

PATENT APPLICATION FOR QQC

APPARATUS FOR VOLTAMMETRIC ANALYSIS The invention relates to apparatus for voltammetric analysis.

Voltammetric analysis is a widely used electroanalytical technique. The art of this technique has been well described in many publications [1,2]. Voltammetric analysis can be carried out using various waveforms, for example, linear, pulse, square and AC or using various operation modes, for example cyclic voltammetry and stripping voltammetry [3].

Many inventors have disclosed specific devices and methods for voltammetric analysis. Girault [4] showed a device for change of electrodes. Fidler et al [5] disclosed a circuit to maintain a stable potential difference between the working and the reference electrodes. Tur Yan et al [6] used a special cell to detect trace metals using stripping voltammetry.

Saloheimo et al [7] introduced ultrasonic vibration during pre-electrolysis in stripping voltammetry. Other researchers have designed special electrodes for anodic stripping voltammetry [8-10].

Kissinger [11] predicted the future of electroanalytical instrumentation as being computerization, miniaturization and automation. Computers have been used to collect electrochemical signals [12], to acquire data and control experiments [13] and to modify waveforms [14]. Computerised voltammetric instruments are now commercially available from many instrument manufacturers.

However, computerised voltammetric instruments are generally large in size and costly. Small polarographic or voltammetric analysers are available for specific analytes, such as aldehyde [15] and lead [16]. Multi-element analysis using this type of equipment can be achieved by using multichannel selection [17] or multi-electrodes [18]. However, the number of elements that can be analysed is physically limited.

In addition, the process of voltammetric analysis often involves many decision making steps. Although computerised voltammeters can run analysis automatically it is still necessary for the supporting electrolytes, calibration standards and experimental conditions, such as initial potential, final potential, scan rate and current range, to be chosen by an operator before the analysis commences. During analysis, voltammograms with peak currents or peak areas need to be acquired, and after analysis, the data requires processing and the results reported. These decision making steps require trained personnel with knowledge and experience.

Although they can be simplified into a computer program as demonstrated by De kreuk et al [19] in an automatic stripping voltammetric method, as indicated by some researchers [20], automation of voltammetric instrument is a more complicated task than that of other electroanalytical techniques. Hence, automation is often at the level of operation rather than the decision making process.

Therefore, even computerised and highly automated systems still require skilled and experienced personnel to operate them.

In addition, conventional electrodes for voltammetry have the problem that it is difficult to ensure good electrical contact between the electrode and the electrical wire coupling the electrode to the analysing equipment. In particular, with some types of electrode, such as glassy carbon, it is not possible to solder the electrical wire to the electrode.

Accordingly, conventional methods of electrically contacting the wire to the electrode include using mercury to bond the wire to the electrode or using conductive glue. Mercury has the disadvantage that it is toxic and the conductive glue has the disadvantage that it is not easy to disconnect the conductor from the electrodes.

In accordance with a first aspect of the present invention, apparatus for voltammetric analysis comprises a processor; a first memory device coupled to the processor, the first memory device comprising a programmable read only memory and storing a number of electrochemical cell control programs; an output device coupled to the processor; an input device coupled to the processor; a second memory device coupled to the processor; an electrochemical cell controller coupled to the processor, the cell controller being adapted to be coupled to an electrochemical cell; the processing device, in response to an analyte input signal entered via the input device, retrieving a corresponding electrochemical cell control program, and instructing the cell controller to apply a scanning voltage to an

electrochemical cell coupled to the cell controller in use, in accordance with the retrieved control program, and processing output signals received by the cell controller from the electrochemical cell, in response to the applied scanning voltage, to obtain an indication of the concentration of an analyte, corresponding to the analyte input signal, in the electrochemical cell, and outputting the indication to the output device.

Preferably, the programmable read only memory is an electrically erasable programmable read only memory (EEPROM).

Preferably, the output device comprises a display device, such as a liquid crystal display (LCD). However, alternatively or in addition, the output device may comprise an interface device which permits the processor to communicate with another processing device, such as a computer, or with a storage device, such as a magnetic disk or tape drive.

Preferably, the cell controller includes a potentiostat and a signal processor.

Preferably, the apparatus is for stripping voltammetric analysis of an analyte in an electrochemical cell.

In accordance with a second aspect of the present invention, an electrochemical cell comprises a container comprising a bottom portion, an upper portion and an opening to permit a liquid to be analysed to be introduced into the container; a stirring device depending from the upper portion into the bottom portion of the container; and a counter electrode and a working electrode depending from the upper portion into the bottom portion of the container.

Preferably, the axis of rotation of the stirring device is substantially co-axial with the central vertical axis of the container and the counter and working electrodes are arranged around the stirring device.

Typically, the upper portion of the container, may be separated from the bottom portion of the container to permit liquid to be inserted into the container prior to engagement of the upper portion with the bottom portion.

Typically, the bottom portion may be in the form of a jar or other liquid containing device and the upper portion may form a lid for the jar or containing device.

Preferably, the electrochemical cell further comprises a reference electrode which also depends from the upper portion of the container such that the reference electrode is adjacent the stirring device and located between the counter and the working electrodes.

Preferably, the electrochemical cell is adapted to be used with the apparatus in accordance with the first aspect of the invention.

In accordance with a third aspect of the present invention, a coupling device for electrically coupling a conductor to an electrode for an electric chemical cell comprises a first conducting member; a second conducting member, electrically coupled and movably coupled to the first conducting member; and a biasing device to bias the first and the second member away from each other; the first conducting member being adapted to be coupled to an electrical conductor and the second member having an engagement portion adapted to engage with and electrically couple the second member an electrode for an electrochemical cell.

Preferably, one of the first and the second members is slidably located within the other of the first and the second members such that the engagement portion of the second portion is biased away from the first member by the biasing device.

Typically, the second member is slidably located within the first member.

Preferably, the engagement surface of the second member is of a material which provides a low electrical contact resistance with the material of the electrode with which the engagement surface is to be engaged. For example, the material of the engagement portion may be a material which is harder than the material of the electrode, such as titanium or iridium, or a material which has a low susceptibility to oxidation, such as gold.

Typically, the first member may include an aperture in the first member adapted to receive a generally cylindrical electrically conducting member such that the conducting member when inserted into the aperture is electrically coupled to the first member.

