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A BIPOLAR DIGIPOTENTIOMETER FOR ELECTROANALYTICAL USES

Direct Conversion of Charge to a Digital Number*

William W. Goldsworthy and Ray G. Clem

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University of California
Berkeley, California 94720

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BRIEF

A bipolar digital potentiostat based upon the principle of charge injection or extraction has been built, tested and applied to polarographic and anodic stripping analyses. It is capable of 0.01% precision.

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ABSTRACT

A bipolar digipotentiometer has been built for the first time. It functions as a potentiostat through pulsed injection or extraction of charge to maintain a control potential, and simultaneously serves as a current to digital converter. Counting and summing these pulses in time allows the instrument to serve as an integrator. It is capable of a measuring precision of 0.01%. This device is the heart of a new system which includes a digital wait-gait, a pulse height analyzer, an analyzer interface, an incremental differentiator, a voltage-step ramp generator, and a program timer. Uses of this system in polarography and anodic stripping analysis are illustrated and possible uses in controlling or digitizing charge in other systems are discussed.

INTRODUCTION

Great technological advances in the field of solid-state physics have resulted in the development of small, inexpensive electronic components which are extremely fast in operation and are capable of controlling either hole or electron current with equal ease while consuming very little power. Although these properties portend digital type applications, most designers prefer to employ the devices using the analog circuit designs and signal measuring techniques developed in the vacuum-tube era. It seems redundant to convert charge, liberated in a detection process, into an analog of current or voltage, perform various analog operations on these quantities, then finally convert them to numerical form. If at all possible, it would be far better to deal directly and immediately with this liberated charge on a digital basis at the output of the detector system itself, since the number of steps required in the conversion of detector output signals into useful digital numbers would be reduced.

Several advantages are gained by employing the direct conversion of charge to a digital number technique described herein. Due to the reduction in the number of steps required between detection and numerical output, considerable savings can be realized in the weight, size, cost, and power requirements of the instrument. Phase shifts, which result on passing a signal through many operational amplifiers and can lead to deleterious oscillations, are generally obviated. And, since the detector output is immediately converted to numerical form, data can be easily and promptly transmitted to remote locations at signal levels much greater than background for more sophisticated analysis.

The presented bipolar digipotentiogrator is a direct outgrowth of the previously developed digital integrator which served as a voltage to frequency converter in our first electroanalytical effort (1). The same principle was later applied to the digitizing of charge liberated in a nuclear-event detector and resulted in the development of a digital nuclear spectrometer (2). Both of these initial instruments served passively to neutralize incoming charge to a preset null through digital feedback of charge of the opposite polarity. Neither was designed to perform a control function on the external system, although in some systems it seemed reasonable that they could be made to do so. The validation of this reasoning took the form of the monopolar digipotentiogrator described in a recent correspondence (3). The present bipolar device is a more sophisticated instrument and is more generally applicable to electroanalytical problems. It is presented as the heart of a new system which includes a digital wait-gate, a pulse height analyzer for data storage, an analyzer interface, an incremental digital differentiator, a voltage-step ramp generator, and a program timer.

Possible uses of this instrument in controlling or digitizing charge in systems other than electrochemical are also briefly discussed below. It is quite likely that a small computer could be programmed to perform the same control and timing functions as the presented device through the use of software.

INSTRUMENTATION

The system shown in Figure 1 is a complete divergence from previous electroanalytical systems. The familiar train of instruments, a potentiostat, a current to voltage converter, and an analog to digital converter are absent.

In their place, one finds a single device, the bipolar digipotentiogrator, which functions as a potentiostat through pulsed injection or extraction of charge to maintain a desired cell control potential, and simultaneously serves as a current to digital converter. If the current pulses are counted and summed in time, the device subsequently functions as an integrator. Discussion of the system will begin with the digipotentiogrator, then proceed to the other modules required for unit operation.

Since the reader probably has no precedence for understanding the operation of this instrument, an overview guided by the block diagram of Figure 2 will be given, followed by a detailed discussion of the more critical parts.

The cell reference electrode is connected through a unity-gain, impedance coupling amplifier to the inputs of the two differential comparitors. The logic circuitry in turn senses the need for rebalance current and controls the rate of pulses current required to satisfy the cell current demands at the control potential selected. Cell potential control is effected either through a local potential source or an external source applied to the other input terminals of the differential comparitor pair. A resistor connected between the signal inputs of comparitors and driven with a constant current source generates a potential separation between the two comparitors to prevent "chattering".

