Pyrolysis system to obtain carbonaceous material from the rice husk precursor

Javier R. Castro-Ladino[1]*, Ana B. Vacca-Casanova[1], Carlos A. Cuy-Hoyos[1]

[1] Escuela de Ingeniería, Universidad de los Llanos, Villavicencio, Colombia

Supplementary document

Temperature in the muffle and in the gas outlet duct.

Due to the characteristics and technical specifications of the pyrolysis system, the K type thermocouple is the appropriate temperature sensor for the measurement of this parameter inside the muffle or reactor (TE-01, figure 1S), where the carbonization of the material will be carried out; as well as in the gas discharge pipe (TE-02, figure 1S). This type of thermocouple measures temperatures up to approximately 1.300℃, and is typically used in foundry systems and furnaces. As output, it delivers a voltage signal in the order of millivolts.

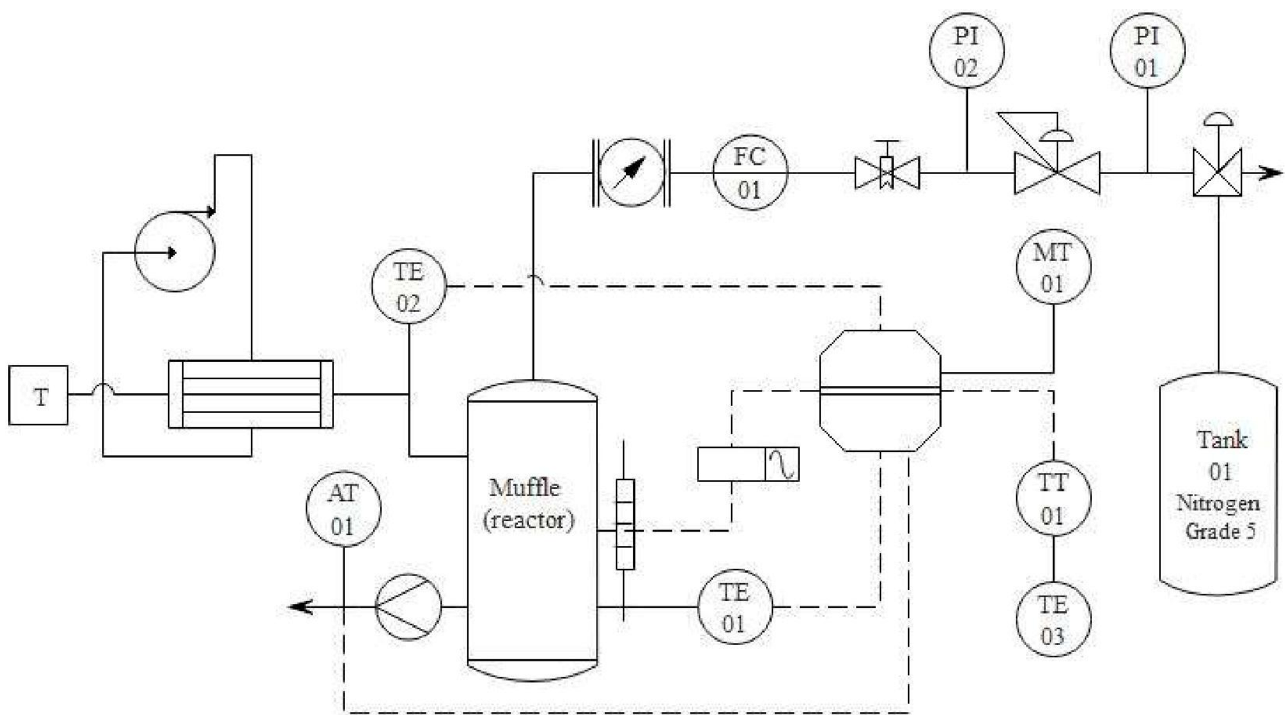

Figure 1S. P&D diagram pyrolysis system

* Correspondence author: jrcastrol@unillanos.edu.co

For the conditioning and acquisition of the voltage signal delivered by each of the thermocouples, the NI-9211 thermocouple input module from the National Instrument (NI) company was used. It has a 24-bit analog-digital converter, anti-aliasing filters, detection of open thermocouples, cold junction compensation for high-precision thermocouple measurements, double barrier of channel to ground isolation for safety, noise immunity and a high level of common-mode rejection. It has traceable calibration certificates issued by NIST, FCC and CE, among others.

