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## A new heterodyne-beat circuit for the determination of static permittivity

Teodosia Arauz Castex, Pedro Díaz de Vivar, María B. Rebollo Paz, and Adrián H. Buep  
 Departamento de Física, Facultad de Ciencias Exactas y Naturales, Universidad de Buenos Aires,  
 Pabellón I, Ciudad Universitaria, 1428 Buenos Aires, Argentina

(Received 22 September 1994; accepted for publication 7 December 1994)

A greatly improved heterodyne-beat oscillator was built using integrated circuits, to measure static permittivity of liquid samples. Frequency variation of the oscillator is of less than 0.7 Hz/h; that corresponds to a capacity variation of less than 0.005 pF/h. Permittivity can be measured with a precision of 0.003% and an accuracy better than 0.01%. © 1995 American Institute of Physics.

The instruments commonly used to measure static permittivity of liquid samples, when dielectric loss is negligible, are based in general on the heterodyne-beat method.<sup>1</sup> Devices for high-precision capacity measurements, first using a valve heterodyne-beat oscillator and later a solid-state circuit, were developed in our lab over a period of 20 years.<sup>2</sup> At present we use a new design heterodyne-beat oscillator built

with integrated circuits and developed with particular care to obtain a very stable clean LC resonance circuit. Figure 1 shows the circuit in detail and its connections to the cell and the oscilloscope. In Table I are listed the values of the elements used in the new circuit.

The fixed oscillator has a 1.6 MHz quartz crystal and a CD4060B integrated circuit to give a fixed frequency of 100

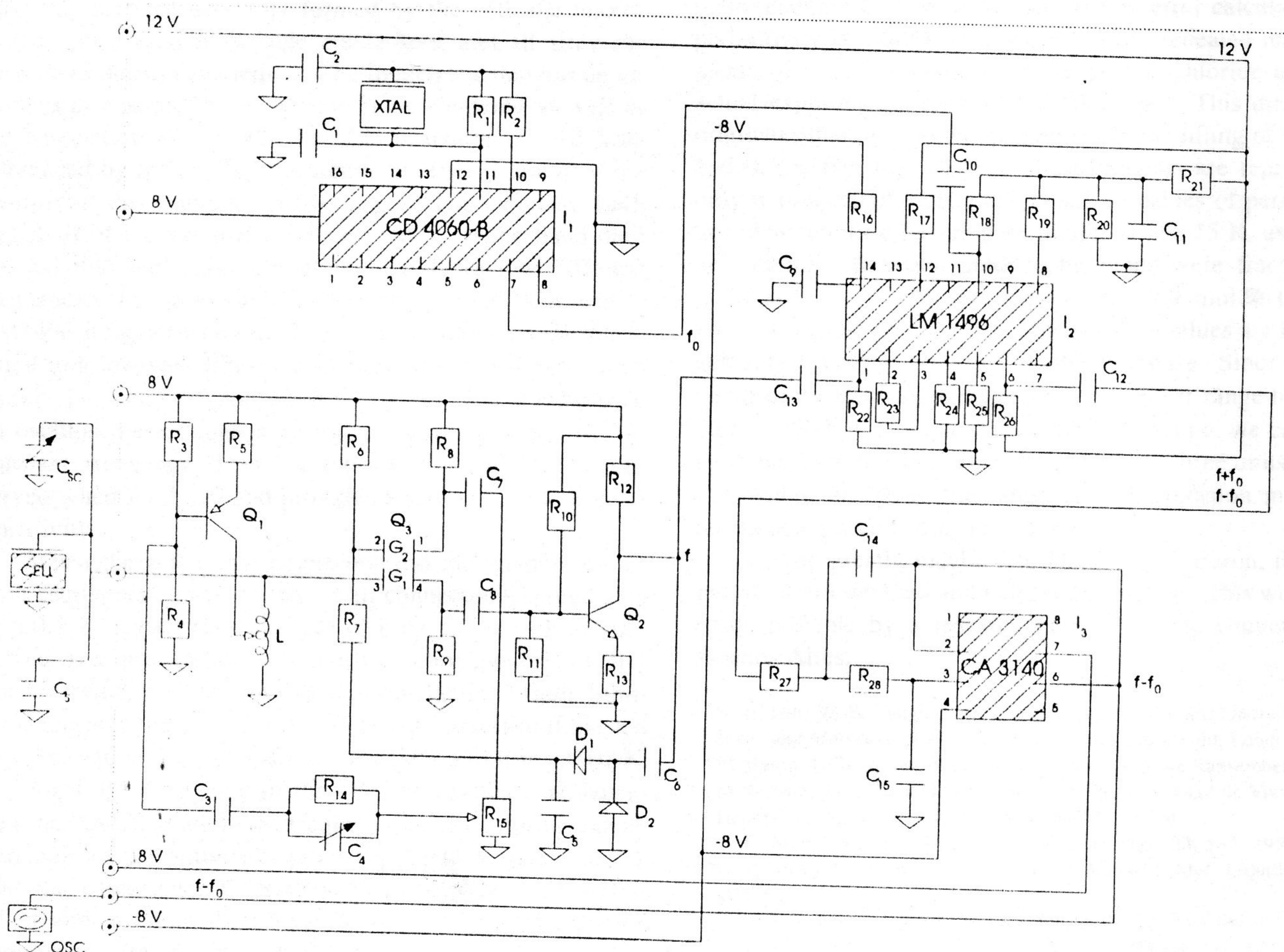


FIG. 1. Circuit diagram of the new heterodyne-beat device.

TABLE I. Values of the circuit components.

