Journal of Chromatography, 91 (1974) 819–822

CHROM. 7261

ISOTACHOPHORESIS

CONDUCTIVITY MEASUREMENT AND SIGNAL HANDLING

A. J. MULDER
Department of Instrumental Analysis, University of Technology, Eindhoven (The Netherlands)
and
J. ZUSKA
Department of Physical Chemistry, Charles University, Prague (Czechoslovakia)

SUMMARY

Electronic circuits for measuring the conductivity in isotachophoresis are described. The arrangement for recording the signal is discussed and a controller for the electronic clock used in the automatic device is considered.

INTRODUCTION

A general method of detection in isotachophoresis is the use of a conductometer. Although many reactions may occur on the sensing electrodes, which are in direct contact with the electrolyte, the detector has a high resolving power. By coating the electrodes, the conductometric method of detection can achieve wider applicability, as discussed by Everaerts and Rommers[1]. The main problem in using this type of detector is that very small time intervals occur between the peaks on isotachopherograms so that for quantitative work the traces on the recorder paper are of little use.

In this paper, an electronic arrangement is described that can be used for measurements of the conductivity in isotachophoresis. The chemical reaction on the electrodes, however, must be suppressed, in order to diminish the changes in capacity between the electrode surface and the electrolyte of which the conductivity has to be measured. A scheme for the automatic unit for measuring the times of passage of the successive zone boundaries is given.

EXPERIMENTAL

From the conductometer cell the impedance is always a function of the composition of the liquid (electrolyte) between the two measuring electrodes. The impedance can be measured by either an a.c. or a d.c. method. In the d.c. method, the potential difference between the measuring electrodes, as a result of the driving current, is

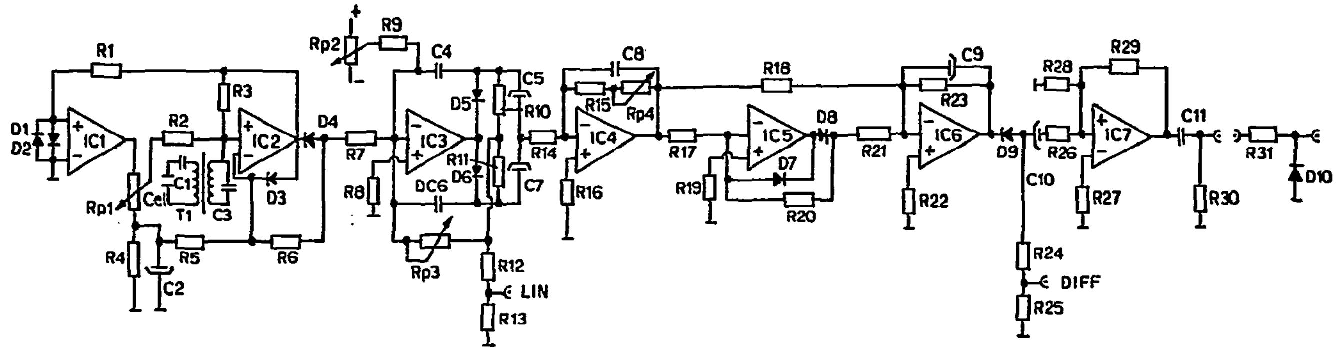

Fig. 1. Electronic circuit for measuring the conductivity in isotachophoresis. The construction of the cell was discussed elsewhere[2]. The components used are as follows:

R1, 5, 15, 17, 18, 20, 23	= 10 k
R2	= 470 k
R3	= 2M2
R4	= 1k2
R6	= 15k
R7, 8	= 3k9
R9	= 39k
R10, 11	= 4k7
R12, 14, 16, 27, 28	= 470Ω
R13	= 56Ω
R19	= 4k7
R21	= 5k1
R22	= 3k9
R24	= 12k
R25	= 100
R26	= 0
R29	= 1M5
R30, 31	= 1k
T_1	= $n_1/n_2 = 4$
n_1	= for frequency *ca.* 1 kHz

C1	= 220 nF, 400 V
C2	= 330 μF, 3 V
C3	= frequency *ca.* 1 kHz
C4, 6	= 220 nF
C5, 7	= 640 μF, 40 V
C8, 11	= 680 nF
C9	= 10 μF
C10	= 47 μF, 63 V
IC1	= 709 μA
IC2, 3, 4, 5, 6, 7	= 741 μA
D1, 2, 4, 8	= 1N914
D3, 5, 6, 7, 9, 10	= 0A202
Rp1	= 1k
Rp2	= 20k, ten turns
Rp3	= 50k
Rp4	= 100k, ten turns

measured. In the a.c. method, an external a.c. signal is applied in order to measure the impedance. The means chosen to carry out this measurement is as follows:

In an oscillator (Fig. 1, IC1 and IC2) an LC network, damped by this cell impedance, is applied as the frequency-setting element. A tuned transformer gives a good galvanic isolation between the circuit and the measuring electrodes, which are at a high potential due to the driving current. The signal finally obtained is detected (D3 and D4) and amplified (IC3). The linear signal sensitivity is smoothed by Rp2 and this signal is fed into a recorder via point LIN.

The information on the rise time and the real time measurement is obtained from the first differential of the linear signal. The circuitry around IC4 forms an active differentiator. In this circuit, C5, C7 and R14 are the differentiating elements.