The characterization of the thermocouples was carried out using the virtual instrument (VI), DAQ Assistant of National Instruments. This VI establishes communication between the modules and the computer, and enables the specification of the channels to be used and their configuration as inputs or outputs. It has the resources to characterize and linearize thermocouples type J, N, R, S, T, B, E and K. It also includes the compensation of temperatures produced by the cold junction.

Relative humidity measurement

To measure the relative humidity present in the laboratory environment, the sensor *HS1101* (MT-01, figure 1S) was used. This is a capacitive sensor with an operating range from $54\ \mathrm{kHz}$ to $100\ \mathrm{kHz}$. The typical signal conditioning circuit is an astable multivibrator with a 555 timer. This circuit is characterized by a continuous output in the form of a square wave (V_f) at a specific frequency, determined by the charge and discharge time of the capacitor. In this case, the HS1101 sensor is used as a variable capacitor.

Figure 2S, shows the schematic diagram of the developed circuit. A voltage follower was added at the output of the oscillator to couple impedances and avoid signal attenuation.

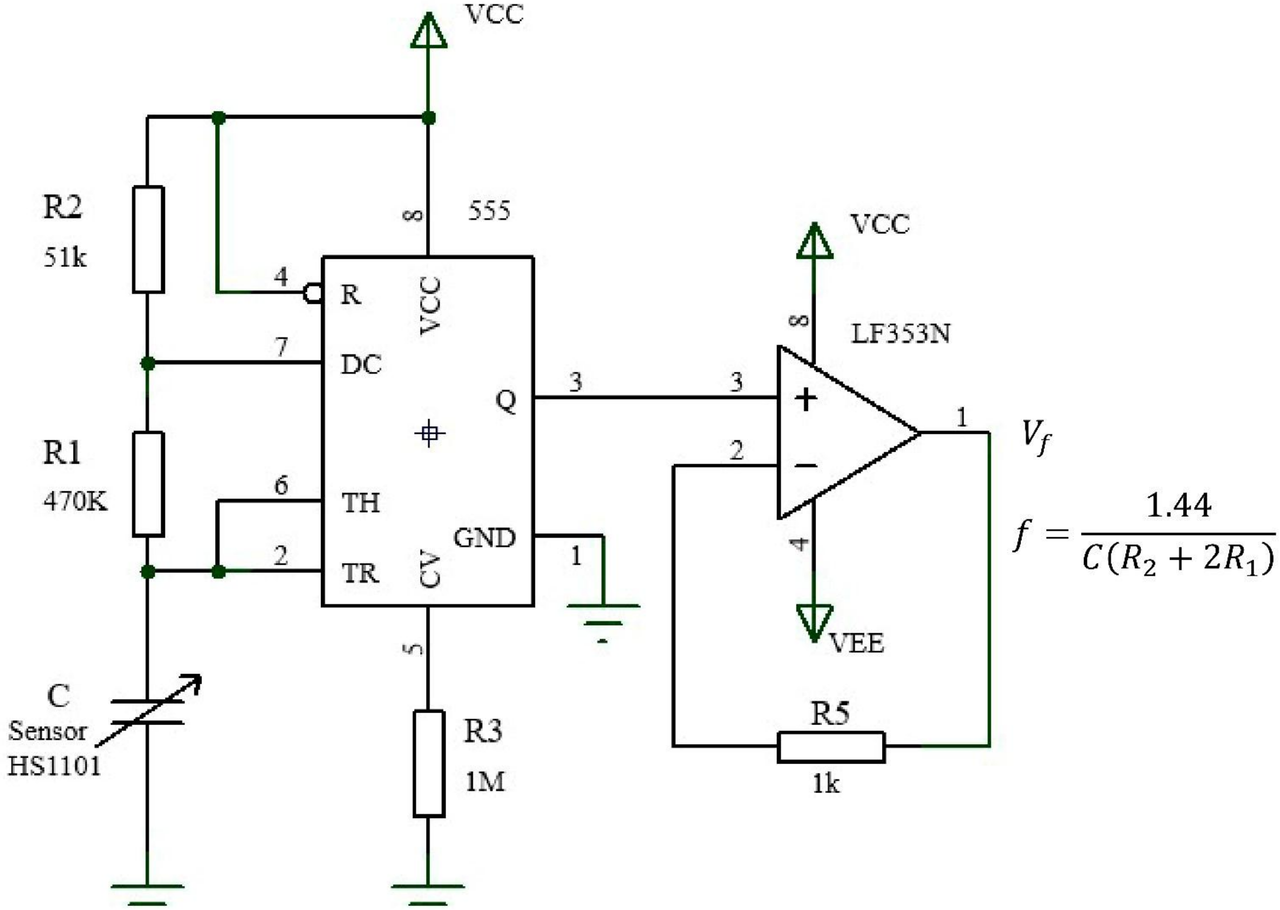

Figure 2S. HS1101 sensor conditioning circuit

Table 1S and figure 3S present the frequency response of the *HS1101* sensor to the variation of the relative humidity.