An example of apparatus for voltammetric analysis in accordance with the invention will now be described with reference to the accompanying drawings, in which:- Figure 1 is a schematic diagram of apparatus for voltammetric analysis; Figure 2 is a side view of an electrochemical cell for use in the apparatus of Figure 1; Figure 3 is a cross-sectional view of an upper portion of the electrochemical cell shown in Figure 2; Figure 4 is a front view of the upper portion shown in Figure 3; Figure 5 is a cross-sectional view of an electrode for use in the cell shown in Figures 2 to 4; Figure 6 shows the apparatus of Figure 1 incorporated into a handheld voltammetric analyser; Figure 7 shows the apparatus of Figure 1 incorporated into an on-line voltammetric analyser; Figure 8 is a flow diagram illustrating the operation of a diagnostic program for use with the apparatus shown in Figures 1,6 and 7; and, Figure 9 is a graph showing calibration results and test results obtained using the analyser shown in Figure 6.

Figure 1 is a block diagram of voltammetric analysis apparatus 1 which includes a microprocessor 2, an electrically erasable programmable read only memory (EEPROM) 3, a display device 4 such as a liquid crystal display device, a memory device 5 which may be a random access memory (RAM), an interface device 6 such as an RS 232, a potentiostat 7, a cell controller 8, a signal processor 9, and an input device, such as a keypad 10.

The cell controller 8 is adapted to be coupled to an electrochemical cell 11 which is generally in the form of a container with electrodes 14,15,16 and a stirrer 17. An example of an electrochemical cell 11 suitable for use with the apparatus shown in Figure 1 is shown in more detail in Figures 2 to 4.

The interface device 6 permits the apparatus 1 to be linked to a computer, for example, to permit programming of the EEPROM 3 or to permit downloading of the memory 5 to a more permanent storage device in the form of for example, magnetic media such as a magnetic disk or tape.

The potentiostat 7 is coupled to the microprocessor 2 and generates a scanning potential which is modified with various waveforms such as DC, AC, square wave or differential pulse, in accordance with a chosen program downloaded from the EEPROM 3 to the microprocessor 2.

The cell controller 8 is coupled to the microprocessor 2 and contains a group of switches. According to the chosen program it will switch on or off the electrochemical cell 11 to allow potential generated by the potentiostat 7 to be applied to the electrodes 14,15,16 and to allow signals detected by the electrodes 14,15,16 to be received by the signal processor 9. It also turns on and off the stirrer 17.

The signal processor 9 is coupled to the microprocessor 2. In accordance with the chosen program it will receive signals from the cell 11, process the signals, convert them into digital information, and pass them to the microprocessor 2 to be stored by the microprocessor 2 in the memory 5.

As shown in Figure 2, the electrochemical cell 11 includes a container comprising a vessel 12 and an electrode holder 13 in the form of a lid for the vessel 12. The lid 13 supports a working electrode 14, a reference electrode 15 and a counter electrode 16 as well as the stirring device 17. The stirring device 17 comprises an electric motor 18 mounted on top of the lid 13, a drive shaft 19 extending through the lid 13 into the vessel 12 and a rotor 20 located at the end of the drive shaft 19 and adjacent to lower ends of the electrodes 14,15,16.

Typically, the stirrer 17 is a 0.1W stirrer and the vessel 12 is a glass bottle with a volume of approximately 20ml. The working electrode 14 may be a 3mm diameter disc glassy carbon electrode, the counter electrode 16 may be a 3mm x 3mm platinum electrode and the reference electrode 15 may be a Ag/AgCl electrode.

The reference electrode 15 has a conventional reference electrode construction. A cross-sectional view of the electrode construction for the working and counter electrodes 14,16 is shown in Figure 5. As shown in Figure 5, each of the electrodes 14,16 comprise an electrode sensing material 50 held in an electrode body 51. In the electrodes 14,16 the sensing material 50 is glassy carbon and platinum, respectively. The body 51 encloses a coupling device which comprises an outer casing 52, a contact pin 53 slidably mounted within the casing 52 and a biasing device 54 within the casing 52. The biasing device 54 acts to bias the pin 53 to an extended position in which a contact point 55 of the pin 53 is biased against the sensing material 50 to make electrical contact with the sensing material 50. The pin 53, biasing device 54 and casing 52 are all electrically conducting and the end of the casing 52, opposite to

the end from which the pin 53 extends, is electrically coupled to a wire 56 which couples the sensing material 50 to the controller 8.

In order to ensure a good electrical contact between the contact point 55 and the sensing material 50, the pin 53, or at least the contact point 55, is formed from a material which is harder than the sensing material 50. This helps ensure a good electrical contact as the contact point 55 "bites" into the sensing material 50. For example, the pin 53 could be manufactured from titanium or iridium.

The lid 13 of the cell 11 is shown in more detail in Figure 3 which shows a cross-sectional view of the lid 13 and Figure 4 which shows a top view of the lid 13. The lid includes a central through bore 21 through which the drive shaft 19 extends and four side through bores 22,23,24,25. The working electrode 14 extends through the bore 23, the reference electrode 15 extends through the bore 24 and the counter electrode 16 extends through the bore 25. The bore 22 is left empty and may be used for introduction of analyte and/or electrolyte into the vessel 12.

Due to the relative simplicity of the apparatus 1, the apparatus can be incorporated into a relatively small handheld analyser 30 as shown in Figure 6. The typical dimensions of the handheld analyser 30 are 40mm x 100mm x 200mm and the overall weight is typically approximately 0.5kg.

The handheld voltammetric analyser 30 incorporates the apparatus shown in Figure 1 with the exception of the electrochemical cell 11. In particular, the handheld voltammetric analyser 30 includes the LCD display 4, the keypad 10, the communication interface 6 and a port 31 which permits the analyser 30 be connected to an external power supply.

However, as an alternative to, or in addition to, the external power supply, the handheld analyser 30 may be powered by an internal battery, such as any form of suitable conventional rechargeable battery. Typically, the battery may be recharged by the external power supply via the port 31 when necessary.

The remainder of the components, including the microprocessor 2, the EEPROM 3, the RAM 5, the potentiostat 7, the cell controller 8 and the signal processor 9 are contained within the housing of the analyser 30.

