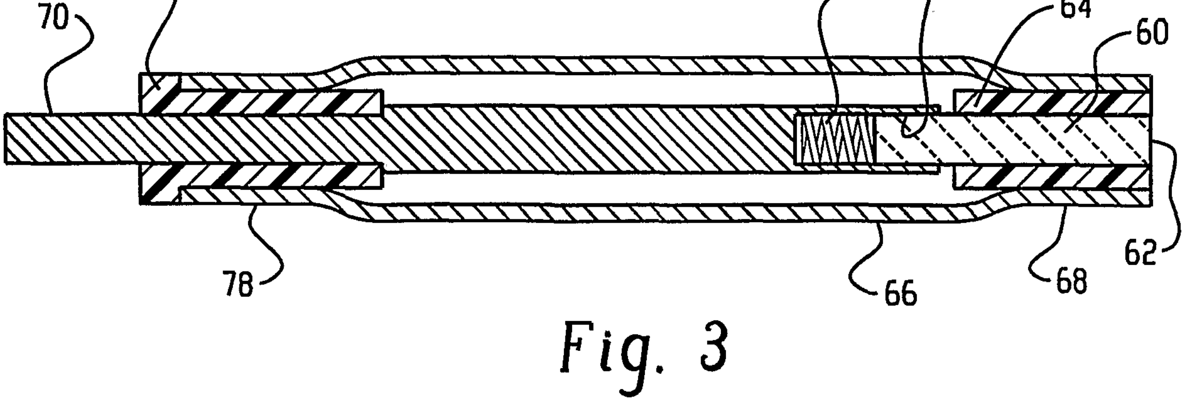

Fig. 3

INTERNATIONAL SEARCH REPORT

Inte onal Application No
PCT/US 01/03775

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H01M4/96 A61L2/00 G01N27/30

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 H01M G01N A61L

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

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

EPO-Internal, PAJ, WPI Data, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 013, no. 415 (M-870), 13 September 1989 (1989-09-13) -& JP 01 153470 A (KUREHA CHEM IND CO LTD), 15 June 1989 (1989-06-15) abstract ---	1,5,6, 12,14
A	WO 96 23215 A (TEMMERMAN EDUARD ;UNIVERSITEIT GENT VAKGROEP TEX (BE); WESTBROEK P) 1 August 1996 (1996-08-01) page 3, line 10 - line 21 page 5, line 26 - line 34 figure 1 ---	1-4,12, 14,17
A	WO 92 22808 A (MINNTECH CORP) 23 December 1992 (1992-12-23) page 2, line 32 -page 3, line 17 page 5, line 10 - line 17 -----	12,13

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search 9 July 2001	Date of mailing of the international search report 16/07/2001
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Gamez, A

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
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