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Development of a Bipolar Pulse Conductometric Detector for Environmental Monitoring and Electrochemical Experiments

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환경수질오염 검증과 전기화학실험을 위한 양극펄스형 전기전도성검출기의 개발

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ABSTRACT. A bipolar pulse conductometric detector has been developed for continuous on-line monitroring of the flowing sample streams. The conductometric detector was fast and easy to use due to the free of interferences by serial or parallel capacitances. The performance of the bipolar pulse conductometric detector was tested using dummy cells with $10^1 \sim 10^8$ ohm resistance range and found a linear response with a 0.01% relative standard deviation. The possible applications of the bipolar pulse conductometric detector was discussed.

요 약. 연속적으로 흐르는 시료의 검출을 위한 양극펄스형 전기전도성 검출기를 개발하였다. 개발한 전기전도성 검출기는 동작이 매우 빠를 뿐더러 직렬 혹은 병렬축전 방해효과가 없으므로 사용이 쉽다. 개발한 양극펄스형 전기전도성 검출기의 성능을 $10^1 \sim 10^8$ 저항을 가진 가상 셀들을 사용하여 시험한 결과 0.01% 상대편차를 보이는 직선성 응답을 얻을 수 있었다. 개발한 전기전도성 검출기의 장단점과 여러 가능한 응용들에 대해서도 검토하였다.

Key Words: Conductometer, Bipolar Pulse Conductometer

1. INTRODUCTION

Recently, there has been a wide interest in environmental monitroring because industrial wastewater can contaminate river and drinking water seriously. Automated environmental monitroring systems with fast response and high sensitivity are required for continuous monitoring and early necessary activity to stop further release of hazardous wastewater. There are many analytical systems for environmental wastewater analysis including chromatography, UV/VIS spectrophotometry, atomic absorption, and mass spectrometry. But, for routine and continuous monitoring of environmental samples, the above analytical methods are tedious and require time consuming processes for sample preparation and handling. Environmental wastewater analysis by electrochemical methods, especially conductometry, is fast and easy to set up to the flowing stream for continuous on-line monitoring.

Traditionally, conductance of substances was measured by bridge technique. But, the bridge conductance measurement is not readily applicable to systems which require continuous or instantaneous conductance measurements. Furthermore, the conductance measurement using conventional instrumentation shows nonlinear results for many situations, especially in those systems with very low or high resistance.[1] The origin of these undesirable effects is due to the complicated nature of electrochemical systems involved. Usually, conventional electrochemical conductance measurement system is associated with the series and parallel capacitances, contact and lead resistances, and a faradaic impedance. These extraneous factors become increasingly important when the solution resistance is either very low or very high.

In this paper, the development of a bipolar pulse conductometric detector which is fast and easy to use is described. The performance of the bipolar pulse conductometric detector and possible applications are also discussed.

2. CIRCUIT DESCRIPTION

The block diagram of the bipolar pulse conductometric detector developed is shown in *Figure* 1. The function generator(ICL8038) generates bipolar square wave (±15V, 3kHz) and its output is fed to a CMOS switch (CD4007) input. The other input of the CMOS switch is divided by two by a D-type flip-flop(74LS74) and the Q output of the flip-flop is level shifted(±15V) to drive the CMOS switch. Therefore, the bipolar square wave and the ground signal will pass through the CMOS switch alternatively so that one cycle of the square wave is followed by one cycle of the ground signal. The clipper using two zenor diodes accurately adjusts pulse

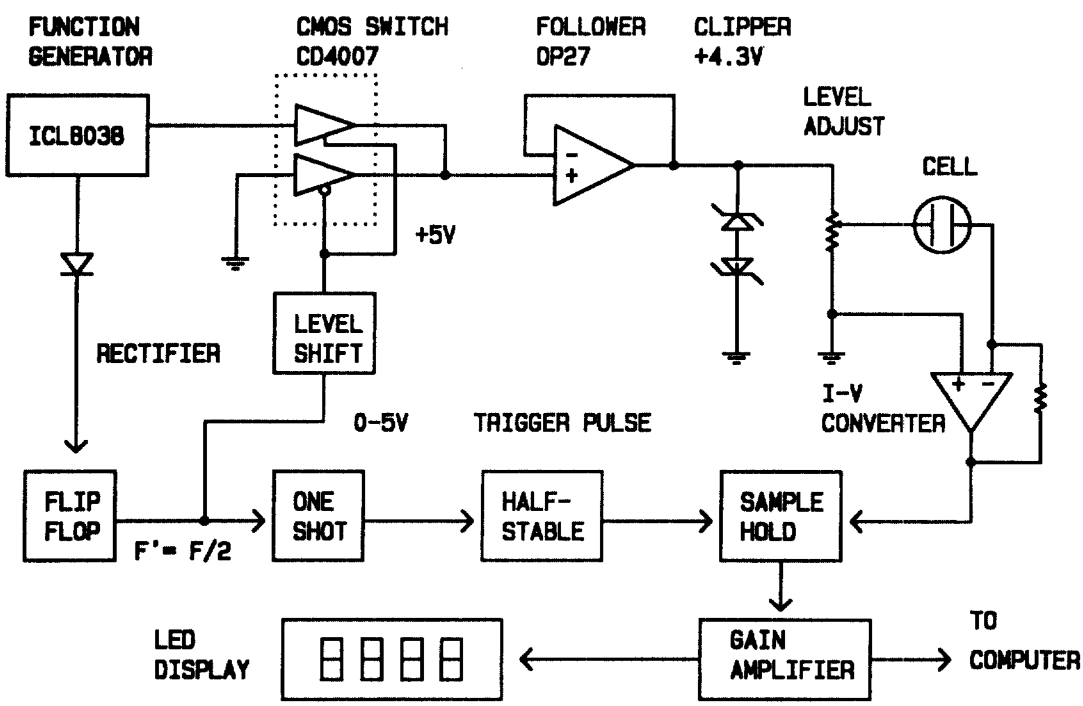

Figure 1. Functional block diagram of the bipolar pulse conductometric detector.