The handheld analyser 30 also has a communication port 33 which is coupled to the electrochemical cell 11 in use and is coupled internally to the cell controller 8. This permits the cell controller 8 to control the scanning voltage applied to the electrochemical cell and to receive the output signals from the electrochemical cell in response to the applied scanning voltage. In addition, the handheld analyser 30 may optionally be connected through the interface 6 to a remote computer, for example, to transfer data to the remote computer and/or to be remotely controlled by the remote computer.

In addition, the apparatus shown in Figure 1 can be incorporated into an on-line voltammetric analyser 60 as shown in Figure 7. The on-line analyser 60 is coupled via the interface 6, local controller 34 and interface 35, to a remote computer 36 which is used to remotely control the analyser 60.

The analyser 60 and the local controller 34 are powered by a power supply 32 which may be battery operated or powered by a mains power supply 37. In the analyser 60, the key board 10 is omitted, as instructions may be entered into the analyser 60 via the remote computer 36. However, apart from the omission of the key board 10, the analyser 60 is identical to the handheld analyser 30, described above and shown in Figure 6. The on-line analyser 60 is coupled via cable 61 and connection port 33 to the electrochemical cell 11 and to metering pumps 62,63,64,65,66 which can be operated to pump liquid from reagent reservoirs 67,68,69, a sampling valve 70 and a rinsing water valve 71, respectively into the electrochemical cell 11. In addition, a vent valve 72 in the electrochemical cell 11 can also be controlled by the remote computer 36 or by local controller 34.

The liquid containing the sample to be analysed is connected to the sampling valve 70, clean water is connected to the rinsing valve 71 and standard reagents are stored in the reagent reservoirs 67,68,69. The vent valve 72 is used to empty the electrochemical cell 11 of liquid.

In use, the handheld analyser 30 and the on-line analyser 60 are set up by programming into the EEPROM 3 digital information corresponding to the configuration of the electrochemical cell 11 to be used with the analyser 30,60.

Such information includes details of the working electrode, reference electrode, stirrer and vessel. For example, typical information may be that the cell contains a 3mm diameter disc glassy carbon electrode, a 3mm by 3mm platinum counter electrode, a Ag/AgCl reference electrode, a 0.1W stirrer, a 20ml vessel and an electrode holder to fix each electrode position.

The second step is to formulate a reagent with a known composition as a testing media and convert it into digital information. The formulated reagent will contain a standard analyte, a buffer, a masking agent, a supporting electrolyte, a preservative and a performance enhancer. The standard analyte with known concentration is used to calibrate the cell 11. The concentration range is typically from 1ppb to 1000ppm. The buffer is used to control pH and usually contains a salt and an acid or a base. The masking agent is used to reduce interference from samples, which may be a complex agent, an organic solvent, a precipitation agent, a redox agent, a salt, etc. The supporting electrolyte is an ionic compound to provide conductivity. The preservative prolongs shelf life of the reagent. The performance enhancer can increase selectivity and sensitivity of the reagent.

A voltammetric analysis is then performed under fixed experimental conditions such as scan waveform, initial potential, scan rate, final potential, current range, etc.

This arrangement makes a voltammetric response position (potential) easily identifiable. The conditions can be obtained according to published papers, previous experience and expert knowledge. All data is converted into digital information.

Voltammetric response is searched for automatically by the analyser 30,60. The voltammetric response potential is determined for the highest current reading in a certain time period while voltammetric response current is calculated by subtracting background current from current reading. This procedure is programmed into the EEPROM 3.

System testing is then performed using the standard reagent.

As cell configuration, testing media and experimental conditions are fixed, voltammetric response to the analyte in the testing media should be a constant. Any significant changes of response potential or current will indicate system errors such as reference electrode failure or working electrode fouling. The purpose of the system testing step is to find and display these errors by comparing measured values with pre-set values. This procedure is programmed into the EEPROM 3.

The electrodes are then calibrated automatically.

Voltammetric analysis of the standard reagent is carried out in the testing media. The response potential and current is stored. This procedure makes sure that the electrodes are calibrated just before sample analysis so that the electrode surface condition remains unchanged. This calibration procedure is programmed into the EEPROM 3.

A test sample is then analysed. This is performed by voltammetric analysis of a test sample by mixing a known volume of the test sample with a known volume of the standard reagent. The response potential and current is compared with stored response for the standard reagent and the result is calculated and reported. This procedure is also programmed into the EEPROM 3.

The EEPROM 3 also includes a diagnostic program which is illustrated schematically in the flow diagram shown in Figure 8. In particular, the diagnostic program checks 70 the results of the analysis to ensure that a peak current is detected in the voltammetric analysis. If the peak current is not detected, the microprocessor displays 71 an error message in the LCD 4 and stops 72 the analysis. The diagnostic program also uses the reference potential value range stored in the data in the EEPROM 3 and compares this with the peak current response at maximum intensity to check 73 the reference electrode status. If the reference potential value is out of range, the analyser outputs 74 an error message to the operator and stops 75 the analysis. The diagnostic program also uses the reference current data in the EEPROM 3 and compares this with the maximum response intensity obtained during analysis to check 76 gain and working electrode status.

If the maximum response intensity detected is out of range of the reference current data, the diagnostic program causes the microprocessor to instruct the signal processor 9 to change 77 to a more suitable gain level and the analysis is continued.

If the diagnostic program detects that no gain is available, the microprocessor outputs 78 an error message to the operator and stops the analysis 79. This permits the diagnostic program to optimise the signal collection potential range and current range which is stored in the memory 5.

In addition, the diagnostic program uses the reference data stored in the EEPROM 3 which is compared with the actual peak current response obtained from the standard reagent to determine whether the peak current obtained from standard reagent corresponds to the peak current expected from the standard reagent. In the event that the peak current obtained is outside a certain deviation from the expected peak current, the microprocessor will output an error or warning message to the operator.

After programming of the EEPROM 3, the analyser 30,60 can be used in conjunction with the cell 11 to run a voltammetric analysis on a particular analyte. Initially, an operator turns on the analyser 30,60 being used and enters the analyte to be analysed and the concentration of interest. The operator then formulates a standard reagent for the analyte of interest and adds this to the electrochemical cell 11 through the aperture 22.