Table 1S. Hs1101 sensor response

%HR	Frequency (Hz)	%HR	Frequency (Hz)
0	7.351	60	6.600
10	7.224	70	6.468
20	7.100	80	6.330
30	6.976	90	6.186

40	6.853	100	6.033
50	6.728		

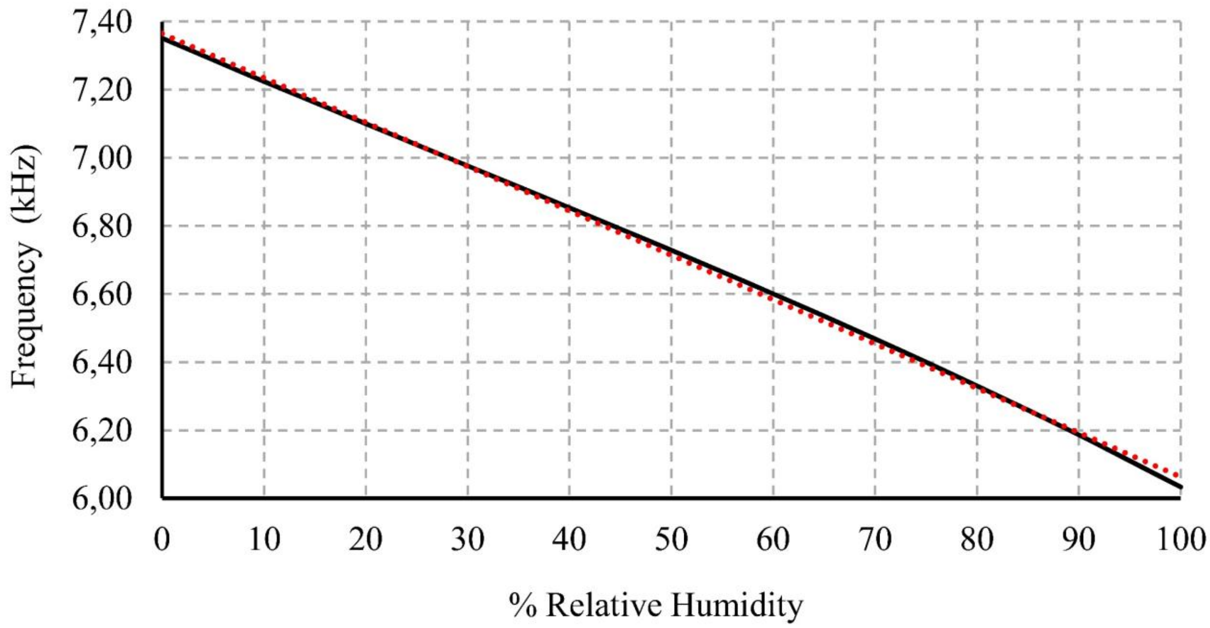

Figure 3S. frequency response of the *HS1101*

By applying linear regression to the data, we find the equation that is used within the *VI* to determine the percentage of relative humidity $(\%RH)$ present in the laboratory environment during the carbonization process of the samples.

Air quality measurement in the laboratory

The MQ135 resistive type electrochemical sensor (AT-01, figure 1S) was selected. It is sensitive to different gases such as: NH_3, NO_x, alcohol, benzene, smoke and CO_2. It is appropriate for monitoring air quality in the laboratory environment when pyroligneous gases are released in the pyrolysis process. The device has a heater to increase the internal temperature, so that the sensor reacts to the presence of gases, varying its resistance. It requires an external resistance or load to close the circuit and generate a voltage divider, which delivers a signal proportional to the quality of air it detects. The conditioning circuit suggested by the manufacturer is basic; it only requires a variable resistance connected to specific device pins and the power supply in direct current (DC). Figure 4S presents the schematic diagram of the circuit.