Pulses derived from the operation of the current gates are fed into either a scaler, which serves as a gross accumulator, or into a pulse height analyzer operated in the multichannel scaling mode where they are distributed as a function of time. These pulses can also be scaled to readout directly on

a scaler the weight of sample oxidized or reduced by varying the magnitude of the gated constant current sources.

In addition to the gated current sources, continuous constant current sources of either polarity are available in switch selectable steps for the zero offsetting of the ranges.

Figure 3 gives detailed information concerning the operation of the rebalancing and digitizing logic block shown in the previous figure. All timing cycles are derived from a local 500 kHz crystal clock. Whether or not the constant current gate is opened depends upon a decision made in the less than 1- μ sec potential sampling interval which occurs between possible current drive intervals. This approach prevents the occurrence of partial width drive pulses which would obviously deteriorate the precision of the method. If the cell reference potential exceeds the sense level of the comparator in one sampling interval, a current pulse having a 1- μ sec duration will appear and will reappear in each subsequent clock cycle until the comparator voltage (cell potential) is driven below the sense level of the input comparator. This explains the thatched appearance of the continuous control potential response shown in Figure 2 of Reference 3.

The block diagram, depicting one of the two balance sensing and digitizing logic circuits is shown in the lower portion of Figure 3 and is a further aid to understand the rebalancing and digitizing logic process. A delay shaper gate is employed to give sampling pulses narrower than the interval between clock pulses and to center these therein. This signal is applied simultaneously to one terminal of the "ON"-gate and to one terminal of the "OFF" gate. The second terminal of the ON-gate is driven by the output signal of one of the comparators. When the input of the comparator is

positive, indicating its threshold level has been exceeded, a signal appears at the output of the ON-gate upon the arrival of the first sampling pulse. This in turn sets the flip-flop by switching it to its Q state. The Q output is applied to one terminal of the "OUTPUT"-gate whose other terminal is driven by clock pulses. When the requisite input conditions are thus satisfied, a clock output of 1- μ sec duration appears at the output of this gate. This signal is used to synchronously drive the attached constant current source.

One terminal of the OFF gate is driven with the sampling pulse. Its other terminal is driven with the inverted output of the comparator. When the input level of the comparator drops below the sense threshold, an output signal is developed from the OFF gate which resets the flip-flop, and thus blocks further generation of drive signals from the OUTPUT gate.

The success of the digital technique hinges upon our ability to drive a cell with a constant current device, since the auxiliary electrode potential can vary over a wide potential range.

Pulses from the output of the synchronous charge injection gate are applied to the switching drive input of a pulsed constant current source driver as shown in Figure 4. Resistors R_1 and R_2 allow coarse and fine adjustments, respectively, for the constant current emanating from transistor Q_1 . This constant current will flow through diode D_1 into the collector of the switching transistor Q_2 unless an input drive pulse is applied to its base. During the drive pulse interval, the potential at the collector of Q_2 is pulled up to +12V through the resistor combination, R_3 and R_4 , resulting in the back-biasing of diode D_1 . Since current can no longer flow through D_1 and Q_2 , it must now pass through D_2 into the auxiliary electrode of the cell.

Capacitor C_2 is employed to neutralize the effects of shunt capacity to ground at the junction of D_1 , D_2 , and Q_1 , thus insuring the proper switching of current at low levels.

In addition to the digipotentiogrator, several other pieces of equipment are required to complete the system. Figure 5, the voltage-step ramp generator, was built specifically for this system. Separate adjustments of both the upper and lower limits of the $\pm 2V$ potential scan range are incorporated. The ramp is capable of scanning in either the cathodic or anodic direction. The step-height is switch selectable to a 1-mV increment or in increments of 2 mV from 2- to 20-mV. The step-heights can be individually calibrated in each switch position through adjustment of high-quality, metal-film trim resistors. The rise-time of the potential step is less than 100- μ sec. Very low drift is assured by employing a monolithic, dual FET input in the active operational amplifier integrator.

The ramp, which can be connected to the control inputs of the digipotentiogrator, provides incremental cell potential drive. The ramp can be stepped upon command from a dual time-base, high-precision, digital timer (LRL 15 $\times$ 841) when employing a solid electrode, or upon sensing the instant of drop-fall when using a DME. In either application, the ramp can send a stop signal through the interface to the analyzer when it reaches the scan limit, thus terminating the experiment.

The dual time-base, digital timer can also be used as a digital wait-gate unit which can be externally trigger or, alternatively, operated in a free running mode. The first of its timing periods may be used to delay the time of signal sampling and the last to determine the data sampling interval.

