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Informe Científico – Tecnológico del IPEN (Instituto Peruano de Energia Nuclear),
1998 – 2001. Lima, Peru

http://ipen.gob.pe/site/publicaciones/infcyt9801/ict9801_PAG_105_107.pdf

Quotation of:

Banica, Florinel Gabriel. A Simple Hanging Mercury Drop Electrode J. Chem. Educ. 2000
(77) 98. (Ref. No. 1)

A LOW COST PC- BASED VOLTAMMETRIC ANALYZER FOR DETERMINATION OF CADMIUM, COPPER, LEAD, NICKEL, ZINC AND OTHER TRACE ELEMENTS IN WATER

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INTRODUCTION

Anodic-stripping voltammetry, like as other electro-analytical techniques of chemical analysis can be very valuable as complements for neutron activation analysis (NAA), for rapid, accurate and simultaneous determination of some trace elements in water and in digested biological samples at the ppb($\mu\text{g/L}$) range. Commercial voltammetric analyzers may have a cost in the range of US\$ 20,000.00 and the limited budget of our NAA laboratory not allow us to buy this kind of complementary instrumentation. In this paper we describe a PC-based voltammetric analyzer, designed and built in our laboratories at a relatively low cost.

EXPERIMENTAL PART

The schematic diagram of the electronic part of the instrument is shown in figure 1. Power supply connections of the operational amplifiers has been omitted for sake of simplicity. All used components were of commercial grade, being its values as follows:

OA1, OA2, OA3, OA4:	OP_07
R1, R2, R3:	15 k Ω (1%)
R4:	1k Ω
R5,R6, R7:	5k Ω (1%)
R8,R9:	2k Ω
R10:	1 M Ω (1%)
P1:	2k Ω (10 turns)
Z1, Z2:	5.1 volts Zener diodes
Z3:	2.5 volts Zener diode
C1:	20 n farads, ceramic

The reference electrode is a simple saturated calomel electrode of commercial type and the auxiliary electrode a short piece of platinum wire. The working electrode is a home made one, being similar to the described by BANICA (1). The hardware used for acquisition and control purposes was the low cost (US\$ 350.00) data acquisition board Advantech PCL711B with 8 single ended analogical inputs of 12 bits resolution, 16 digital inputs, 16 digital outputs and one analogical output of 12 bits resolution. The home made working SMDE (see figure 3) is automatic and its functions (drop formation,

drop dislodge and capillary purge) are handled by the ADVANTECH PCL711B card, by means of solenoids driven by an auxiliary relay card under control of the PCL711B. The implemented prototype of voltammetric analyzer takes profit of the virtual instrumentation concepts, being the software interface elaborated in LAB WINDOWS CVI (National Instruments). The potential applied to the summing point of potentiostat (OA1), the current follower (OA3) response and sampling time schedule are shown in figure 2. The waveform and potential ramp of the excitation signal are produced by software and taken directly from the analogical output of the PCL711B card. Due the fact of that this analogical output produces voltage values between 0 and +5 volts, it was necessary to use the voltage inersor OA3 and the Zener diode Z3 (see figure 1) to be able of feed the summing point of OA1 with a range of voltages between -2.5 and +2.5 volts. Before the connection of the electrochemical cell the circuit and software were tested with a dummy cell as described by NYHOLM and WIKMARK (2).

RESULTS AND DISCUSSION

The figure 4 shows the current follower response to a single square wave impulse applied to the summing point of the potentiostat, using the electrochemical cell with a supporting electrolyte 0.1 M NH₄Cl - NH₃ (pH 10); with sodium sulfite to eliminate dissolved oxygen. The vertical axis of figure 4 is in LSB units, each one of that represents 0.25 nA of current. Figure 5 shows a voltammogram of a synthetic sample with 4 ppb of cadmium. Each point in the voltammogram is the diference between two single sampled current follower responses for two consecutive even and odd sampling intervals, respectively (see figure 2). In this way we avoided the need of use sample & hold circuits as is usually done in this kind of instrumentation (3). The vertical axis in figure 5 is also in LSB units each one of that represents 0.153 nA of current. Finally, figures 5 and 6 shows that the prototype achieves good sensitivity and linear response for trace analysis of cadmium in water samples.

REFERENCES

1) Banica, Florinel Gabriel. A Simple Hanging Mercury Drop Electrode J. Chem. Educ. 2000 77 98.

2) N. Nyholm & G. Wikmark, Analytical Chemistry, 58(9) 1959-1964, 1986.
 3) A. J. Bard & L. R. Faulkner, 1980, "Electrochemical Methods, Fundamentals and Applications", John Wiley & Sons, New York, ISBN 0-471-05542-5.

Figure 1: Schematic of the voltammetric analyzer circuit

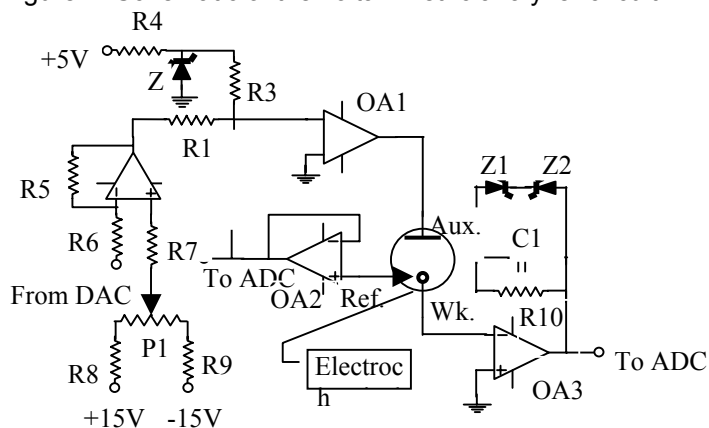


Figure 2: Potential programming, current follower response and timing schedule.