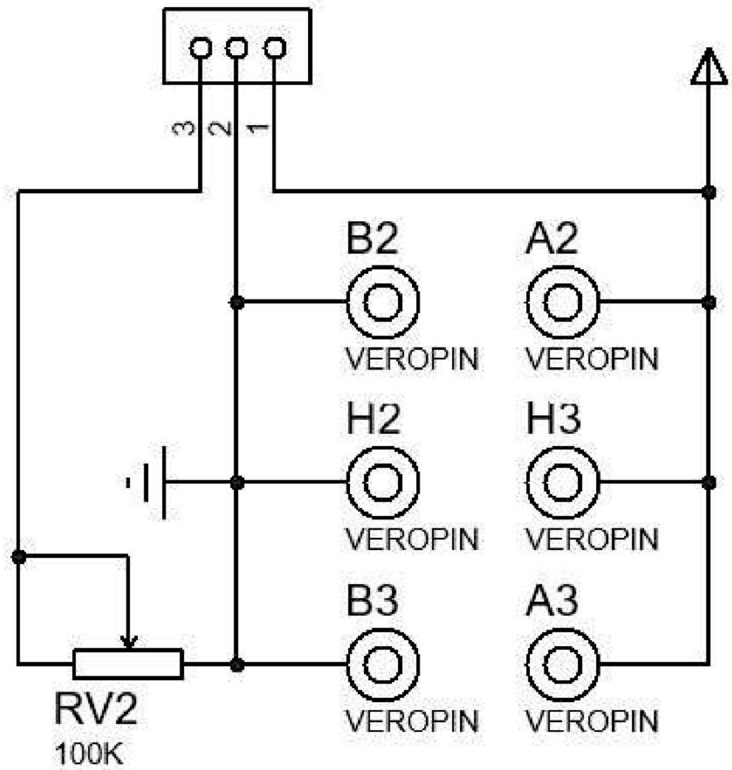

Figure 4S. Schematic diagram of the circuit MQ135 sensor

For the calibration of the MQ135 sensor, because the technical information given by the manufacturer is limited (**Olimex,** 2017) it was necessary to digitize the curve for CO_2 and ethanol. Regression was applied to the data to determine the function that makes it possible to establish the relationship between the number of particles per million (ppm) present in an environment and the variation of the parameter R_S/R_0. This, in turn, allows for the calculation of the voltage at the output of the sensor $\left(v_{mq}\right)$. The result is the power equations (1) and (2) for CO_2 and ethanol respectively.

$$ppm = 111{,}45\left(\frac{R_S}{R_0}\right)^{-2{,}885} \quad (1)$$

$$ppm = 105{,}45\left(\frac{R_S}{R_0}\right)^{-4{,}037} \quad (2)$$

R_0 refers to the resistance of the sensor to $100\ ppm\ NH_3$ in clean air, and R_S to resistance of the sensor according to the concentration of gases present in the environment (**Olimex,** 2017). The sensor was heated for 24 hours, as recommended in the datasheet, and the output voltage (V_0) was measured by exposing it to the laboratory environment (air) to establish the value of R_0, and to two ethanol solutions of $78.9\,mg/L$ and $100\,mg/L$, to validate the response of the sensor to the presence of gases in the environment

Figure 5S(a) presents the results of twenty samples taken after the stabilization of the response. Two measurements were made for each of the described experimental assemblies, and the average of the data was used to characterize the sensor, by means of equations (1) and (2) and the equation of the voltage divider generated between R_S and the load resistance of the conditioning circuit. The result is presented in figure 5S(b), where it is evident that the sensor responds to the variation of the concentration of gases in the environment, according to technical specifications and established parameters. This same procedure was repeated to verify the level of CO_2 in an environment generated in the laboratory.