This allows the accurate synchronization of the data acquisition cycle with the DME drop period. The precision for a 1-sec wait or gate interval is better than one part in a million.

Start, stop, reset, and trigger commands to the digital timer are supplied with the main programmer and analyzer interface unit. See Figure 6. The interface also permits the controlling of the memory sense, address and storage functions of the analyzer. Read, stop, and analyze modes of the analyzer generate signals to control directly the data acquisition cycle. These signals activate the run line in the interface and thus initiate the start of the program and the stepping of the ramp. They can also terminate the data acquisition period, and effect the automatic resetting of the ramp and digital timer. Incremental derivative polarography (1) is made possible through appropriate interface control logic, so that at a given cell potential, current information is stored first subtractively. After incrementing, current information is then added to the same data storage channel.

To do anodic stripping analysis, a switch selectable timing delay period of multiples of minutes can be set employing an electromechanical timer, thus permitting amalgam accumulation on the electrode prior to the anodic going scan.

In addition to the foregoing, much of the equipment termed "miscellaneous" in the previous paper is re-employed here (1). The new system also includes a Cipher Model 70 magnetic tape recorder and a Northern Model 406M tape controller. The latter unit permits communication between the analyzer and the tape in either a READ or a WRITE mode.

Figure 7 shows the time relationship of the detection and data processing functions of the completed instrument while performing various polarographic-type operations.

Line A shows the typical timing trigger pulses generated by the digital wait-gate unit when it is in its free-running mode. These pulses simultaneously step the ramp and advance the analyzer address with each generated interval of delay and sampling. Line B illustrates a typical pulse-train emanating from the digipotentiogrator when working with a solid electrode. The periodic, anodic going pulses shown in line C, which are discussed in more detail in the following section, are typical of the response obtained in DME studies. Each time drop separation occurs, a void appears in the digital reduction output. During this void interval, pulses appear in the oxidize output line for perhaps 100- μ sec. The first oxidize pulse in this burst develops a drop separation trigger pulse. See line D. This trigger pulse is fed into the analyzer interface unit where it initiates the data acquisition and time synchronization cycle in the programmer and simultaneously advances the ramp one step.

Line E depicts the analog equivalent of the pulse-train of line C. Line F shows the delay period developed by the digital sequential timer while line G indicates the sampling period during, which data is accumulated, and line H, the data pulses stored at an appropriate analyzer address.

Line J depicts the ramp generator stepping to new potential values while lines K and M show successively how the data can be stored in each address additively for integral storage or, stored first subtractively then additively in the same address to generate an incremental derivative.

EXPERIMENTAL

Reagents. Supporting electrolytes and standard metal-ion solutions were prepared by dissolving weighed quantities of reagent-grade materials, then

diluting to volume in distilled water. The nitrogen and mercury employed was of the same purity as that described previously (4).

Apparatus and Procedure. The coulometry cell (4), salt-bridge and preparation thereof (5), DME assembly and the Smoler capillary employed (1), were also described previously. Additionally, a modified version of the anodic stripping cell and procedure described by Matson and co-workers was used (6). Prior to voltage scanning all solutions were sparged of oxygen. Subsequently, an oxygen-free atmosphere was maintained with a continuous flow of nitrogen over the solution during the electrochemical operation.

RESULTS AND DISCUSSION

The purpose of this project was to develop a sensitive, inexpensive instrument, employing the direct conversion of charge to a digital number technique, capable of high precision, and of sufficient simplicity to permit its operation by a technician. These objectives were realized.

Despite the fact that the parts for the entire bipolar digipotentiogrator cost less than the control amplifier employed in the previous system (1), the precision of the two systems are directly comparable. See the results below. Since extensive use is made of integrated circuits, the size of the package is quite small. The device is housed in a box measuring 6" x 4 1/2" x 1 1/2". Furthermore, the very low power demands (<1 watt) eminently qualify this instrument for battery-operated, field use.

In practice, once the potential scan range, step-height and time-dwell per channel are set, the operator initiates the experiment by switching the analyzer from DISPLAY through STOP to its START-MEASURE state. Controlling the instrument is therefore elegantly simple. It could be operated by

untrained personnel. In the laboratory, data are accumulated in a preprogrammed manner, and are displayed automatically on the analyzer oscilloscope at the end of the scan. Four programs available are DME or solid electrode polarography, and anodic or cathodic stripping voltammetry. The recorded data can be written onto magnetic tape and subsequently be analyzed with programs compatible with the Control Data 7600 computer. The ability to read previously recorded runs back into the analyzer permits a visual comparison of a presently recorded unknown with a previously recorded standard. Alternatively, in the field, the data can be recorded concurrently with the scan on a small strip-chart recorder and processed manually.

