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Part 2

Applied Polarography for Analysis of Ordnance Materials

Part 2. An Inexpensive Solid-State Field Polarograph With Digital and Analog Output

by
Walter J. Becktel
Test and Evaluation Department
and
Gerald C. Whitnack
Research Department

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Naval Weapons Center
CHINA LAKE, CALIFORNIA 93555

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R. G. Freeman, III, RAdm., USN .... Commander
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FOREWORD

The work described in this report is part of a continuing research project entitled "Applied Polarography for Analysis of Ordnance Materials." This work is supported by the Naval Sea Systems Command, Code 0332, under Task Area Number SF57572301 and represents a final report on Phase I of the work covering the period fiscal years 1975 and 1976.

Phase I of the work is divided into two final reports under the above general title. Part 1 is "Determination and Monitoring of 1,2-Propyleneglycoldinitrate in Effluent Water by Single-Sweep Polarography" and Part 2 is "An Inexpensive Solid-State Field Polarograph With Digital and Analog Output."

This report has been reviewed for technical accuracy by Robert L. Fowler and Gordon R. Doyel.

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(U) A new rapid, specific, and unique polarographic method of analysis of 1,2-propyleneglycoldinitrate (PGDN) in effluent water is described. A portable and inexpensive digital polarograph and monitoring system, built at NWC, are presented for the field analysis of PGDN in an effluent water obtained from a Navy carbon column cleanup process to remove the explosive from *Otto Fuel* wastewater. Data obtained by the NWC-developed method of analysis and field equipment compare favorably with data obtained by a vapor-phase chromatographic method on the same samples of effluent water. This report describes in detail the digital polarograph, circuitry, and operational characteristics of the complete polarographic monitoring system.

CONTENTS

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The authors gratefully acknowledge the assistance of personnel at the Naval Torpedo Station, Keyport, Washington, for furnishing the effluent water, and the Naval Ordnance Station, Indian Head, Maryland, for supplying the pure PGDN used in this study.

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INTRODUCTION

Although there are many commercial polarographs available today, there are relatively few with the performance, flexibility, and operational characteristics required for field use in systems needed for monitoring parts-per-billion concentrations of ordnance-derived pollutants in effluent and natural water. Both the polarographs and monitoring systems available are costly and too complex to be used reliably by plant or field personnel. It is the purpose of this report to describe in detail the construction, circuitry, operational characteristics, and performance of a low-cost solid-state digital polarograph that is simple to operate and readily adaptable to field use.

CIRCUITRY AND OPERATIONAL CHARACTERISTICS

A block diagram of the complete digital polarographic unit is shown in Figure 1. The circuitry is shown in the schematic circuit diagram, Figure 2. The component parts and function of each, starting with the master clock, follow.

MASTER CLOCK

The master clock consists of the integrated circuit U_1 and its related components. Essentially, this circuit is a pulse generator producing a selectable pulse width and a selectable duty cycle. R_1, R_2, and C_1 combine to control the pulse delay and R_3, R_4, and C_2 are combined to control the pulse width. Reference to the timing drawings will depict the resultant waveform. Although the pulse width and duty cycle are variable, they are preset to produce a delay of 5 sec and a pulse period of 2 sec. This setting is made by adjustments on the printed circuit board. This particular timing cycle was chosen to make tests made with this unit compatible with previous tests made with other systems. This timing sequence controls directly or indirectly most of the succeeding functions of this system and any changes to its settings will result in changes in other circuit timing.

