Magnet current control system for fast field cycling NMR*

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In electronic field cycling NMR experiments, the magnetic field applied to the sample is generally switched between two or more constant levels. The principal difficulty involved when switching the current in the magnet, is the recovery time and the field stability necessary to do an NMR experiment. Therefore, electronic magnetic field cycling requires versatile control systems in order to obtain NMR stability under switching conditions. Up to now, serial PID feedback amplifiers have been using as main control unit, sampling the magnet current in a serial resistor. The aim of the article is to describe the circuit and performance of a new parallel PID control unit. Several technical details involved and differences with the series configuration are also discussed. Important auxiliary systems are described.

I. Introduction

Several experiments involving time dependent Zeeman magnetic field have been developed from the early years of the NMR[1-5]. Some recent important examples are nuclear magnetic relaxation dispersion studies[6,7], zero field spectroscopy[8,9] and a large variety of nuclear magnetic double resonance experiments[10,11]. Most of these experiments cannot be carried out in static magnetic fields. Any experiment at low field becomes difficult using standard NMR because of the loss of signal to noise ratio. When studing Larmor frequency dispersion of the spin-lattice relaxation time (T_1), field cycling technique becomes necessary to cover the low field limit. It is commonly assumed that T_1 spin-lattice relaxation at low fields can be replaced by rotating frame spin-lattice relaxation time $T_{1\rho}$, or dipolar relaxation time T_{1D} measured in static high magnetic fields. However, it is important to note that each of these relaxation times depend on different spectral densities and therefore, they become complementary experiments. On the other hand, there are several double resonance experiments that can only be done using magnetic field cycling[10].

A common feature of field cycling technique, is that magnetic field timing sequences are strongly dependent on the magnetic relaxation time constants of the sample. In particular, for several liquid crystals and biological compounds, spin-lattice relaxation times can take values under 3 ms at very low magnetic fields[12,13]. Therefore, magnetic field switching times had to be short enough to avoid energy flows from the spin system

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to the lattice and vice-versa (relaxation effects).

The home-built electronically switched field cycling spectrometer is equipped with a Schweikert-Krieg-Noack magnet[14] and a MOSFET-GTO power switch[15], built up with 60 parallel BUZ48 transistors and a Gate Turn Off thyristor (GTO). The control system driving the MOSFET-GTO switch allows to commute the magnetic field between different levels, with a stability better than 1:10^4 (measured in the NMR signal for an interval of about 30 minutes). This stability is reached for a 0.41T field single pulse 1.3 millisecond after the current is switched on from zero. On the other hand, the switching off takes about 700 microseconds. Figure 1 shows a block diagram including the current loop configuration for a three level field sequence. Technical details of the MOSFET-GTO switches as well as their basic function principles are extensively discussed in the literature[15,16].

2. Parallel PID regulator

The main control system is a parallel PID (proportional - integrator - differentiator) regulator[17,18]. When switching the field between two different stationary levels, two independent PID regulators are commonly used[19]. This control strategy is useful when the dynamic range of the system is very broad. In reference [19], the authors had to switch the field from zero up to 1T, and probably the transfer function of the system changes too much in this range. Therefore different characteristic are necessary for high and low level regulations in order to obtain a good stability.

In our case, the dynamic range is limited to a maximum field of 0.5T. In order to decide if two PIDs would be necessary, we carried out a study of the transfer function of the system. The results obtained conclude in that the transfer function is approximately constant over the whole range. This is the reason we opted to develop one single PID in parallel configuration. The calibration of the system is easier than the series configuration[20], because it is possible to add each stage at time, controlling different parameters one by one.

To make the corresponding calculations of the circuit, it was necessary to determine the transfer function of the whole system without switching the GTO[21]. This was done experimentally by pulsing the current under open loop conditions, and recording the response of the system by a sample signal taken at a sensing resistance SR (see fig. 1). The signals were recorded using a Nicolet 4094B digital oscilloscope. The transfer function of the system was obtained with the aid of a control simulation software. However, there are some effects originated when pulsing the GTO not included in the transfer function, such as damped oscillations when turning off the thyristor. Anyway, the parallel PID configuration allows to compensate easily this counterpart, by a suitable modification of its parameters (i.e., the individual gain of the stages plus the involved time constants).