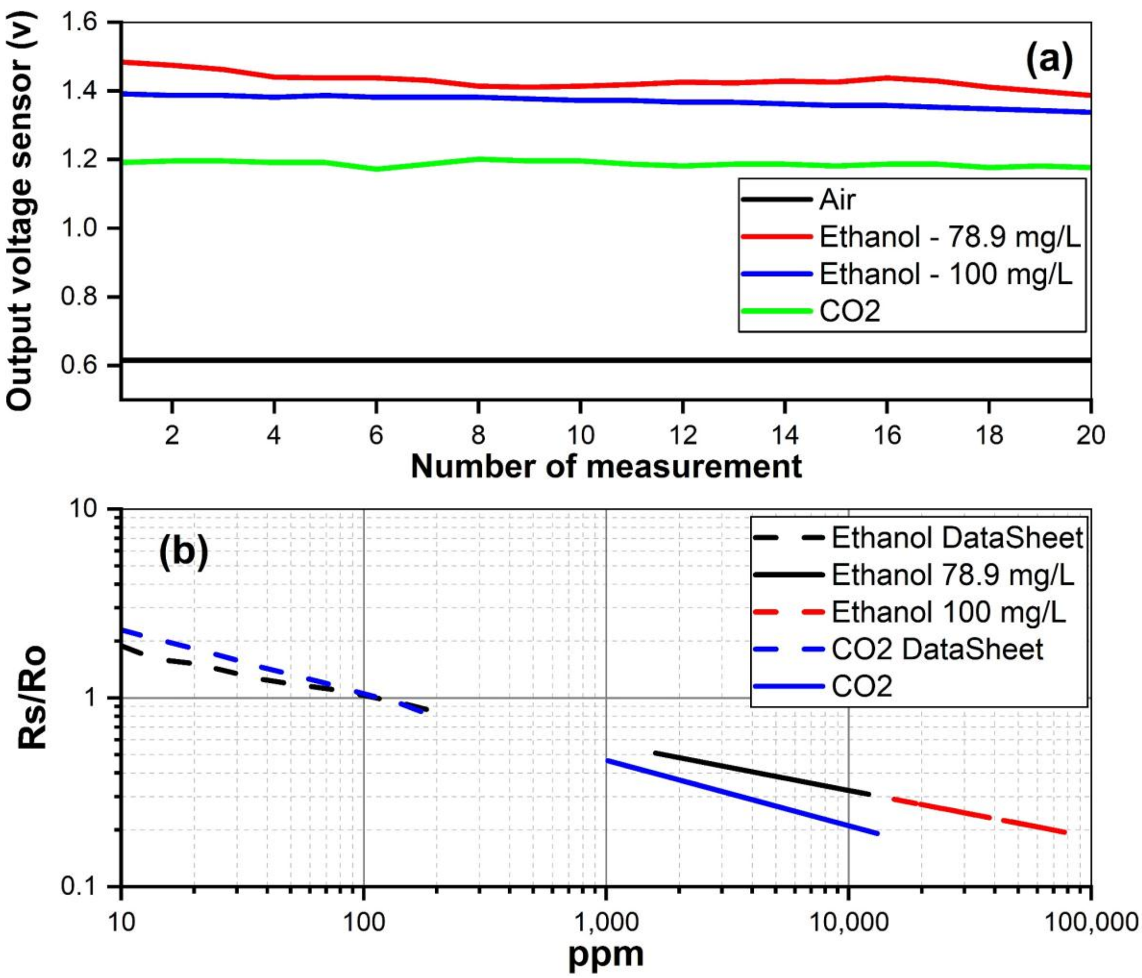

Figure 5S. MQ-135 sensor calibration

Temperature in the laboratory

To monitor the temperature in the laboratory T_L a Resistance Temperature Detector (*RTD)* type PT-100 (TE-03, figure 1S) with thermal coefficient $\alpha = 3.85 \times 10^{-3}$ was used. Figure 6S presents the conditioning circuit. The main component of the circuit is a source that supplies constant current to the sensor, regardless of its ohmic variation, to convert the variation of its resistance R_T produced by the temperature variation, in a voltage v_s whose magnitude is given by equation (3).