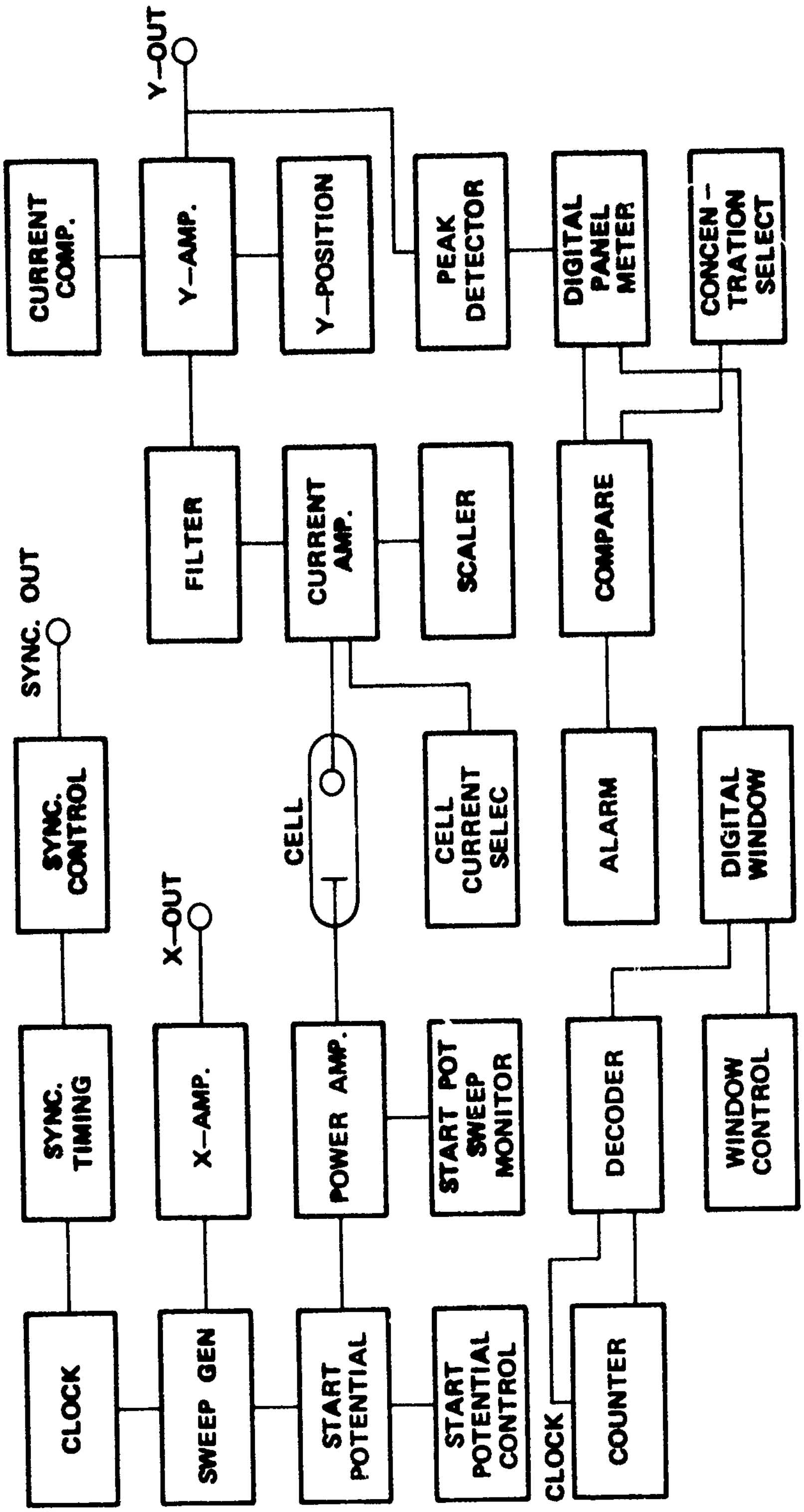


FIGURE 1. Digital Polarograph (Block Diagram).