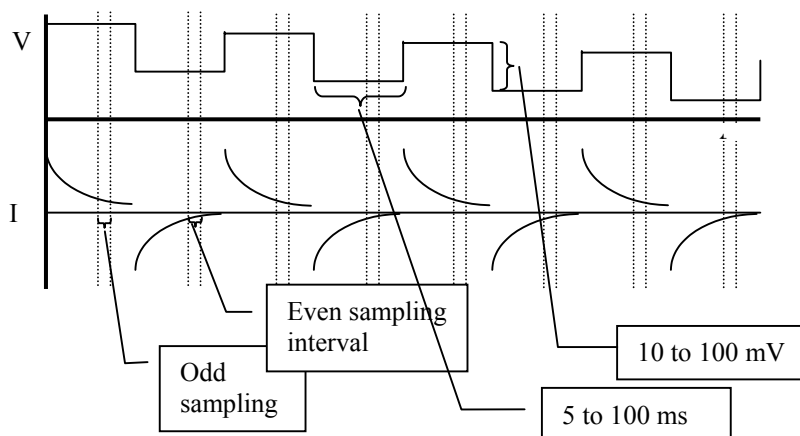


Figure 3: schematic of the home made static mercury drop electrode (SMDE)

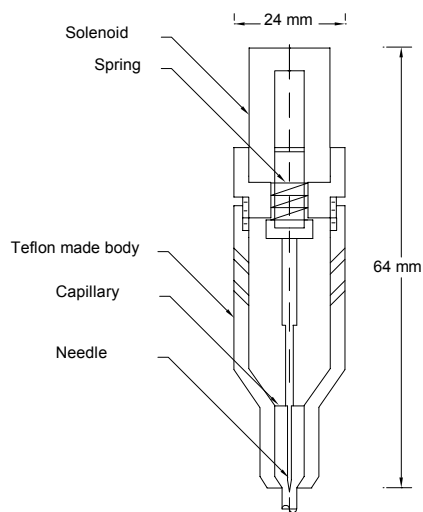


Figure 4: Current follower response to a 25 mV square wave stimulus, centered at -300 mV vs saturated calomel reference electrode, supporting electrolyte: 0.1 M $\text{NH}_4\text{Cl} - \text{NH}_3$ (pH 10).

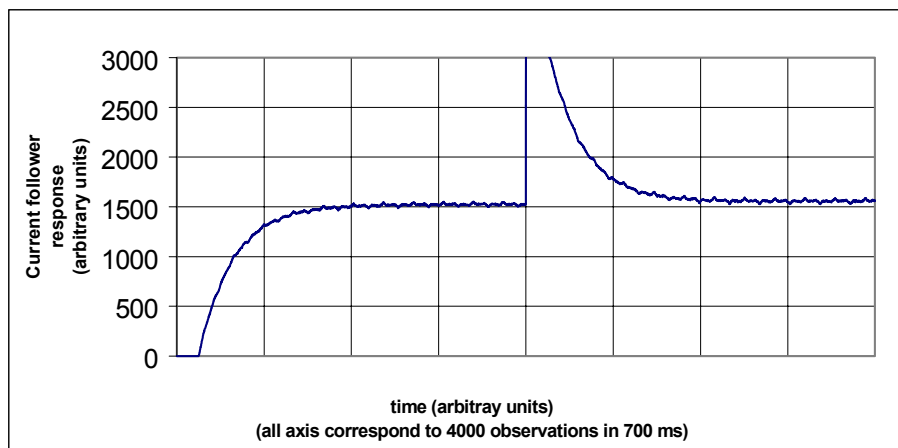


Figure 5: Synthetic sample of water with 4.0 ppb of $\text{Cd}(\text{II})$, being the supporting electrolyte 0.1 M $\text{NH}_4\text{Cl} - \text{NH}_3$ (pH 10); with sodium sulfite to eliminate dissolved oxygen.

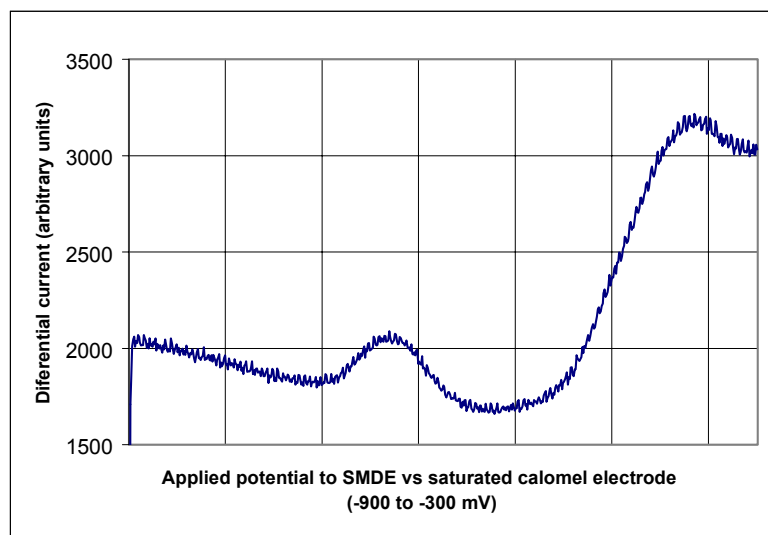


Figure 6: Peak height versus cadmium concentration.