$$v_s = \left(\frac{V_r}{R}\right) * R_T \qquad (3)$$

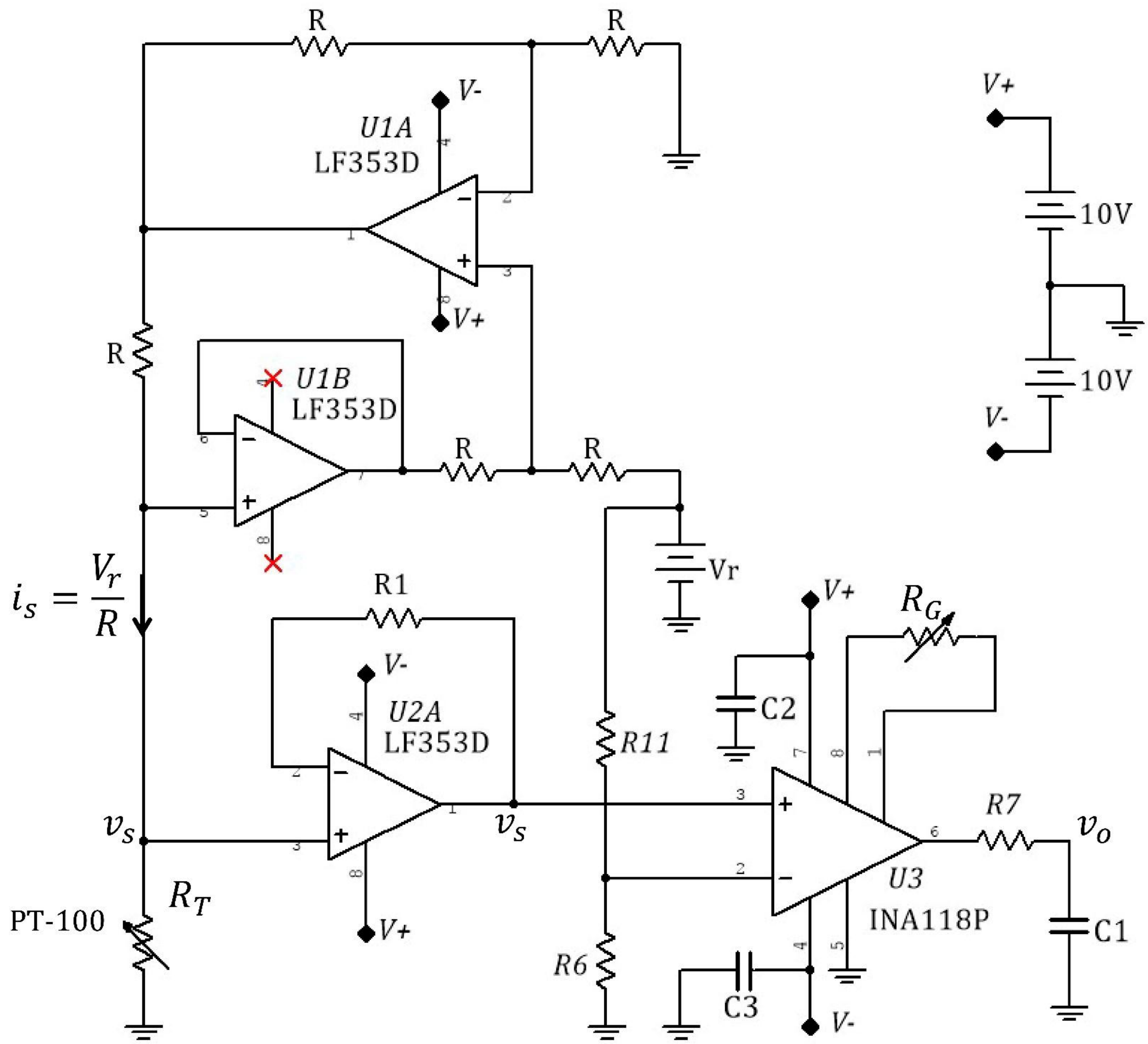

Figure 6S. PT-100 sensor conditioning circuit

The voltage v_S is applied to a voltage follower for impedance coupling, and it is also the input of the instrumentation amplifier (IA), whose function is to condition this signal so that the output voltage (v_0) is zero volts at a temperature of 0 ℃, and 5 volts at a temperature of 100℃. Equation (4) corresponds to the operation performed the IA.

$$v_0 = G(v_s - 0.1) \tag{4}$$

Where G is the gain factor of the IA and v_s is voltage delivered by the conditioning of the sensor. The voltage v_0 is applied to the VI, using the voltage input module NI-9201. Among the functions performed by the VI, is the conversion of v_0 to the corresponding temperature level.