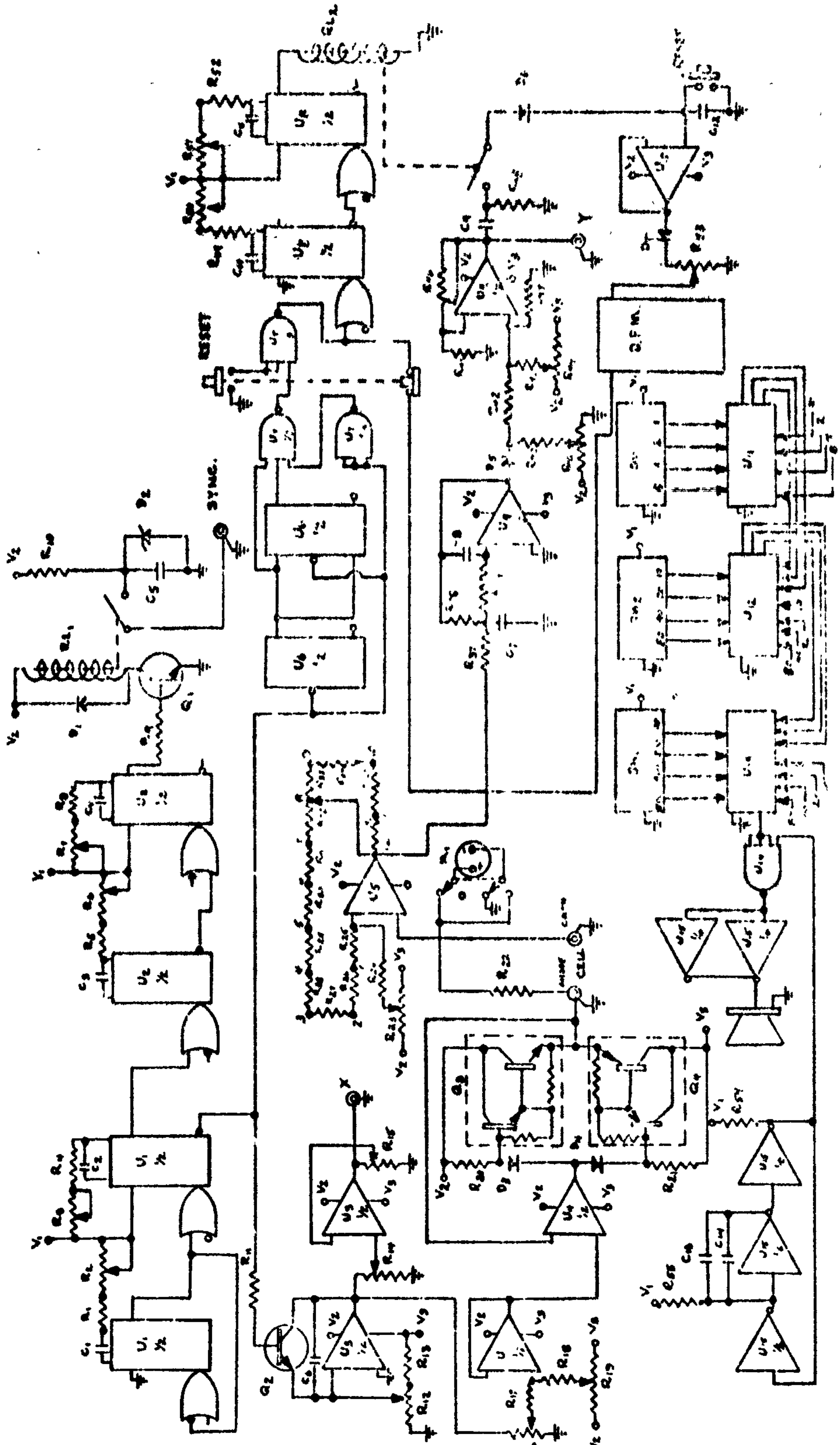


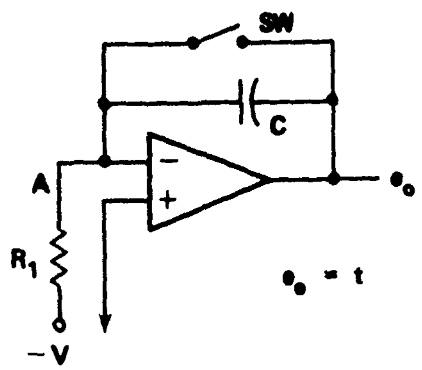
FIGURE 2. Digital Polarograph (Schematic Circuit Diagram).

SYNC

Clock pulses coming from the second half of U_1 are connected directly to the first half of U_2. R_5, R_6, and C_3 combine to produce a variable delay of the master clock pulse. The negation output of the first half of U_2 is sent to the second half of U_2 and R_7, R_8, and C_4 combine to vary the width of this pulse. Sync pulses must occur at the end of the sampling period and U_2 is adjusted via its varying components to perform this function. The output of this pulse control is connected to an electronic switch, Q_1. When the second half of U_2 is high, Q_1 is turned on and its collector goes to its emitter potential. This allows current to flow through RL_1 and actuate this relay closing its contacts. C_5 is charging exponentially through R_{10} and is clamped by D_2 to its Zener potential. During the on time of Q_1, when RL_1 contacts close, this capacitive potential is allowed to flow to the output connector which in turn is connected to an electromechanical device. This potential actuates a device which knocks a drop of mercury from a glass capillary known as a dropping mercury electrode (DME). C_5 was chosen to produce enough energy to actuate this device from the following expression (capacitive energy = $1/2\ CV^2$).

SWEEP GENERATOR

U_3, Q_2, and C_6 combine to form the stage known as the sweep generator. Master clock pulses control this function. The basic function of this stage is to produce a linear ramp which is in sync with the master clock. The ramp generator changes elapsed time into a voltage. When Q_2 receives a low base signal, it acts as a switch and opens. This corresponds to zero time in the formation of the ramp. Waveform No. 3 (Figure 3) shows this control pulse. When this pulse goes low, the ramp begins to form during this time and, as shown in waveform No. 2 (Figure 3), as soon as the control pulse returns to a high, the ramp cuts off.



The above example is used to explain this stage operation. SW is normally closed from the signal Q_2 has received from the master clock. With this signal removed, SW or Q_2 is open, and as can be seen, C can begin to charge.

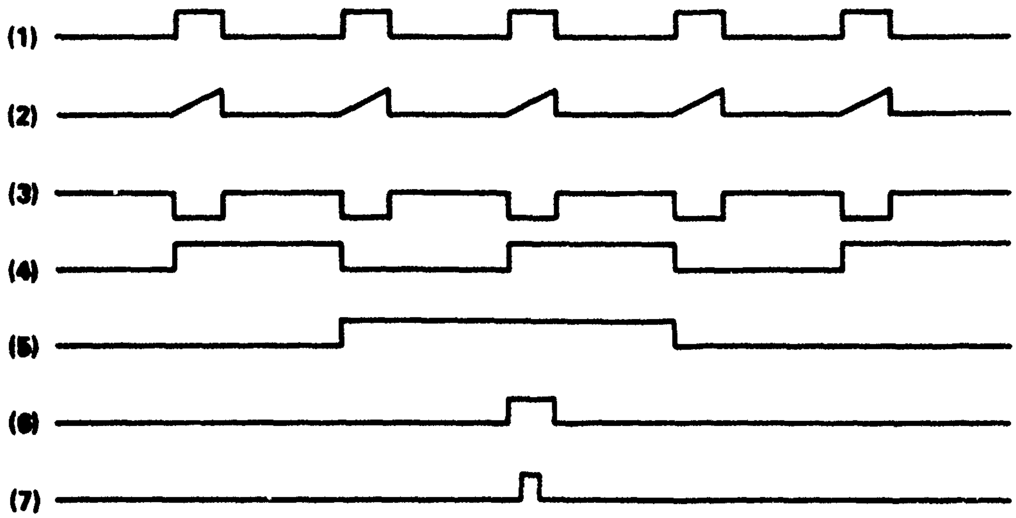


FIGURE 3. Digital Polarograph (Wave Forms).

