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Studies on the Controlled Potential Electrolysis. (VIII)

An Improved Potentiostat

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A new sensitive and stable potentiostat for the purpose of the controlled potential electrolysis was constructed.

The D.C. source supplies to the cell, 5 amperes and 30 volts at the maximum and the sensitivity of the potentiostat to the change in the potential of the working electrode is about ± 2 millivolts.

INTRODUCTION

Previously, the authors reviewed the instrumentation of the controlled potential electrolysis and also reported on the new potentiostat provided with galvanometer-switch¹⁾²⁾³⁾.

In this paper, a new sensitive and stable potentiostat which consists of the converter and amplifier to operate the reversible motor for controlling the electrolytic current is reported. It is completely line operated from 110 volt alternating current source except one 1.5 volt dry battery for reference potential. The sensitivity of this potentiostat is about ± 2 millivolts to the change in electrode potential. The instrument consists of two parts, one the direct current supply for electrolysis, and the other the potentiostat for controlling the potential of the working electrode; the former can be used independently of the latter in the case of ordinary electrolysis at constant applied voltage.

APPARATUS

Direct Current Power Supply. As shown in Figure 1, power is drawn from 110 volt, 60 cycle line and goes through the variac transformer T_1 , UZ terminal (which is short-circuited in the ordinary electrolysis or is connected to plug P_2 of the potentiostat in the case of controlled potential electrolysis), step down transformer T_2 (110 : 30), selenium full wave rectifier Rec., LC filter, electrolytic current meter A, applied voltage meter V_1 and finally to the electrolysis cell. In parallel with this circuit, a stirring motor and a heater with their control resistance R and a transformer T_3 are equipped separately.

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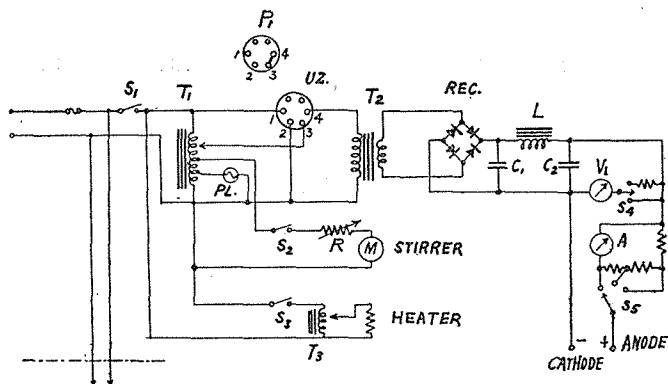


Fig 1. Direct current power supply.

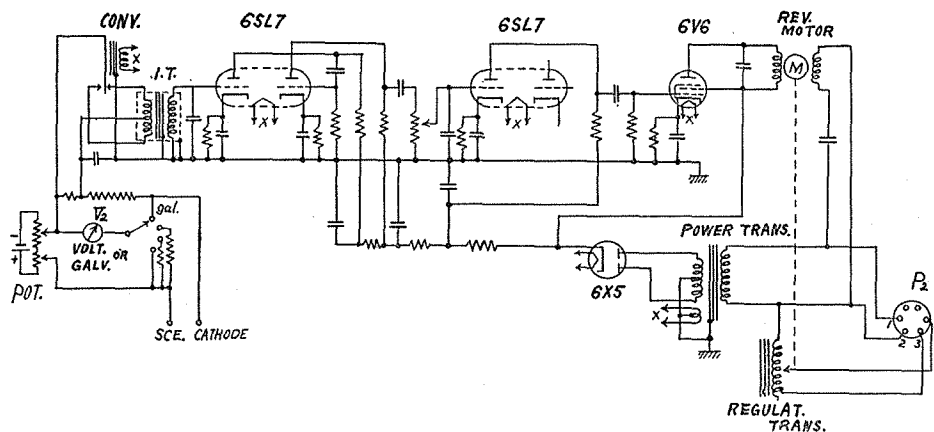


Fig. 2. Potentiostat.

Regulator (Potentiostat). When regulating plug P₂ is inserted to the UZ terminal on the direct current supply, the regulating variac transformer REGULAT. TRANS. (110 : 130) works to vary the applied voltage as dictated by the control circuit, thereby holding a constant solution-cathode (or solution-anode, if necessary) potential in the cell. It is driven by a balancing motor M through a gear reduction and friction clutch. The clutch permits a quick setting to be made by hand at any time required (especially at the start of electrolysis). The variac T₁ in the power supply acts as the range control transformer to set the upper limit of direct current voltage. It is advisable to set this a little higher than the voltage expected during the electrolysis. The meters A and V₁ read electrolysis current and voltage respectively. A range switch with multiplier resistors provides ranges of 10 and 30 volts for V₁ and ranges of 0.1, 1.0, and 5.0 amperes for A.

The heart of the regulating system is a usual converter and A.C. amplifier. Voltage is supplied by the reference electrode and the cathode (or anode) and this voltage is balanced against a reference voltage set up on potentiometer POT. whose voltage is read on the meter V₂ (full scale ranges of 1.0 and 3.0 volts). When

Studies on the Controlled Potential Electrolysis. (VIII)

the cathode-reference electrode potential differs from the opposing reference voltage (the difference can be observed also on the same meter V_2 by switching it to "gal"), the unbalanced D.C. is converted to A.C. and the amplified A.C. causes the balancing motor to operate the regulating variac in the appropriate direction until the potential of the cathode returns to the value of the reference voltage.

OPERATING PROCEDURE

As is mentioned above, when the ordinary electrolysis at constant applied voltage is performed, the lead to the regulator is disconnected and the short-circuiting plug P_1 is inserted to it. The applied voltage or the electrolysis current is regulated by the variac transformer T_1 manually.

When controlled potential electrolysis is examined, the plug P_1 is drawn out, the plug P_2 of the regulator lead is inserted to the UZ plug, the cell with the cathode, the anode and the reference electrode is connected to the instrument in the usual way the range control variac T_1 is set a little higher than the maximum applied voltage expected during the electrolysis and the motor driven variac REGULAT. TRANS. is set at zero volt. The desired reference potential is established on V_2 . Then, subsequent operation is completely automatic.

EXPERIMENTAL

The ripple voltage due to A.C. source was measured by cathode-ray oscillograph and the result is shown in Table 1.

Table 1. Oscillographic observation of ripple voltage.

Applied Volt.	Current	Ripple Volt.	Ripple of Cathode	
20.0 V	5.0 A	430 mV		Coil resistance, instead of solution
	3.0	300		
10.0	2.0	180		
5.0	1.0	60		
1.0	0.1	20		
0.9	0.13	17.6	3.5 mV	Electrolysis of acidic copper solution
0.8	0.08	8.5	2.1	
0.7	0.036	3.5	2.8	
0.5	0.007	3.5	1.4	

It was observed that the ripple voltage of cathode-reference electrode was much less than the applied voltage and was below 5 millivolts, but the stirring of the electrolytic solution caused a large fluctuation on the cathodereference electrode voltage and the instant deviation often exceeded ± 50 millivolts.

The deviation of cathode-reference electrode voltage from the reference voltage

is shown on the galvanometer V_2 . The observed deviation was about ± 5 millivolts when the reference electrode of resistance less than 1 kilo ohm was used.

Generally, these experimental results showed that the instrument--power supply and the potentiostat--was satisfactory usable for any electrolysis, especially at controlled cathode potential. The result of electrolysis obtained with this instrument will be reported later.

ACKNOWLEDGMENT

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