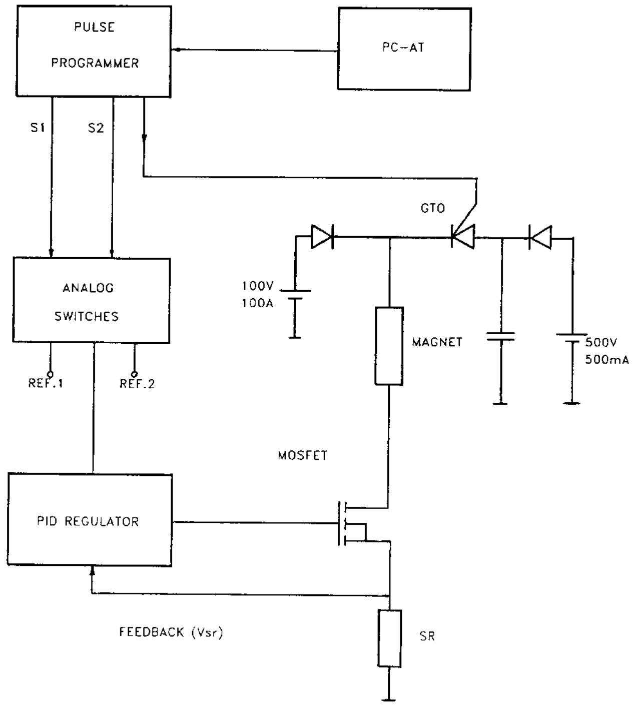


Figure 1. Block diagram of the current control system and the power switching device.

Figure 2 shows the circuit of the PID regulator, built up using Burr Brown OP27 operational amplifiers because of their satisfactory thermal drift characteristic and broad frequency band. The operational amplifier OA1 gives the difference between the control and feedback signals (differential configuration). P_{14} is used to fit the offset voltages, as do P_4, P_7, P_9