X-AMP

A portion of the ramp is selected by the voltage divider R_{14} and the second half of U_3 is connected as a noninverting amplifier, with a gain adjust to amplify the signal received from the sweep generator. This amplified signal is available to external equipment via a coaxial connector. R_{15} can be adjusted to cause the x-ordinate to be swept over the desired length.

START POTENTIAL

Another portion of the ramp signal is selected by R_{16} and summed at the input of U_4 with an electromotive force (e.m.f.) of a desired potential from R_{18}. The output of this noninverting amplifier is fed directly to the noninverting input of the power amplifier. The start potential selected by R_{18} can be either a positive or negative potential.

POWER AMP

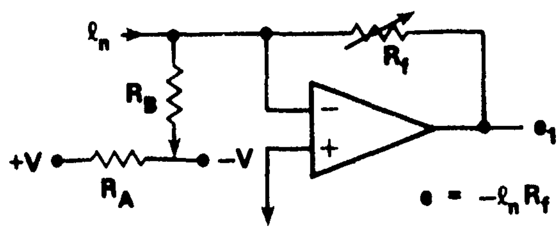
The signal to be impressed across the cell consists of a ramp voltage as well as a start potential. However, the ordinary operational amplifier does not have the capability of possessing enough power to drive this signal across the cell. The first portion of this stage is connected as a voltage follower, possessing a very high input impedance. The feedback is connected to the anode of the cell. Output signals from the amplifier are connected to a complementary pair of Darlington connected transistors, giving an output with great current drive capability and having the capability of handling current requirements from the cell of almost a dead short. D_3 and D_4 act as steering diodes.

START POTENTIAL SWEEP MONITOR

This is the last circuit in this system to be described and it was added to assist the operator in selecting the correct start potential placed across the cell. Because it is placed at the anode, there is the added advantage of monitoring the sweep potential, which appears as a sweep of the meter indicator, in addition to the start potential. If the sweep is not functioning, there will be no sweep movement on the meter. The meter will monitor plus or minus potentials by merely selecting the proper polarity with the polarity switch. This is an analog monitoring device with approximately 20 kΩ/V input impedance.

CELL CURRENT AMPLIFIER

Reference to the circuit schematic (Figure 2) will disclose that this stage is represented by U_5 and related components. Information to this stage comes directly from the cell via the DME and is in the form of a varying electric current. The magnitude of this current is a function of the concentration contained within the solution of the cell and in order to monitor and measure this quantity, a current-to-voltage transducer is employed. The current coming from the cell is fed directly into the summing node of the transducer. The only current-to-voltage error involved is the bias current and this is summed algebraically with the cell current.



For an output voltage of 0.1 V, ℓ_n can be resolved in nano or pico amps. This is made possible by the various values of R that can be selected. Selection of various start potentials will cause a DC offset in the transducer and in order to remove or minimize the effects of this, an e.m.f. of the correct polarity is added at the summing node by adjusting R_A and summing through resistor R_B. On the block diagram (Figure 1) this start potential compensation is known as the scaler.

FILTER

Reference to the block diagram (Figure 1) will disclose that output of the current-to-voltage transducer is connected directly to an active filter stage, comprised of U_9 and related components. A filter is added at this point because extremely high gain settings are used in the cell current amplifier to detect minute quantities, and thus extraneous noise is also amplified. The result is a lowering of the signal-to-noise ratio. The response of the filter is shown in the gain vs. frequency plot (Figure 4). This plot shows a roll-off of better than 6 dB/octave and the 3 dB point is well within the spectrum of information derived from the cell.