Because negative peaks are sometimes obtained in the isotachopherograms, this information may not be missed as a real zone boundary[1]. In order always to obtain electronically negative going pulses, an absolute value amplifier is used (IC5 and IC6). The high frequency signals are reduced by C4, C6, C8 and C9. The signal-to-noise ratio is optimized by using the diode D9. The differential signal is smoothed by Rp4 and this signal is fed into the recorder via point DIFF.

In order always to obtain a good comparable time measurement between two differential pulses, a signal was required that could start and stop an electronic clock exactly at the time when the pulse is at its maximum value (Fig. 2). In order to isolate

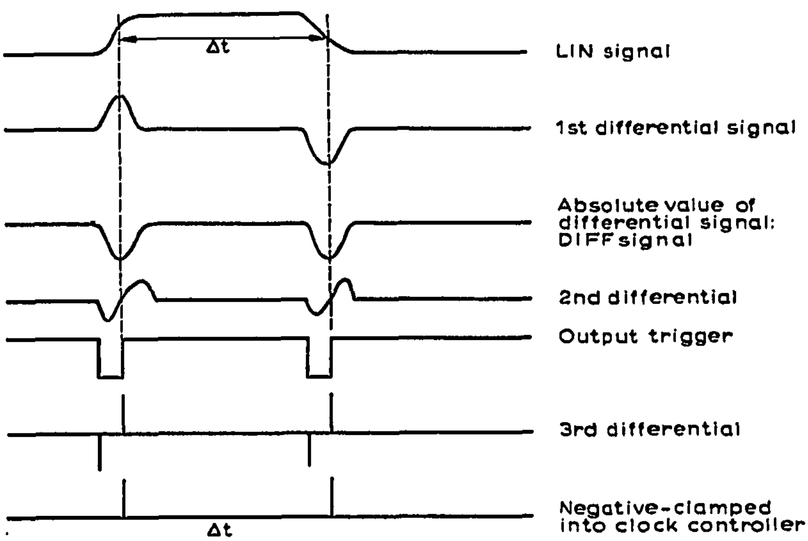

Fig. 2. Signal handling of the automatic device for measuring peak-to-peak times during isotachophoretic analyses.

the desired signal, the pulses are differentiated (C10, R26) and then led into a trigger (IC7) with a small hysteresis. The signal obtained is again differentiated (C11, R30) and negative-clamped by a diode (D10). The resulting positive pulse is very useful for automating an electronic clock.

For the automatic recording, there are three important components. Firstly, an electronic counter (common type) with a display and a latch register is needed. Secondly, a printer, *e.g.*, a Sodeco PN 107, is needed in order to print the time measured. The printer used in our experiments requires a maximum of 1.3 sec in order to print out the time. During this time period, the electronics associated with the printer act as a memory. Thirdly, a controller must be incorporated.

With this circuitry, in addition to the normal time measurements, the latch register from the clock can be used as a kind of buffer memory, which is sometimes needed because the printing time of the printer is rather long (Fig. 3). This controller can be built with several TTL ICs. In our equipment we used two SN 7474 components, some gates and some RCs as a pulse delay.

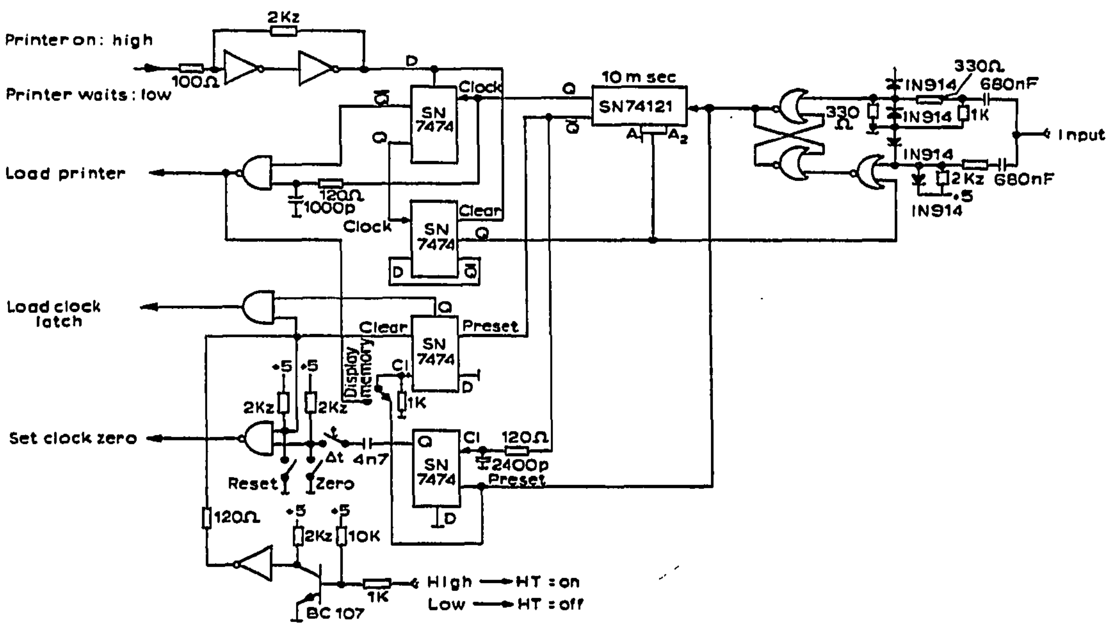

Fig. 3. Controller for the electronic clock used in the automatic device.

REFERENCES

1 F. M. Everaerts and P. J. Rommers, *J. Chromatogr.*, 91 (1974) 809.
2 F. M. Everaerts and Th. P. E. M. Verheggen, *J. Chromatogr.*, 73 (1972) 193.