The analyser 30,60 then conducts a test on the reagent to check the conditions of the system automatically.

After this test, electrode status and instrument readiness will be reported and some analysis conditions such as gain and peak position will be set automatically. Any system errors detected by the diagnostic program will stop analysis with correction instructions. This enhances the performance and reliability of the apparatus.

As soon as the instrument is ready, the system is calibrated automatically. During the calibration process, the system collects and processes raw data and stores a response which represents a known concentration of the analyte to be tested for in the memory.

After the calibration process, a fixed volume of the test sample is added into the cell 11 and mixed with a fixed volume of the standard reagent. The system then collects and processes the voltammetric raw data again. The response which represents the concentration of the standard plus the sample is stored in the memory. The microprocessor 2 performs a calculation to determine the concentration of the analyte in the test sample using the information from the calibration sample and the result will be reported and displayed.

An example of use of the analyser 30 and cell 11 to analyse the concentration of Cu^{2+} in a waste water sample will now be described. However, the analyser 60 could be used in the same manner to perform the analysis.

A standard reagent containing 2 ppm copper nitrate, 0.5 M acetic acid, 0.5 M sodium acetate and 20 ppm mercury chloride was prepared. The formulation of the reagent was digitally coded and entered into the EEPROM 3. In addition, a linear anodic stripping voltammetry scan was also programmed into the EEPROM 3 with the following conditions: (i) An initial potential of -0.8V; (ii) Pre-electrolysis for 30s; (iii) A scan rate of 100mVs⁻¹; (iv) A final potential of +0.5V; and (v) An electrode cleaning time of 20s at +0.5V.

10ml of the reagent was then introduced into the vessel 12 through the aperture 22 as a standard and the voltammetric analysis process was started using the keypad 10. When started the microprocessor 2 prompts a user via the display 4 to enter the analyte to be tested for. The operator in response to this enters "copper". The microprocessor 2 then prompts a user via display 4 for the concentration of interest and the user enters "1ppm" using the keypad 10. In response to this information, the microprocessor 2 selects the appropriate program stored in the EEPROM 3 for an analyte of copper with a concentration of interest of 1ppm and this is downloaded from the EEPROM into the microprocessor 2. The microprocessor 2 then runs the chosen program and in response to the program the microprocessor 2 generates control signals to control the output from the potentiostat 7 to the cell controller 8 and the signal processor 9 and the cell controller 8 directly. By causing the cell controller 8 to apply the pre-programmed linear anodic stripping voltammetric conditions (i) to (v) listed above which form part of the program downloaded from the EEPROM 3 into the microprocessor 2. Using the output signals from the reference and counter electrodes 15,16 which are temporarily stored by the microprocessor 2 in the RAM 5, the microprocessor 2 then analyses the test results and stores the results as a calibration.

After the calibration is completed, the microprocessor 2 prompts a user via the display 4 to add the actual test sample. At this point, the user replaces the 10ml of the standard calibration reagent with 5ml of the calibration reagent and 5ml of the waste water sample to be analysed. The operator then informs the analyser 30 using the keypad 10 that the cell 11 contains the sample to be tested and the analyser repeats the linear anodic stripping voltammetry scan under the same conditions as that conducted for the standard calibration sample.

The microprocessor 2 then compares the results of the test sample with the calibration sample and outputs the concentration of Cu²⁺ found in the test sample by means of the display 4.

In addition, an operator can subsequently or simultaneously download the results to the computer 36 for permanent storage and/or for graphical display and/or printing of the results.

A typical graphical display obtained from the analyser 30 is shown in Figure 9 for the above example. The graph shows the applied voltage versus the sensed current for the calibration sample (A) containing 1ppm Cu²⁺ ions and the waste water sample (B). It can be seen from the graph that sample B has a peak which is approximately 1.5 times greater than the calibration sample A indicating that the concentration of Cu²⁺ ions in the waste water sample B is approximately 2.2ppm. It should be noted that the same graphical display would be obtained using the analyser 60 for the analysis instead of the analyser 30.

In addition to copper, the apparatus 1 and electrochemical cell 11 can be used to detect concentrations of most ions.

Examples of typical ions that can be detected are ions of titanium, vanadium, chromium, manganese, iron, cobalt, nickel, zinc, gallium, germanium, arsenic, silver, cadmium, indium, tin, antimony, tungsten, platinum, gold, mercury, thallium, lead and bismuth, as well as organic compounds such as aromatics, aldehydes, alcohols, ketones, ethers, quinones, halides, heterocyclics, nitrocompounds, amines, phenols, organic acids and organic metallics.

In fact the apparatus 1 and the cell 11 can be used to detect any ion which can be detected using conventional voltammetry.

Concentrations that can be detected can be any concentration which can be detected using conventional voltammetric analysis, and typically any concentration in the range from approximately 1ppb to 1000ppm.

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QUANTUM QUALITY CONTROL

DOUBLE Q C

QQC (trademarked)

A DEVICE FOR TESTING THE TRIVECTOR ELECTRICAL SIGNATURE OF HOMEOPATHIC ITEMS

Description of Device:

Thus device is designed to capture a polographic or voltammetric electrical signature pattern of a liquid compound. Electro chemical analysis of compounds is a tried and tested method of analysis. A review article is contained in the appendix.

The International Journal of the Medical Science of Homeopathy has published a series of articles on this technique. First the early process of analysis was heralded in 1997, and later reviewed in the 2005 volume. Copies of these articles are in the appendix.

The device uses a set of electrodes made of different metals. The different metals invoke an electro-potential. This variant electro potentials will vary the displaced electrons to reflect the electro potential variations to reflect the substance changes.

The device will send a low level variant current thru a substance to be tested. The changes in the potential are then measured thru this scale. A second pass will vary voltage and measure current variations.

Changes in the magnetic or inductance field will be measured. And changes in the dielectric or static field will be reflected in the measurement.

Thus an electro-magnetic-static picture will evolve from the test. Thus three dimensional reflection of the liquid crystal structure of the substance will be measured. This three dimensional field is termed the Trivector (trademarked).

The Trivector field reflects the electro-signature of any item tested.

Potential of measurement:

The system operates in a range of Voltages distributed from zero to four volts. The amperage current ranges from zero thru 4 milliamps. The system is designed to test substances not for patient testing.