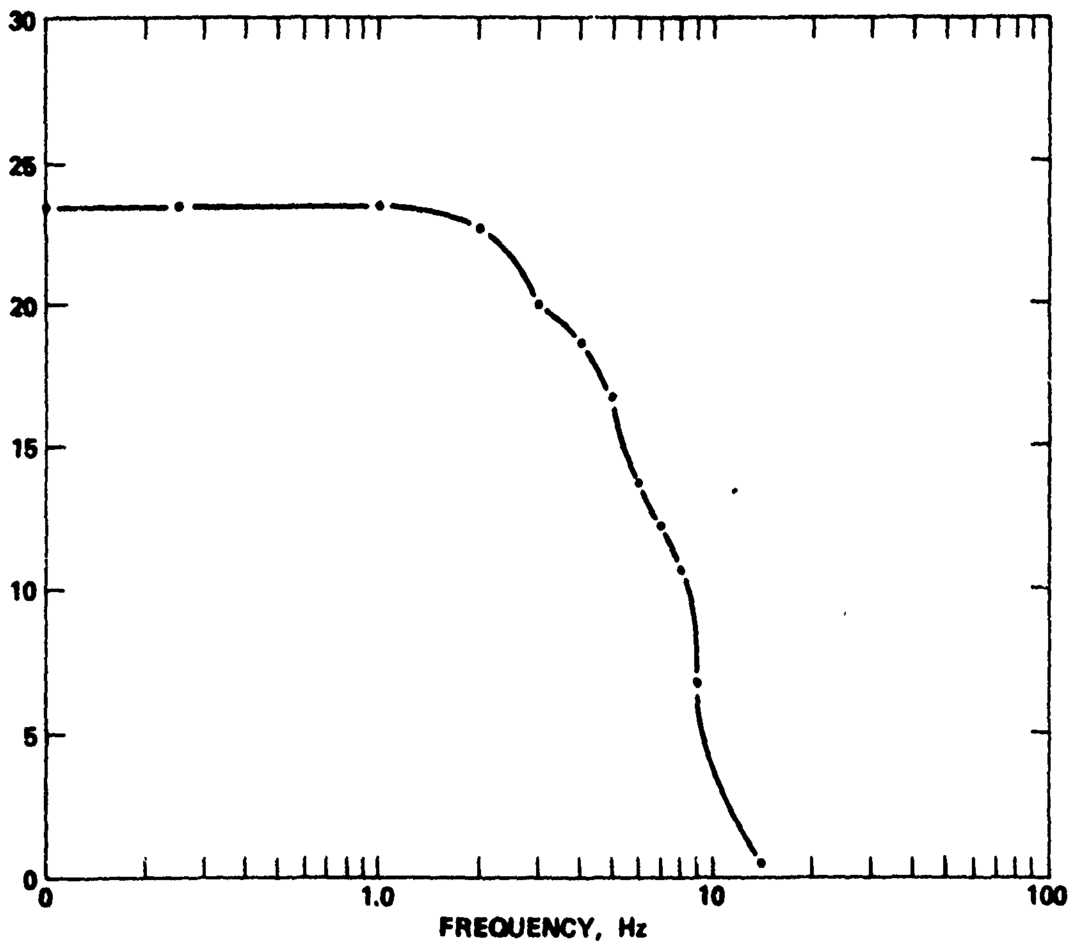


FIGURE 4. Digital Polarograph (Active Filter Response).

CURRENT COMP

D_5, R_{42}, R_{41}, and V_2 comprise a stage known as the current comp. When the initial e.m.f. appears across the cell, a transient occurs which is reminiscent of instantaneous current across a capacitor. In order to eliminate or greatly attenuate this unwanted portion of the signal, additional circuitry was added at this point. In essence, it is a biased shunt clipper and unwanted portions of the initial surge can be eliminated by simply adjusting the bias potential. The only error introduced by this stage is the drop across the diode due to its barrier potential. However, this is negligible compared to the signal at this point.

Y-AMP

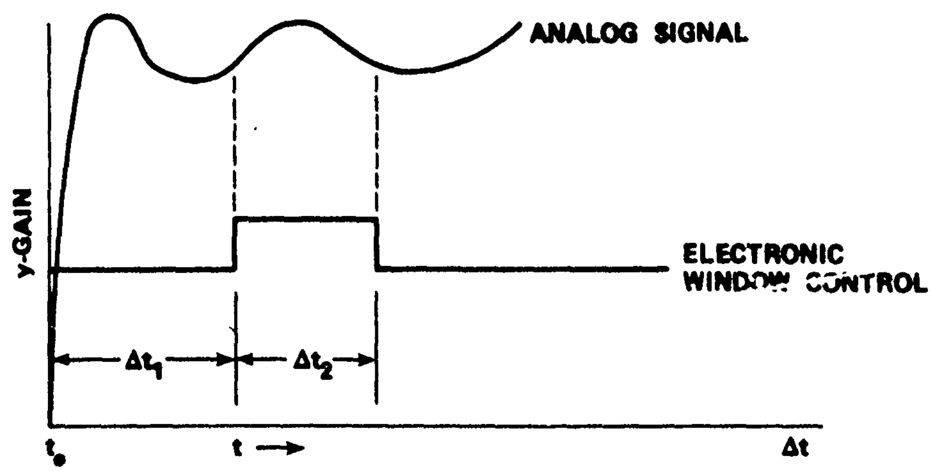
Reference is again made to both the block and schematic diagrams. The next portion of circuitry to condition the signal is the y-amp, composed of the second half of U_9 and its discreet components. The signal to this point has received some minor amplification from the current amp and the active filter. But when dealing with very small signals from the cell, additional amplification had to be employed. U_9 fills this requirement. Basically, it is an amplifier with the capability of adjustment of its reference point. By positioning R_{44} to some selected point, a change in the position of the signal out will be noted. The output of this stage is sent to two points: one is available to the outside for real time monitoring and the other is AC coupled to the peak detector. The output of this stage contains enough power to operate various pieces of peripheral equipment.