No.	$R(k\Omega)$	No.	$R(k\Omega)$	No.	$C(\mu F)$
1	1000	16	0.047	1	0.000015
2	10	17	0.68	2	0.000015
3	10	18	0.68	3	0.1
4	39	19	0.68	4	0.000015
5	3.3	20	1	5	0.5
6	22	21	1	6	0.1
7	10	22	1.2	7	0.1
8	0.47	23	1	8	0.5
9	0.51	24	1.2	9	0.47
10	82	25	6.8	10	0.08
11	22	26	0.68	11	0.1
12	2.2	27	100	12	10
13	1	28	100	13	0.05
14	1			14	0.01
15	2.2			15	0.01

$Q_1$ : PNP silicon transistor BC558  
 $Q_2$ : NPN silicon transistor BC548  
 $Q_3$ : dual-gate MOSFET MFE201

$D_1$ : rf diode BAY45  
 $D_2$ : rf diode BAY45  
 $L$ : 2.81 mH

$C_{sc}$ : assembly formed by a 1422-N precision capacitor (General Radio Company) and a noncommercial micrometric condenser (Refs. 2 and 3). (An example of a set of reading values of  $C_{sc}$  for one determination of  $C$ , using  $CCl_4$  as standard liquid,  $\epsilon=2.2279$  at  $T=298.15$  K, is  $C_{sc}^i=788.500$ ,  $C_{sc}^o=608.720$ ,  $C_{sc}^\epsilon=394.807$ , and  $C_{sc}^f=788.500$  pF.)

kHz. The variable oscillator is an LC resonator in which the capacitance is the assembly formed by the cell, the micrometric and precision variable condensers, and all stray capacitances due to connections. The inductance that has an air nucleus is separated from the remaining elements, as well as the connection to the cell. The LC resonator is then least influenced by the rest of the circuit. In order to improve the stability of this oscillator, a two gate FET is included. Both signals ( $f$  of the variable oscillator and  $f_0$  of the fixed one) are fed into the mixer circuit that produces two different frequencies  $f-f_0$  and  $f+f_0$ . This mixer was built with a LM1496 integrated circuit. The  $f+f_0$  frequency was eliminated in a low-pass filter that includes a CA3140 integrated circuit. The output signal, when  $f-f_0=100$  Hz, is viewed on an oscilloscope through a Lissajous figure against a 50 Hz constant frequency. This 2:1 ratio avoids the lock-in observed when  $f-f_0=0$  and provides a substantial increase in sensitivity.

When the cell is not connected and the temperature of both oscillators as well as that of all connections is regulated at  $\pm 0.5$  K, capacitance changes of only  $\pm 0.005$  pF are observed over periods of up to 1 h that corresponds to a frequency variation of the oscillator of less than 0.7 Hz/h. Similar changes in the precision condenser are observed if the cell is connected and the temperature regulation in it is  $\pm 0.01$  K.

Since the frequency of the variable oscillator is dependent on the LC relation, the frequency stability during a measurement of permittivity requires that both the inductance  $L$  and the capacitance  $C$  remain constant. When the cell is filled with a liquid there is a change in the total capacity; therefore to return to the initial  $C$  value, the standard variable capacitor must be adjusted to another value of capacity. So the permittivity is calculated from four values of the standard variable capacitor:

TABLE II. Measured permittivities for pure liquid samples at 298.15 K.

Compound	Experimental	Literature	References
Cyclohexane	2.0150	2.0150	4
		2.0151	5
Benzene	2.2727	2.2725	6
		2.2727	7
		2.2730	8
Toluene	2.3759	2.3757	7
		2.3760	9
Ethylbenzene	2.3919	2.3920	4

$$\epsilon = \frac{C_f}{C_e} = \frac{C_{sc}^f - C_{sc}^\epsilon - C_s}{C_{sc}^i - C_{sc}^o - C_s},$$

where  $C_f$  is the cell capacitance filled with dielectric,  $C_e$  the capacitance of the empty cell (190 pF),  $C_{sc}^i$  and  $C_{sc}^f$  the initial and final standard condenser values when measured with cell not connected,  $C_{sc}^o$  and  $C_{sc}^\epsilon$  the standard condenser values measured, respectively, with the cell empty and filled with sample, and  $C_s$  the stray capacitance.

The value of  $C_s$  was determined using carbon tetrachloride ( $\epsilon=2.2279$ ) as the permittivity standard. If the connections to the cell are inside a thermally equilibrated box with  $\pm 0.5$  K of variation in temperature, the difference between the maximum and minimum measured values of  $C_s$  is 0.006 pF. Using a standard variable capacitor with a minimum scale reading of 0.001 pF, the theoretical error calculated for permittivity is  $\pm 0.00008$  units. However, repeated measurements of identical samples of carbon tetrachloride gave an actual experimental error of  $\pm 0.0002$  units. This difference originates mainly in small differences in the filling of the cell and in the fact that capacity measurements are reproduced only within  $\pm 0.003$  pF. Table II shows values of permittivities obtained for different pure liquids at 298.15 K, using the new circuit. (Reaction grade compounds were fractionally distilled twice. Purity was better than 99.9 mol % through GPC.) It can be seen that the experimental values are in good agreement with those cited in the literature. Since in our equipment the standard capacitor has a linear range between 200 and 900 pF and our cell capacity is 190 pF, we can only measure permittivity of liquids of less than three units. But it is possible to extend the range of high-precision measurements using a cell of lower capacity.

We are greatly indebted to Dr. Máximo Barón, this Department, for detailed and helpful discussions. This work was made possible by a research grant from the University of Buenos Aires.

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