Potential for Homeopathic Enhancement:

The QQC system can run an energy into a homeopathic substance to measure the electrical trivector voltammetric field or the energetic signature. The energy of the measure effects the tested entity and alters it. The QQC system can be used thus alter or improve a homeopathic. This is called the enhancement of the product. The zeta flickering rate of the water is effected, and the trivector field can be stabilized for use.

Portray of System

Show diagram ATTACHED

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Family list

5 application(s) for: **EP1636575 (A1)**

1	VOLTAMMETRIC DETECTION OF METABOLITES IN PHYSIOLOGICAL FLUIDS	<input type="checkbox"/> in my patents list
	Inventor: LEE-DAVEY JON [GB] ; MALECHA MICHAEL MARKUS [GB] (+2) EC: G01N27/48; G01N33/487B2	Applicant: UNIV CRANFIELD [GB] IPC: G01N27/48; G01N33/487; G01N27/48; (+1)
	Publication info: CA2531033 (A1) — 2005-01-06	

2	VOLTAMMETRIC DETECTION OF METABOLITES IN PHYSIOLOGICAL FLUIDS	in my patents list <input type="checkbox"/>
	Inventor: SAINI SELWAYAN [GB] ; BESSANT CONRAD [GB] (+2) EC: G01N27/48; G01N33/487B2	Applicant: UNIV CRANFIELD [GB] IPC: G01N27/48; G01N33/487; G01N27/48; (+2)
	Publication info: EP1636575 (A1) — 2006-03-22	
3	VOLTAMMETRIC DETECTION OF METABOLITES IN PHYSIOLOGICAL FLUIDS	in my patents list <input type="checkbox"/>
	Inventor: EC: G01N27/48; G01N33/487B2	Applicant: IPC: G01N27/416; G01N27/27; G01N27/28; (+9)
	Publication info: JP2007534926 (T) — 2007-11-29	
4	Voltammetric detection of metabolites in physiological fluids	in my patents list <input type="checkbox"/>
	Inventor: SAINI SELWAYAN [GB] ; BESSANT CONRAD [GB] (+2) EC: G01N27/48; G01N33/487B2	Applicant: IPC: G01N33/00; G01N27/48; G01N33/487; (+3)
	Publication info: US2007105232 (A1) — 2007-05-10	
5	VOLTAMMETRIC DETECTION OF METABOLITES IN PHYSIOLOGICAL FLUIDS	in my patents list <input type="checkbox"/>
	Inventor: SAINI SELWAYAN [GB] ; BESSANT CONRAD [GB] (+2) EC: G01N27/48; G01N33/487B2	Applicant: UNIV CRANFIELD [GB] ; SAINI SELWAYAN [GB] (+3) IPC: G01N27/48; G01N33/487; G01N27/48; (+3)
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- *40. "Fast-Scan Stripping Voltammetry Imaging with Scanning Electrochemical Microscopy," David O. Wipf, Mario A. Alpuche Aviles, and Luis Diaz-Ballote at Journées d'Électrochimie 2003, Poitiers, France, June 3-6, 2003.
- #*39. "Multidimensional Imaging in Scanning Electrochemical Microscopy", David O. Wipf, Université Claude Bernard, Lyon, France, May 23, 2003. **(Invited)**
- #*38. "Multidimensional Methods in Scanning Electrochemical Microscopy", David O. Wipf, Université de Paris VI, Jussieu, Paris, France, May 13, 2003. **(Invited)**
- *37. "Scanning Electrochemical Microscopy Examination of the O₂ Reduction on Cast Iron - E.E.-D. El-Giar and David O. Wipf, at the 203rd Meeting of the Electrochemical Society, Paris, France, April 27-May2, 2003.
- #*36. "Multidimensional Imaging with Scanning Electrochemical Microscopy", David O. Wipf, University of Warwick, England, Feb. 27, 2003. **(Invited)**
- #*35. "Multidimensional Scanning Electrochemical Microscopy", David O. Wipf, Truman State University, Kirksville, MO, Sept. 27, 2002. **(Invited)**
- *34. "Constant-Distance Scanning in SECM," David O. Wipf, Mario A. Alpuche Aviles, and Emad El-Deen M. El-Giar at PittCon 2001, New Orleans, LA, March 4-9, 2001.
- #*33. "Applications of Scanning Electrochemical Microscopy," David O. Wipf, CINVESTAV Unidad Mérida, March 16, 1999, Mérida, Yucatan, Mexico. **(Invited)**
- #*32. "Microscale Surface Chemistry with the Scanning Electrochemical Microscope," David O. Wipf, at the XIV National Congress of the Mexican Electrochemical Society, August 24-28, 1999, Mérida, Yucatan, Mexico. **(Invited)**
- #*31. "Measurement of Local Electron-Transfer Rates on Electrodes with SECM," David O. Wipf and Robert C. Tenent at the 25th Annual Conference of the Federation of Analytical Chemistry and Spectroscopy Societies, October 11-15, 1998, Austin TX. **(Invited)**
- #*30. "Micro-Scale Electrochemistry with the Scanning Electrochemical Microscope," David O. Wipf, Auburn University, AL, May 15, 1998. **(Invited)**
- #*29. "Heterogeneously Modified Carbon Fiber Ultramicroelectrodes," D. Wipf, R. C. Tenent, F. Ge, and J. Baur at the 193th Meeting of the Electrochemical Society, May 5, 1998, San Diego CA. **(Invited)**
- *28. Modified Carbon Fiber Ultramicroelectrodes," D. O. Wipf, R. C. Tenent, F. Ge, at Mississippi EPSCoR Conference, January 22, 1998, Jackson, MS.
- #*27. Investigation of Localized Corrosion with the Scanning Electrochemical Microscope," J. W. Still and D. O. Wipf, at the 1st International Workshop on Scanning Electrochemical Microscopy, September 7, 1997, Freiburg, Germany **(Invited)**
- *26. Formation and Imaging of Heterogeneously Modified Carbon Fiber Ultramicroelectrodes," D. O. Wipf, R. C. Tenent, F. Ge, at Electrochem '97 August 29, 1997, London, England
- #*25. Local Modification of Electrode Surfaces by the Scanning Electrochemical Microscope," D. O. Wipf, University of South Dakota, November 18, 1996. **(Invited)**