The approach we have taken here to sense the instant of drop fall is much simpler than that used in the previous effort (1). Again see Figure 7, lines C and D. As the drop begins to separate from the capillary, the mercury is drawn out into a thread. At the instant of drop fall, the now broken thread snaps back to or into the capillary with an attendant sudden decrease in surface area. This sudden decrease causes a temporary excess of charge to accumulate on the incipient next drop. The oxidize differential comparator senses this as a potential increase and immediately activates the digital current extractor to eliminate the excess. It is, therefore, only necessary to sense the first pulse of oxidation current demand to start the next timing cycle for the new drop. This approach to drop fall sensing requires only a minimum of electronics and is not critical in adjustment.

The present instrument, with its $\pm 10V$, 10mA constant-current gated sources, has the ability to control the cell potential over a $\pm 2V$ range. The gated current is divisible in switch selectable steps of 2, ranging from 2^0 to

2^{10} with a fine adjustment within each range of a factor of 2. The charge range covered, then, is from 1×10^{-8} to 5×10^{-12} coul/pulse. The maximum current leakage through the closed gates amounts to only 10^{-10} A. The potential-control rise-time of this device is limited primarily by the RC characteristics of the cell, which in turn depends upon the capabilities of the gated current sources. The 50% dead-time is ubiquitous to this and all subsequent devices of this nature. Examination of similarities and differences between analog potentiostats and this digital device will help clarify the above statements.

First of all, it must be recognized that an analog potentiostat, under less than current limiting conditions, is a voltage source; whereas, our device, albeit pulsed, is always a current source. This difference makes it very difficult to elicit a direct comparison between the two.

One might measure the rise-time of a minimally damped analog potentiostat by observing the response differences between the input and output waveforms when, for example, employing a square or sine-wave in conjunction with a purely resistive simulated cell. The response time measured would obviously be longer if an actual cell of large capacitance were employed, since the cell is located in the feedback loop of the control amplifier. Furthermore, the potentiostat may oscillate owing to the attendant phase shift afforded by the capacitance of the cell. Once the specter of oscillation appears, the tedious task of building a stabilization network begins.

In contrast to the foregoing, it is not possible to operate the digitipotentigrator without capacitance being present. Since the device continually alternates between its sense and possible charge pulse states, some memory of

the cell potential must be retained from one sense cycle to the next. This memory is supplied either through the natural capacitance of the cell, or through an external capacitor. How closely the device can control a given potential depends upon the RC time constant of the cell. If, for example, the RC constant is 1- μ sec the control potential would sag $\sim 40\%$ by the end of the potential sensing interval. Because of the necessity of providing a fairly long RC constant in a simulated cell the rise time of the device is increased.

How rapidly the digipotentiogrator can drive a capacitive load depends upon the current capabilities of the gated current sources. To drive, for example, a 1- μ f load from 0V to ± 1 -V when employing the full output of either gated source requires 200- μ sec during which time 100 pulses are recorded. While the response time of the present instrument is probably too slow for some workers, it is adequately fast for our present electroanalytical purposes. The digital resolution is also quite acceptable. The time response could be improved 10-fold through the use of a 100mA gated source, but at the expense of a 10-fold loss in digital resolution. Use of a much higher clock rate would certainly improve the speed of response, however, as yet unexplored but predictable problems await those who might try this approach. It becomes progressively difficult to switch increasingly lower currents at increasingly higher frequencies.

Fortunately, the response time problem is not as great as it might first appear. The response time could be reduced to < 20 μ sec through the use of the successive approximation technique employed by computer designers. The digital resolution, however, would remain the same as that above provided the 10mA - 1- μ sec pulse were retained as the last significant byte. We have not yet needed this degree of complexity.

Judging from our previous observations of stability in analog potentiostats, the present digital device is much superior. The instrument has been used for both anodic stripping and DME polarography, and cursorily for coulometry employing the new rotated mercury cell (4), with no signs of instability. These cells represent quite a wide range of frequency response. The same small value capacitor shown in Figure 2 is used to tailor the high-frequency response of the reference electrode in all the applications just mentioned.