PEAK DETECTOR

This stage is connected to the output of the y-amp via the normally open contacts of RL_2. This relay contact must become closed before any portion of the analog signal can be acted upon by U_{10} and its related components. Assuming that the contacts have momentarily closed, the signal from the y-amp, which is AC coupled to the input of U_{10}, is impressed across C_{12}. This capacitor begins to charge, and when the contacts of RL_2 are opened, this peak charge remains stored in C_{12}. U_{10} is connected as a voltage follower, and by virtue of this high input impedance circuit, little or no charge can leak off of C_{12} through this path. RL_2 controls the portion of signal selected by this stage. The digital panel meter (DPM) will accept this peak reading only on a command from the decoder. All of the above action is in sync with the clock to assume the correct selection of the signal. D_6 and D_7 are placed into the circuit in a forward biased position and present a high reverse impedance to the signal stored in C_{12}, thus aiding the accuracy of the reading.

WINDOW CONTROL

Both halves of U_8 and the related component constitute a portion of the circuitry known as the electronic window. R_{49} and R_{50}, acting with C_{10}, control the time the window opens, and R_{51}, R_{52}, and C_{11} control the length of time the window remains open. This might be made more clear if reference is made to the timing diagrams (Figure 3). The window control receives its command from No. 6 waveform and this pulse is delayed and its width is controlled as shown in waveform No. 7. This spectrum of time occurs during the sampling period and is actually selecting a portion of the sample signal. When U_8 is adjusted to its maximum aperture, most of the signal is passed to the peak detector. When it is adjusted to a minimum, only a small increment, Δt_2 in the following diagram, is selected. This action is brought about by the control of RL_2.



COUNTER

Both halves of U_6 are connected as a divide-by-4 counter and the pulse actuating this circuit comes from the master clock. This counter can be substituted by some other division of sample time and this particular division was made as an example to demonstrate the capability of this circuitry, all of which was added to assist the operator in more accurate readings. The clock pulses controlling this counter are shown in waveform No. 3 of the timing diagrams (Figure 3). Pulses depicted in waveforms No. 4 and No. 5 are outputs from this counter.

DECODER

Waveforms No. 3 through 5 (Figure 3) are ended together in U_7 which together form what is known in this system as the decoder. This decoded output pulse is depicted in waveform No. 6 and is used as the control pulse for the electronic window. The same decoder output is used to control the display appearing on the DPM. A reset switch is connected within this circuit and actuation of this switch will reset the DPM.

DIGITAL PANEL METER

The DPM is an AR2000 automatic ranging, 3-1/2 digit, light emitting, diode display meter. This unit will display the reading in a binary coded decimal (BCD) output. Signals to this meter are received from the peak detector and will only change when a signal is received from the decoder. This gives the system the added capability of holding the last reading taken. The reading can be removed when the reset button is actuated. This is an added advantage to the operator since this time lapse of four readings before a change in meter reading gives him an opportunity to record or note the last reading and compare with the real time display on an X-Y recorder or oscilloscope.

CONCENTRATION SELECT

Thumbwheel switches SW_1, SW_2, and SW_3 are connected to produce a BCD code. When a base-10 number is dialed into one of these switches this number is used as a concentration level select and is electronically compared with the reading shown on the DPM. If the base-10 number selected on the switch is less than equal to the number displayed, an output is developed by digital comparators U_{11}, U_{12}, or U_{13}, and is sent as an electronic signal to the alarm circuit. There are three switches which represent (1) base-10 numbers 0 to 9, (2) numbers 10 to 90, and (3) numbers 100 to 900, thus making it possible to dial in or select a concentration level of 000 to 999, the upper limit of the 3-1/2 digit DPM. To use the concentration select system, a known concentration is first placed in the cell and the digital number displayed on the DPM noted. A number greater than this reading is dialed into the switches and if the concentration level exceeds this switch setting, an alarm system will be actuated. For added monitoring capability, the BCD number being compared is also brought out to an external monitoring point via a connector.

ALARM

U_{15} is connected to form an oscillator circuit and also possesses the capability of a driving portion to power a speaker output. When

U_{14} receives a signal from U_{13} and the oscillator, this signal is power amplified and the output turns the speaker on, producing a tone that will continue to sound until the reset switch is actuated. The speaker is mounted within the unit.

TIMING DIAGRAMS

Timing waveform No. 1 shows a number of pulses that represent the high output of the master clock. As shown in Figure 3, the timing sequence has been adjusted to a repetition rate of 5 sec off and a pulse on time of 2 sec. However, all successive timing is related to this master clock sequence and any changes at this point will appear to be circuitry-dependent upon this timing. Both the duty cycle and pulse width are variable.

Waveform No. 2 depicts a number of pulses developed by the sweep generator. In this particular case, they appear as a linear ramp but the slope of the ramp can be adjusted as well as the duty cycle and pulse width. This adjustment can be made by the operator and this pulse can be monitored at the x-output jack.

Waveform No. 3 is the low output of the master clock and any changes or adjustments to the clock appear at this point. The sweep generator is controlled by this pulse and a clock pulse is provided to the counter.