- #*24. Local Modification of Electrode Surfaces by the Scanning Electrochemical Microscope," D. O. Wipf, University of Wisconsin - Eau Claire, November 15, 1996. **(Invited)**
- #*23. Local Modification of Electrode Surfaces by the Scanning Electrochemical Microscope," D. O. Wipf, University of Southern Mississippi, October 25, 1996. **(Invited)**
- *22. Examination of Passive Layer Breakdown and Formation with the Scanning Electrochemical Microscope", John W. Still and David O. Wipf at the 190th Meeting of the Electrochemical Society, Oct. 10, 1996, San Antonio, TX.
- #*21. Local Modification and Imaging of Surfaces by the Scanning Electrochemical Microscope," D. O. Wipf, Paper #8, Great Lakes Regional Meeting of the American Chemical Society, May 19-22, 1996, Normal, IL. **(Invited)**
- #*20. Local Modification and Imaging of Surfaces by the Scanning Electrochemical Microscope," D. O. Wipf, April 4, 1996, presented at the University of Alabama, Tuscaloosa. **(Invited)**
- #*19. Local Modification and Imaging of Surfaces by the Scanning Electrochemical Microscope," D. O. Wipf, March 8, 1996 presented at Illinois State University, Normal, IL. **(Invited)**
- *18. Initiation of Localized Corrosion on Iron with the Scanning Electrochemical Microscope," David O. Wipf and John W. Still, Paper #746, Pittsburgh Conference and Exposition, March 6, 1996, Chicago, IL.
- *17. Generation and Imaging of Derivatized Regions on Carbon Electrodes by the Scanning Electrochemical Microscope," David O. Wipf, Robert C. Tenent, and Louis H. Bluhm+, Paper #745, Pittsburgh Conference and Exposition, March 6, 1996, Chicago, IL.
- *16. Analytical Applications of Paired Carbon-Fiber Electrodes," John E. Baur and David O. Wipf, Paper #097, Pittsburgh Conference and Exposition, March 6, 1996, Chicago, IL.
- *15. Initiation of Pitting Corrosion on Iron with the Scanning Electrochemical Microscope," David O. Wipf and John W. Still, Mississippi EPSCoR Conference, Jan. 31, 1996, Jackson, MS, No. 4.
- *14. Ohmic Potential-Drop Compensation by Iterative Correction of the Applied Potential," D. O. Wipf, 47th Southeast / 51st Southwest Joint Regional Meeting of the American Chemical Society, Nov. 29-Dec. 1, 1995 Memphis, TN, No. 83.
- #*13. Scanning Electrochemical Microscopy", D. O. Wipf, Oct. 6, 1995 presented at Tennessee Technological University, Cookeville TN. **(Invited)**
- *12. Activation of Carbon Electrodes by UV/Ozone Treatment," D. O. Wipf and J. Zhou, Paper #256, 187th Electrochemical Society Meeting, May 21-26, 1995, Reno NV.
- #*11. Formation and Study of Localized Corrosion by Scanning Electrochemical Microscopy," David O. Wipf, April 20, 1995, presented at Florida State University, Tallahassee FL. **(Invited)**
- #*10. Initiation and Study of Localized Corrosion with the Scanning Electrochemical Microscope," David O. Wipf, October 21, 1994, presented at Jackson State University, Jackson MS. **(Invited)**
- *9. Examination of Pitting Corrosion with Scanning Electrochemical Microscopy," David O. Wipf, presented at 1994 SEAAC (Southeast Academic Analytical Chemistry) Meeting, Florida State University, Tallahassee FL, September 23-24, 1994.
- #*8. Formation and Study of Pitting Corrosion by Scanning Electrochemical Microscopy," David O. Wipf, Paper #202, presented at the 208th American Chemical Society National Meeting and Exposition, Washington DC, August 21-25, 1994. **(Invited)**
- #*7. Scanning Electrochemical Microscopy" David O. Wipf, presented at the University of Mississippi, Oxford, MS, January 21, 1994. **(Invited)**
- #*6. Initiation and Study of Localized Corrosion by Scanning Electrochemical Microscopy," David O. Wipf, *Surface Characterization of Adsorption and Interfacial Reactions*, Engineering Foundation Conference, January 9-14, 1994, Kona, HI. **(Invited)**
- 5. Constant-Current Imaging with the Scanning Electrochemical Microscope," David O. Wipf and Allen J. Bard, Paper #811, Pittsburgh Conference and Exposition, March 10, 1993, Atlanta, GA.
- 4. Tip-Modulated Scanning Electrochemical Microscopy," David O. Wipf and Allen J. Bard, Paper #944, Pittsburgh Conference and Exposition, March 11, 1992.
- #3. Scanning Electrochemical Microscopy for Characterization of Electrochemical and Chemical Processes at Surfaces," David O. Wipf, Guy Denuault, and Allen J. Bard, presented at the SE/SW Combined Regional Meeting of the ACS, December 6, 1990. **(Invited)**
- 2. Measurement of Heterogeneous and Homogeneous Rate Constants with Fast-Scan Cyclic Voltammetry," David O. Wipf and R. Mark Wightman, presented at the International Electroanalytical Symposium, May 28, 1987.
- 1. Measurement of Heterogeneous and Homogeneous Rate Constants with Fast-Scan Cyclic Voltammetry," David O. Wipf and R. Mark Wightman, presented at the Pittsburgh Conference and Exposition, March 9, 1987.