height to ±4.3V and the level adjusted bipolar pulses are applied to the conductance cell. The conductance cell is made with two Pt wires separated by 0.5cm apart. The current flow through the cell is measured by the current-to-voltage converter(I-V converter) using an operational amplifier(OP27). Two selector switches are used to adjust the pulse height and the gain of the I-V converter. A one-shot(74LS121) is used to adjust the sampling point during the second half cycle of the bipolar pulse. A half stable circuit generates sampling pulses at the end of the second half cycle of the bipolar pulses. Since the conductance cell is excited by the series of pulses, the sample-and-hold(LF398) is used for gated signal detection as shown in *Figure* 2. The sampled signal is further amplified and its output is displayed by a four digit seven segment light emitting diodes(LEDs) using a 12-bit analog-to-digital converter(ICL7107).

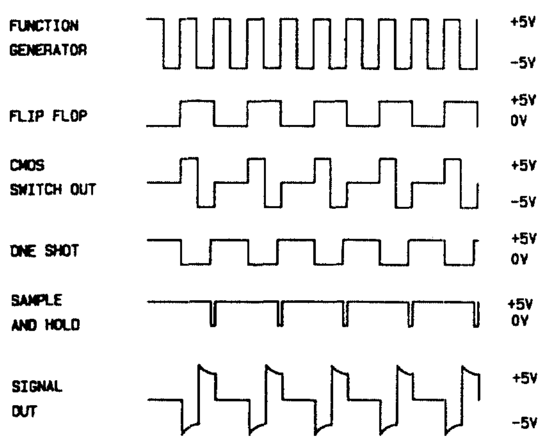

Figure 2. Timing diagram of the bipolar pulse conductometric detector.

3. RESULTS AND DISCUSSION

The biploar pulse concept for conductance measurement was originally developed by Johnson and Enke[2] which eliminates many of the classical experimental problems encountered with other methods. The bipolar pulse conductance measurement technique involves the application of two successive constant voltage pulses of equal magnitude and pulse with but of opposite sign sequentially to a conductance cell. The resulting current is sampled at the end of the second pulse. Therefore, the bipolar pulse technique is accurate and independent of series and parallel capacitances. In addition, this method has the advantage of being capable of measuring very rapid changes in conductance.

The performance of the bipolar pulse conductometer was tested with a dummy cell using precision resistors ($10^1 \sim 10^8$ohm). The inverse of the measured intensity which is the conductance plus a finite offset showed a linear change with 0.01% relative standard deviation of the full scale as shown in *Figure* 3. Although the conductometric analysis is limited due to its nonselective nature, it may be an ideal detection method which require a universal detector such as in liquid chromatography. The conductometric titration for the end point determination can be applied to the numerous substances. The big advantage of the conductometric titration over other titration methods is its high sensitivity which can be applied to the titration of very dilute solutions($10^{-5} \sim 10^{-7}$M) and to systems in which the reaction between the titrant and analyte is relatively incomplete.[3] For example, phenol, boric acid and other quite weak acids can be successfully titrated in aqueous solution conductometrically, which is very difficult to locate their end point potentiometrically.

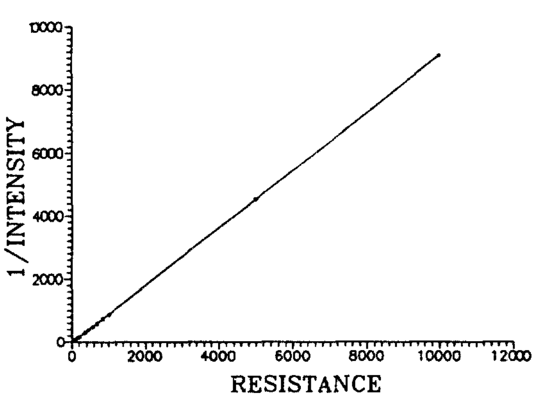

Figure 3. A plot of the inverse of the measured intensity vs resistance of the dummy cell in kilo-ohm unit.

The percent change in conductivity during the course of titration is the same regardless of the concentration of the solution. Also, the good linearity revealed by the conductometric titration requires only 3～4 titration data before and after the end point.

In addition, the conductometric studies of electrolyte solutuions may present valuable knowledges of the properties and behavior of the solutes in solutions. In particular, there have been intensive investigations of the ionization and association of solutes as a function of dielectric constant and of the acidic or basic character of the solvent.[4,5]

4. CONCLUSIONS

A bipolar pulse conductometric detector has been developed for continuous on-line monitoring of the flowing sample stream. The conductometric detector was fast and easy to use due to the free of interferences by the serial or parallel capacitances. The high sensitivity and linear response over eight decasdes of the solution resistance make it ideal detector for various liquid sample analysis. The possible applications of the bipolar pulse conductometric detector are numerous including liquid chromatography, flow injection analysis, environmental wastewater monitring and electrochemical experiments. Furthermore, it can be interfaced to a microcomputer for automated data acquisition and experiment control with the least expense. Various possible applications of the developed bipolar pulse conductometric detector are under investigation.

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