Waveforms No. 4 and 5 are pulses coming from the high outputs of the counter.

Waveform No. 6 is the resultant decoding pulse and generated from inputs of No. 4, 5, and 3. The function of this pulse is to control U_8, which in turn controls the width and position of a control pulse connected to RL_1.

Waveform No. 7 is represented as occupying some point in time and of some pulse width. However, both functions are variable on the No. 6 waveform pulse area. This pulse, in conjunction with RL_1, controls the window of the analog system.

PARTS LIST

Because of the unique design and flexible parameter, numerous components can be substituted throughout this electronic system and its operation would still be functional. Therefore, the parts list (Table 1) is descriptive only and represents one selection to a workable model. If changes are made, care should be exercised to monitor each result and its effect on the desired outputs.

TABLE 1. Parts List of Digital Polarograph.

No.	Part	Description	No.	Part	Description
1	U_1	9602	14	U_{14}	7410
2	U_2	9602	15	U_{15}	7404
3	U_3	747	16	Q_1	2N-2222
4	U_4	747	17	Q_2	2N-2222
5	U_5	740	18	Q_3	MJE 1103 (Motorola)
6	U_6	74107	19	Q_4	MJE 1093 (Motorola)
7	U_7	7410	20	D_1	IN-914
8	U_8	9602	21	D_2	12V, 1W, Zener
9	U_9	747	22	D_3	IN-914
10	U_{10}	740	23	D_4	IN-914
11	U_{11}	7485	24	D_5	IN-916
12	U_{12}	7485	25	D_6	IN-916
13	U_{13}	7485	26	D_7	IN-916

PERFORMANCE

Since the initial effort to construct this instrument stemmed from the research being sponsored at the Naval Weapons Center by the Naval Sea Systems Command, Code 0332, on "Applied Polarography for Analysis of Ordnance Materials," the performance test of the polarograph was made on part-per-billion concentration levels of the explosive 1,2-propyleneglycoldinitrate (PGDN) in effluent water. Part 1 of this report describes the use of this NWC-developed field polarograph in determining and monitoring explosives in aqueous media.

In order to test the digital polarograph output of digital reading with sample number, a test as shown in Figure 5 was made using a passive cell load. Note the excellent reproducibility of the data, well within polarographic limits.

Figure 6 shows typical single-sweep polarograms for a 0.20-ppm concentration of PGDN in effluent water produced by the field polarograph and drawn out by a Moseley 2D X-Y recorder on 25.4 by 25.4 to 1.27 cm (10 by 10 to 1/2 in.) graph paper. Note the excellent definity and reproducibility of these polarograms. The digital count, with a window setting in the instrument of 100 mV, is shown by each polarogram. The digital number for each represents an average current recorded by the polarograph from four successive drops of mercury from a DME with a drop time of 7 sec and m = 7 mg per drop. The variation in digital numbers and X-Y recorded polarograms, respectively, represent an error well within the limits of single-sweep polarography, which is ± 3 to 5% of the true concentration of 0.20 ppm or about 10 parts-per-billion.

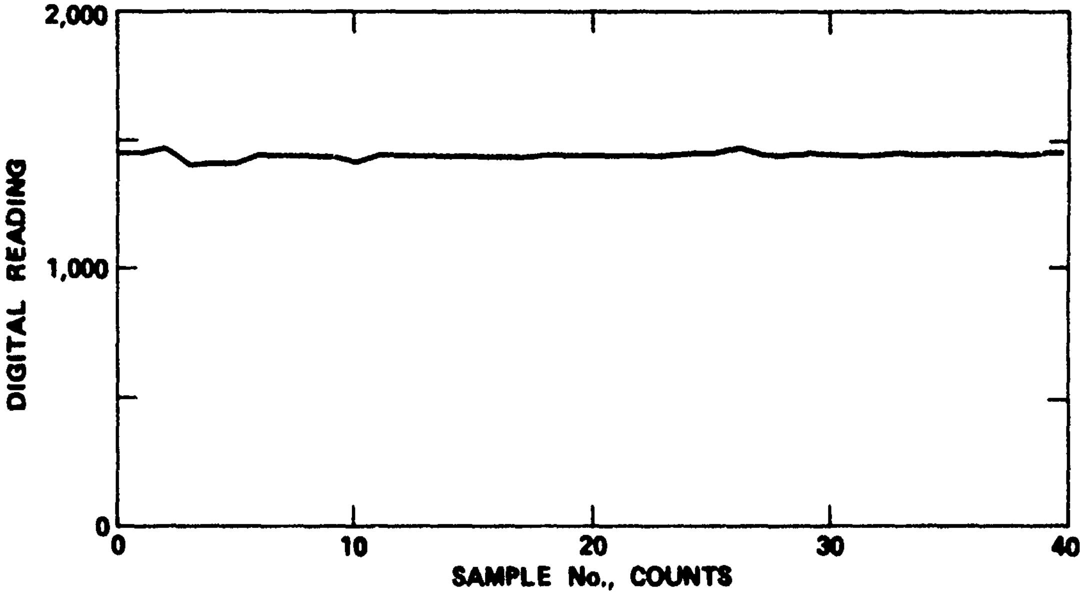


FIGURE 5. Digital Polarograph (Passive Cell Load Test).