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- **Student and Collaborator Presentations**

- *45. "An Electrochemical STM Study of Coarsening and Corrosion of Platinum Films," Qingmin Xu, Eric Kreidler, David Wipf, and Ting He at the 212th Meeting of the Electrochemical Society, Washington DC, Oct 7-12, 2007.
- *44. "Application of Combined Stripping Voltammetry Techniques and SECM to Electro-Deposition and Generation Studies" M. A. Alpuche Aviles and D. Wipf, 210th Meeting of The Electrochemical Society and the XXI Congreso de la Sociedad Mexicana de Electroquímica Joint International Meeting, Cancun, MX, Oct 29 – Nov 3, 2006.
- 43. "Simple Fabrication of a Silver Epoxy Coated Microelectrode for SECM Imaging in Small Volumes," L. F. Diaz-Ballote and D. Wipf, 210th Meeting of The Electrochemical Society and the XXI Congreso de la Sociedad Mexicana de Electroquímica Joint International Meeting, Cancun, MX, Oct 29 – Nov 3, 2006.
- *42. "Low Temperature Oxygen Reduction Reaction Kinetics on Polycrystalline Pt Electrodes," Xiaojing Sun, David O Wipf, The 57th Southeast/61st Southwest Regional Meeting, Memphis, TN, November 1-4, 2005.
- *41. "A Modulated Mass-Transfer Technique for Voltammetry at Low Concentrations," A. Antonyraj, David O. Wipf, The 57th Southeast/61st Southwest Regional Meeting, Memphis, TN, November 1-4, 2005.
- *41. "Scanning Electrochemical Microscopy – A Useful Tool for Surface Characterization of Composite Materials," L. Díaz-Ballote, M.A. Pech-Canul, M.I. Pech-Canul, L. Veleva and David O. Wipf, XIV International Materials Research Congress 2005, Cancún, Quintana Roo, México, August 21-25, 2005.
- *40. "Evaluation of VGCF/Paint-Coated Mild Steel Using Electrochemical Impedance Spectroscopy (EIS) in 3% NaCl Solution," Sahar M. Atwa and David O. Wipf, 2004 Southeast Regional Meeting of the ACS, Research Triangle Park, NC, November 10-13, 2004.
- *39. "Visualization of the Electrochemical Activity of Silicon Carbide Particles in Aluminum Composites by Scanning Electrochemical Microscopy," L. Díaz-Ballote, David O. Wipf, M. Pech Canul, and L. Veleva, XIII International Materials Research Congress, Cancún Mexico August 22-26, 2004.
- #*38. "Imaging Model Neurons With the Scanning Electrochemical Microscope," John E. Baur, Ruwan Kurulugama, Paul A. Garris, and David O. Wipf, at The 226th ACS National Meeting, New York, NY, September 7-11, 2003.
- *37. Stripping Voltammetry Imaging With Scanning Electrochemical Microscopy - M. Alpuche-Aviles David O. Wipf, at the 203rd Meeting of the Electrochemical Society, Paris, France, April 27-May 2, 2003.
- *36. Electrochemical Study and Surface Analysis of Passive Films on AISI 316 Stainless Steel Grown in Alkaline Solutions," Lucien Veleva, Mario A. Alpuche-Aviles, Melissa K. Graves-Brook, and David O. Wipf at the 15th International Corrosion Congress, Granada, Spain, September 22-27, 2002.
- *35. Electrochemical Detection for Microchip Separation Devices," Charles S. Henry, Yan Liu, Joseph C. Fanguy, David O. Wipf at the 201st Meeting of The Electrochemical Society in Philadelphia, PA, May 12-17, 2002.
- *34. Constant-Separation Imaging of Live Mammalian Cells with the Scanning Electrochemical Microscope Using Carbon Ring Probes," John E. Baur and David O. Wipf at PittCon 2002, New Orleans, LA, March 17-22, 2002.
- *33. Improving Impedance Feedback Control in Scanning Electrochemical Microscopy," Mario A. Alpuche Aviles and David O. Wipf at PittCon 2002, New Orleans, LA, March 17-22, 2002.
- *32. Scanning Electrochemical Microscopy Imaging with pH Sensitive Iridium Oxide Microelectrodes," Emad El-Deen M. El-Giar and David O. Wipf at PittCon 2002, New Orleans, LA, March 17-22, 2002.
- *31. Fast Scan Cyclic Voltammetry-Scanning Electrochemical Microscopy," Luis Díaz-Ballote and David O. Wipf at PittCon 2002, New Orleans, LA, March 17-22, 2002.
- *29. Conductivity Detection for Microchip Capillary Electrophoresis," Charles S. Henry, David O. Wipf, Jamie Vollenweider, at SmallTalk2001, San Diego, CA, Aug. 29-31, 2001.
- *28. An Impedance Feedback Method for Examination of Corrosion with SECM," Mario A. Alpuche Aviles and David O. Wipf at PittCon 2001, New Orleans, LA, March 4-9, 2001.
- *27. A Microparticle Iridium Oxide pH Ultramicroelectrode for SECM," Emad El-Deen M. El-Giar and David O. Wipf at PittCon 2000, New Orleans, LA, March 4-9, 2001.
- 26. Characterization of a Conductivity Detector for Microchip Capillary Electrophoresis," Yan Liu, David O. Wipf, and Charles S. Henry at the 52nd Southeast / 56th Southwest Joint Regional Meeting of the American Chemical Society, New Orleans, LA, Dec. 6-8, 2000.