A further stability check was made by simultaneously observing the output response of the device to a sine-wave on a dual trace oscilloscope over the frequency range from 10 Hz to 500 kHz. The dynamic levels were kept low to circumvent the aforementioned limitations regarding the pulsed current sources. The simulated cell had an RC constant of about 10 μ sec. No phase shift or diminution of the output signal was noted up to 100 kHz. Above this frequency some display confusion resulted because the input signal was approaching the clock frequency of the digipotentiogrator.

The drift of this device is 0.02% per hour and the linearity as measured with regression analysis is 0.02% or 0.01% at full scale in the reduce mode or oxidize mode, respectively.

Figure 8 shows a difference polarogram of lead taken at the 6.8×10^{-7} M level. Considering the number of counts in the residual current and on the diffusion plateau, a sensitivity to the 6×10^{-8} M level is attainable under the conditions employed. If smaller values of the gated current source were available and continuous variation of the offsetting constant current source were possible, greater sensitivities could be reported.

Note that since the working electrode is grounded, pickup of 60 Hz line frequency is eliminated.

Ground-loop problems are circumvented since the digipotentiogrator is very small and can be mounted next to the cell. Also, the leads to the cell are kept short.

Figure 9, trace A shows an anodic stripping polarogram of lead and copper taken at the 2.5 and 12 nanomolar levels, respectively. The very large peak at the right is due to the stripping of the added mercury plated during the amalgam accumulation step. The copper and lead peaks are barely discernable in the background. After a ten-fold display expansion of trace A shown in trace B, the lead and copper peaks are much more in evidence. High resolution digital recording is an almost indispensable aid to anodic stripping analysis. It permits the recording of an entire sweep with the option of expanding full-scale, after the fact, any portion thereof for visual inspection. This convenience feature is entirely lacking when analog signal recording is employed. Further, after transferring to magnetic tape, the digital data can be processed with a computer to remove the sloping background contribution and to integrate the peak areas. The digipotentiogrator is presently being used in an environmental study to determine lead in natural waters.

The present device with its $\pm 10V$ pulsed current sources does not have sufficient potential drive to operate the rotated mercury coulometry cell (4)

over the full requisite $\pm 2V$ vs. SCE range, however, it is perfectly stable over the $\sim \pm 500mV$ range it can presently control. Future design of higher voltage sources and gates will result in a more suitable instrument for coulometric applications.

FUTURE WORK

Considering the many threats to the ecology from human, industrial, and transportation wastes, it will be importunate upon the scientific and engineering community to develop adequate means for making the necessary measurements of pollutants. These measurements must be made with adequate accuracy and at a minimum cost since large numbers of analyses will be required to force compliance. The direct conversion of charge to numerical form technique described herein seems to be quite promising in fulfilling these needs and should be considered. A single digipotentiogrator could be made serve many different measurement or control functions. It could, for example, measure sequentially, current related spectrometric data, perform polarography, do constant current titrations, sense temperature, humidity, digitize specific-ion electrode and pH information, plus a host of other functions. The digital output data could be sent continuously, conveniently, and inexpensively by telephone or micro-wave relay to a central processing center for data reduction and analysis. The fact that the instrument is inexpensive, easy to construct, and requires little power makes it very attractive for the above mentioned uses.

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FIGURE CAPTIONS

Fig. 1. Block Diagram of the System.

Fig. 2. The Bipolar Digipotentiogrator.

Fig. 3. The Synchronous Charge Injection Gate.

Fig. 4. Pulsed Constant Current Driver.

Fig. 5. Voltage-Step Ramp Generator.

Fig. 6. Programmer and Analyzer Interface Unit.

Fig. 7. Time Sequence Chart.

Fig. 8. Difference Polarogram of Pb^{2+} at 6.8×10^{-7} M level.

Conditions: Wait = 500 msec

Gate = 700 msec

Gated current source = 5.00 μA (effective)

Solution: 0.1 M KCl.

Fig. 9. Anodic Stripping Voltammogram.

Solution: 15 ml H_2O , 250 μl sat'd.

KCl, and 13.4 μg Hg^{2+} .

Trace A. The entire sweep.

Trace B. Ten fold expansion of selected portions of trace A.

Conditions: Amalgam accumulation time = 20 min.

Voltage step increment = 9 mV/channel.

Dwell time = 2 sec/channel.

Fig. 1

XBL7111-4638

XBL7111-4642

Fig. 2.

XBL7III-4640

Fig. 3

XBL7III-464I

Fig. 4

Fig. 5

XBL7111-4637

Fig. 6

XBL7111-4636

XBL7111-4639

Fig. 7

XBB 7112-6110

Fig. 8

E vs. SCE

XBB 7112-6109

Fig. 9

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