Table 2 shows data obtained for low level concentrations of pure PGDN added to an effluent water containing no measurable PGDN to start. Note the sensitivity obtained with the digital count, which is about 100 for a PGDN concentration change of 20 parts-per-billion.

TABLE 2. 1,2-Propyleneglycoldinitrate Added to Effluent Water.

Start potential of polarograph set at -0.54 volts vs. Hg pool.

PGDN added, ppm	X-Y Recorder,[a] graph paper divisions	Digital count,[b] cell current 11
0.19	8.0	1.205
0.21	9.0	1.312
0.24	10.0	1.407
0.29	12.0	1.628

[a] Average of six readings.
[b] x = 1, y = 2.

A check of the reproducibility of the digital count output of the polarograph on a sample of PGDN (0.20 ppm) over a 5-min period in time showed that the digital count is reproducible over this time period to within 1 to 2%.

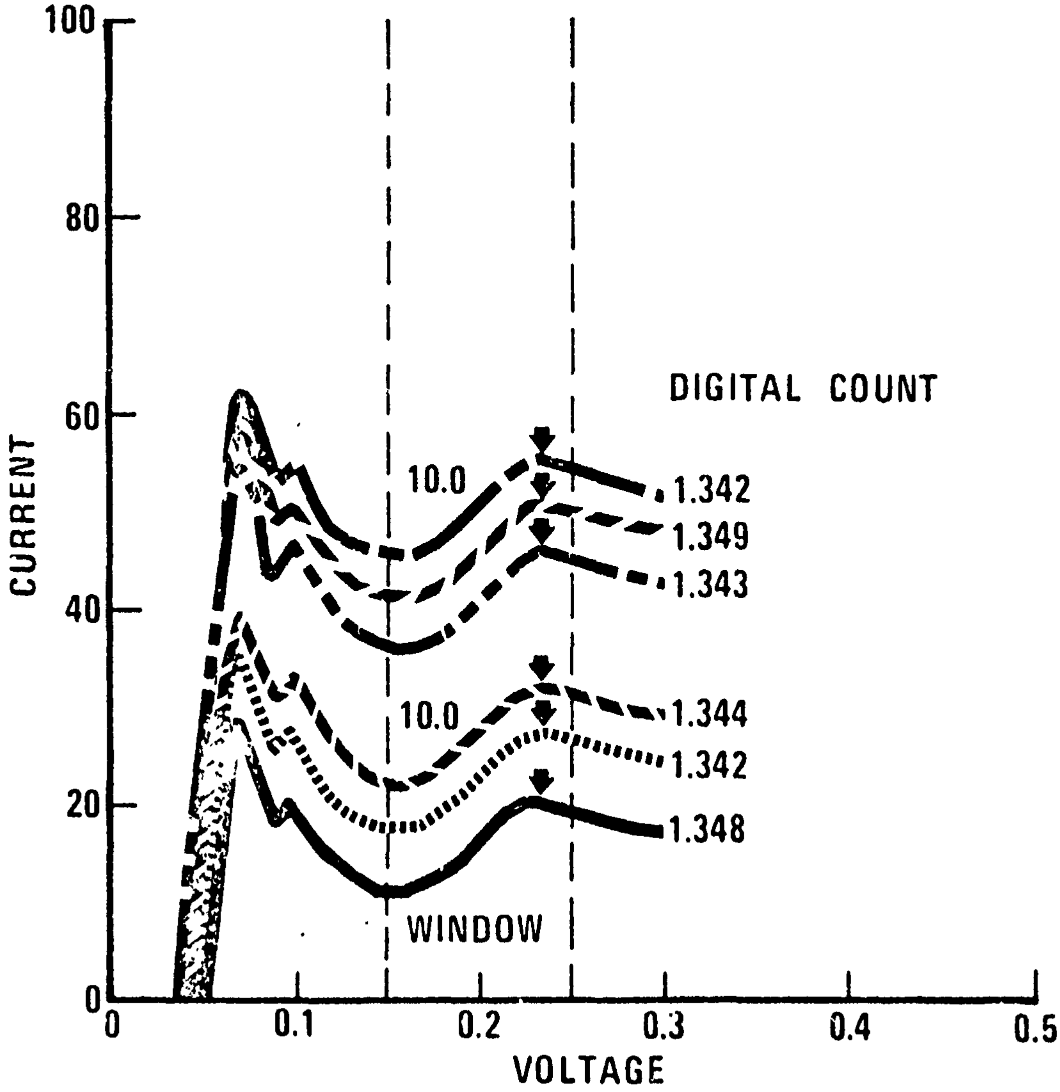


FIGURE 6. Single-Sweep Polarograms Obtained With Solid-State Field Polarograph (0.20 ppm PGDN in Effluent Water). Start potential = 0.54 V; cell current = 11 V; window set for 100 mV (-0.69 to -0.75 V); i_p = -0.775 V vs. Hg.

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