- *25. In-Channel Conductivity Detection for Microchip Capillary Electrophoresis," Jasper R. Clarkson+, Charles S. Henry, and David O. Wipf at the 52nd Southeast / 56th Southwest Joint Regional Meeting of the American Chemical Society, New Orleans, LA, Dec. 6-8, 2000.
- *24. Shear Force Distance Regulation for Scanning Electrochemical Microscopy," Mario A. Alpuche Aviles and David O. Wipf at PittCon 2000, New Orleans, LA, March 12-17, 2000.
- *23. A New Procedure for Construction of Small Carbon-Fiber Electrodes," Emad El-Deen M. El-Giar and David O. Wipf at PittCon 2000, New Orleans, LA, March 12-17, 2000.
- *22. Direct Observation and Kinetic Measurements of Active Sites on Glassy Carbon Electrodes", Robert C. Tenent and David O. Wipf at PittCon '99, Orlando, FL, March 7-12, 1999.
- *21. Direct Observation and Kinetic Measurements of Active Sites on Glassy Carbon Electrodes," Robert C. Tenent and David O. Wipf Mississippi EPSCoR Conference, Jackson, MS, March 2-3, 1999.
- *20. Metal-Metal Bonding in M₂(form)₄ Complexes," Kathryn M. Carlson-Day, Judith L. Eglin, Laura T. Smith, Richard J. Staples, and David O. Wipf, Mississippi EPSCoR Conference, Jackson, MS, March 2-3, 1999.
- *19. Studies of Heterogeneously Modified Carbon Electrodes by Scanning Electrochemical Microscopy," Robert C. Tenent and David O. Wipf at PittCon '98, March 1-5, 1998, New Orleans, LA
- *18. Localized Passive Layer and Corrosion Studies of Various Aluminum Alloys Using the Scanning Electrochemical Microscope", John Still and David O. Wipf at PittCon '98, March 1-5, 1998, New Orleans, LA
- 17. Localized Avidin/Biotin Derivatization of Glassy Carbon Electrodes Using Scanning Electrochemical Microscopy", Wilbur B. Nowall, David O. Wipf, and Werner G. Kuhr at the Electrochemistry Gordon Conference, January 18-23 1998, Ventura CA
- *16. Use of Formamidine Ligands in the Synthesis of Quadruply Bonded Dichromium Complexes", Kathryn M. Carlson-Day, Judith L. Eglin, Laura T. Smith, Richard J. Staples, and David O. Wipf, Mississippi EPSCoR Conference, January 21- 22, 1998, Jackson, MS
- 15. Synthesis of Dichromium Formamidinate Complexes" Kathryn M. Carlson-Day, Judith L. Eglin, Laura T. Smith, Richard J. Staples, and David O. Wipf, 214th National Meeting of the ACS, September 11 - 15, 1997, Las Vegas, Nevada.
- *14. A Method for the Heterogeneous Derivatization of Carbon-Fiber Ultramicroelectrodes", Robert C. Tenent and David O. Wipf at PittCon '97, March 16-21, 1997, Atlanta, GA
- *13. Localized Corrosion Studies of Iron using the Scanning Electrochemical Microscope," John W. Still, David O. Wipf, Mississippi EPSCoR Conference, Jan. 29, 1997, Jackson, MS, No. 9.
- *#12. Development of a Scanning Electrochemical Microscope for the Surface Modification of Carbon Fiber Electrodes," W. B. Nowall, S. E. Rosenwaldt, W. G. Kuhr, R. Tenent and D. O. Wipf, *Microfabricated Sensors, Instruments and Systems for Biological and Medical Applications*, May 6-8, 1996, University of California, Davis.
- *11. UV/Ozone Pretreatment of Carbon Electrodes," Junfeng Zhou and David O. Wipf, Paper #112P, Pittsburgh Conference and Exposition, March 6, 1996, Chicago, IL.
- *10. Patterning and Imaging of Conducting Polyaniline with the Scanning Electrochemical Microscope," David O. Wipf and Junfeng Zhou, Mississippi EPSCoR Conference, Jan. 31, 1996, Jackson, MS, No. 21.
- *9. Surface Modification and Characterization of Carbon Electrodes," L. H. Bluhm+ and D. O. Wipf, 47th Southeast / 51st Southwest Joint Regional Meeting of the American Chemical Society, Nov. 29-Dec. 1, 1995 Memphis, TN, No. 240
- *8. Generation and Imaging of Locally Oxidized Regions on Carbon Electrodes by Scanning Electrochemical Microscopy," R. C. Tenent and D. O. Wipf, 47th Southeast / 51st Southwest Joint Regional Meeting of the American Chemical Society, Nov. 29-Dec. 1, 1995 Memphis, TN, No. 20
- *7. Study of Pitting Corrosion Using Iron in Aqueous Trichloroacetic Acid," J. W. Still and D. O. Wipf, 47th Southeast / 51st Southwest Joint Regional Meeting of the American Chemical Society, Nov. 29-Dec. 1, 1995 Memphis, TN, No. 82
- *6. UV/Ozone Treatment to Activate Carbon Electrodes," J. Zhou and D. O. Wipf, 47th Southeast / 51st Southwest Joint Regional Meeting of the American Chemical Society, Nov. 29-Dec. 1, 1995 Memphis, TN, No. 84
- *5. Chemical Activation of Carbon Electrodes," L. H. Bluhm+ and David O. Wipf, presented at the 27th Annual Southeast Regional American Chemical Society Conference of Undergraduate Student Chemists, Clemson, SC, March 16-17, 1995
- *4. Examination of Pitting by Scanning Electrochemical Microscopy," Jeannette C. Polkinghorne+, John W. Still, and David O. Wipf, presented at the 209th Meeting of the American Chemical Society, Anaheim, CA, April 2-6, 1995.

- *3. Examination of Pitting by Scanning Electrochemical Microscopy," Jeannette C. Polkinghorne+, John W. Still, and David O. Wipf, presented at the 47th Southeast Regional Meeting of the American Chemical Society, Birmingham, AL, October 16, 1994.
- *2. Electrochemical Investigation of the Reduction of Trichloroacetic Acid in Aqueous Solution," David O. Wipf, John Still, at the Advances in Modern Nuclear Magnetic Resonance Techniques, June 9-11, 1994 Mississippi State University, John Still, presenter.
- *1. Chemical Oxidation of Glassy-Carbon Electrodes," David O. Wipf, Jiwen Chen, at the Advances in Modern Nuclear Magnetic Resonance Techniques, June 9-11, 1994 Mississippi State University, Jiwen Chen, presenter.

• Theses and Dissertations

- "Electrode Modification by UV/Ozone Treatment and Scanning Electrochemical Microscope Polyaniline Deposition," Junfeng Zhou, M. S. Thesis, Mississippi State University, 1997.
- "A Study of Localized Corrosion by Scanning Electrochemical Microscope," John W. Still, Ph.D. Dissertation, Mississippi State University, 1999.
- "Localized Modification and Studies of Carbon Electrode Surfaces by Scanning Electrochemical Microscopy," Robert C. Tenent, Ph.D. Dissertation, Mississippi State University, 2000.
- "Preparation and Scanning Electrochemical Microscopy (SECM) Applications of Carbon Fiber and Metal Particle Ultramicroelectrodes (UMEs)," Emad El-Deem Mohamed El-Giar, Ph.D. Dissertation, Mississippi State University, 2004.
- "Development of Multidimensional Methods for Scanning Electrochemical Microscopy (SECM)," Mario Alberto Alpuche Avilés, Ph.D. Dissertation, Mississippi State University, 2005.
- "Study of the Oxygen Reduction Reaction on Platinum with Scanning Electrochemical Microscopy and Rotating Disk Voltammetry," Xiaojing Sun, M.S. Thesis, Mississippi State University, 2007.