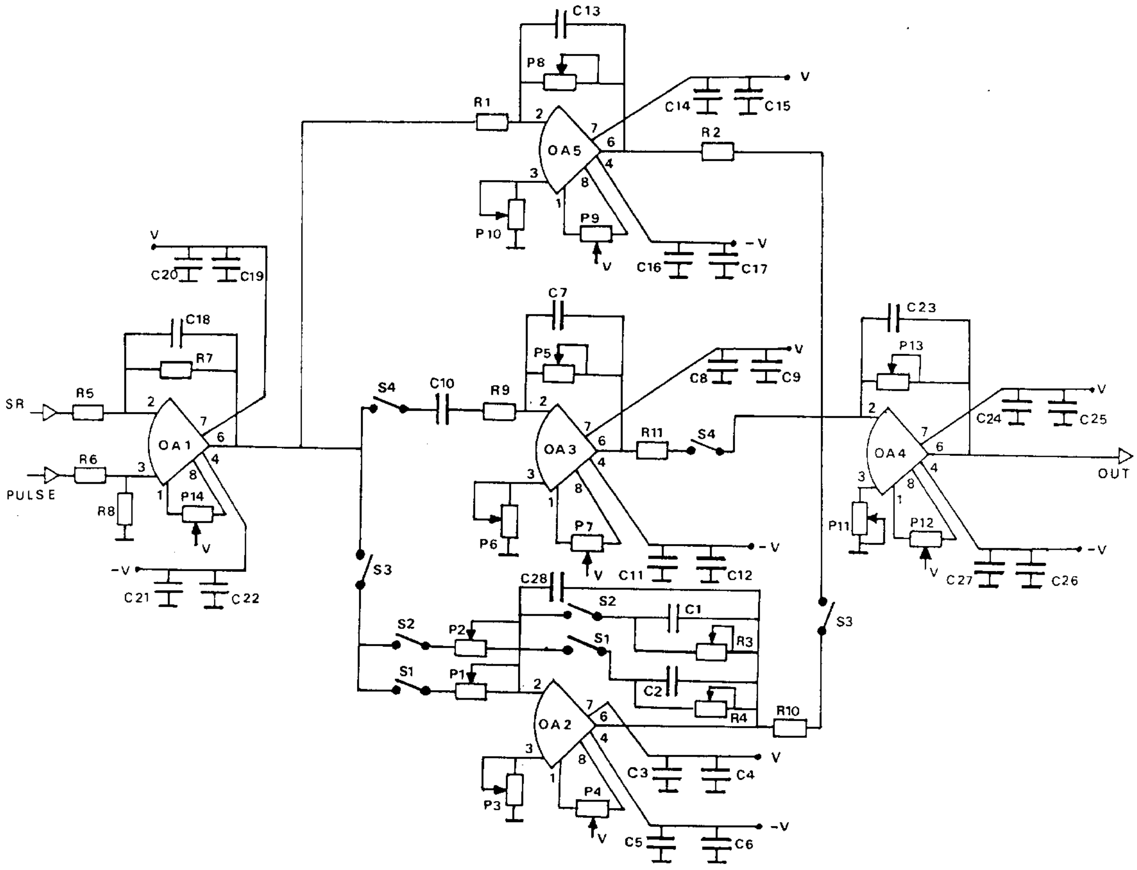


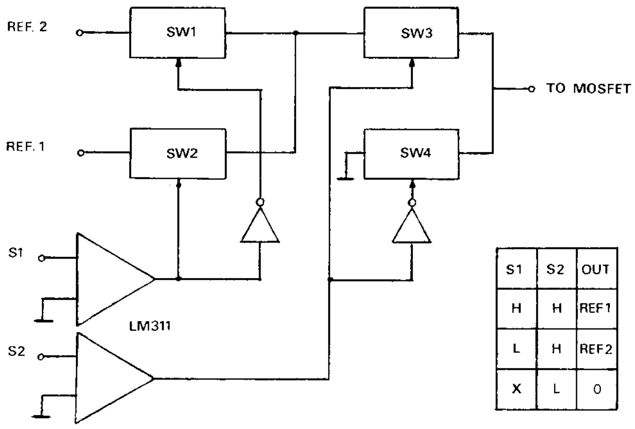
Figure 2. Parallel PID controller circuit. Resistors (values in ohms): $R_1 = R_2 = R_5 = R_6 = R_7 = R_8 = R_{10} = R_{11} = R_{12} = R_{13} = 10\text{K}$, $R_3 = 200\text{K}$, $R_4 = 500\text{K}$, $R_9 = 500$. Variable resistors (values in ohms): $P_1 = 50\text{K}$, $P_2 = 20\text{K}$, $P_3 = P_8 = P13 = 100\text{K}$, $P_4 = P_6 = P_7 = P_9 = P_{10} = P_{11} = P_{12} = P_{14} = 10\text{K}$, $P_5 = 5\text{K}$. Capacitors (values in microfarads): $C_1 = C_4 = C_6 = C_9 = C_{10} = C_{12} = C_{15} = C_{17} = C_{20} = C_{22} = C_{25} = C_{27} = 1$, $C_2 = 0.02$, $C_3 = C_5 = C_8 = C_{11} = C_{14} = C_{16} = C_{19} = C_{21} = C_{24} = C_{26} = 0.1$, $C_7 = C_{13} = C_{18} = C_{23} = 6$ pF. Operational amplifiers: OA1-OA5 = OP27.

and P_{12} in the rest. A frequency compensation capacitor (6 pF) between terminals 2 and 6 in all operationals was included. Switches 3-3' and 4-4' makes possible to disconnect the integrator (OA2) and differentiator (OA3) stages, allowing the system to function in proportional, integral-proportional and differentiator-proportional configurations. To provide an adjustable frequency band of the integral action, two different time constants are included in the integrator feedback, which can be selected by 1-1' and 2-2' switches. This allows to change the cut off frequency of this stage from 1.6 to 159 Hz with 1-1' (P_1) and from 160 to 4000 Hz with 2-2'(P_2). The integral action is principally related to the current stability in steady periods. The differentiator action covers a broad frequency band from about 30 Hz to up (P_5), and is specially concerned with the transition periods. The inverter amplifier OA5 is proportional to the error signal while OA4 adds OA2, OA3 and OA5 outputs. P_3, P_6, P_{10} and P_{11} are included to improve a correct compensation of the polarization input current unbalances. The gain of OA4 and OA5 can be adjusted with P_{13} and P_8, while P_5, P_1 and P_2 also allows to make corrections of the gains of differentiator and integrator amplifiers.

3. Electronic reference of the PID control unit.

The system allows to select three different references: a fixed electronic stable reference (National LM336), a programmable electronic reference developed with a 80C31BH microcontroller and ground (zero field level). The fixed reference is used during polarization periods of the sample and detection of the NMR signal, while the programmable reference is used in improving the Zeeman-field modulation during the relaxation or double resonance periods. Details of the programmable high stability function generator will be published elsewhere. Each reference can be selected by a set of analog switches, which are controlled from the main pulse programmer.

Figure 3 shows the circuit of the analog switches unit and the corresponding truth table. The LM311 voltage comparators are used as buffers for the control pulse sequences S_1 and S_2.



S1	S2	OUT
H	H	REF1
L	H	REF2
X	L	0

Figure 3. Analog switching device circuit and corresponding truth table for a three level field sequence. Reference 2 can be a time dependent voltage.

4. Pulse programmer unit

The home built pulse programmer is based on a programmable timer 8253 (INTEL). Up to eight pulse sequence and each pulse alone are available simultaneously. The eight pulses can also be distributed on two channels. The circuit was built up on a card, which can be directly installed in a slot of a PC. Figure 4a shows the circuit of the pulse generator module and the corresponding timing diagram. Each module is conformed of two timers, one 74LS74 D type flip flop (DFF), and outputs couplers. The timers are used as retriggerable monostables by positive edges. The DFF is used to synchronize the trigger signal of the computer with the clock. As can be seen in figure 4a, when timer 1 is triggered, a precise delay in triggering timer 2 is originated. The DFF reset is controlled by a programmable timer to reproduce periodically the pulse sequence automatically. In the pulse summing circuit (fig. 4b) each pulse can be selected by the corresponding bit value of a selection register applied to AND gates; while the sum of selected pulses is made by an OR gate.

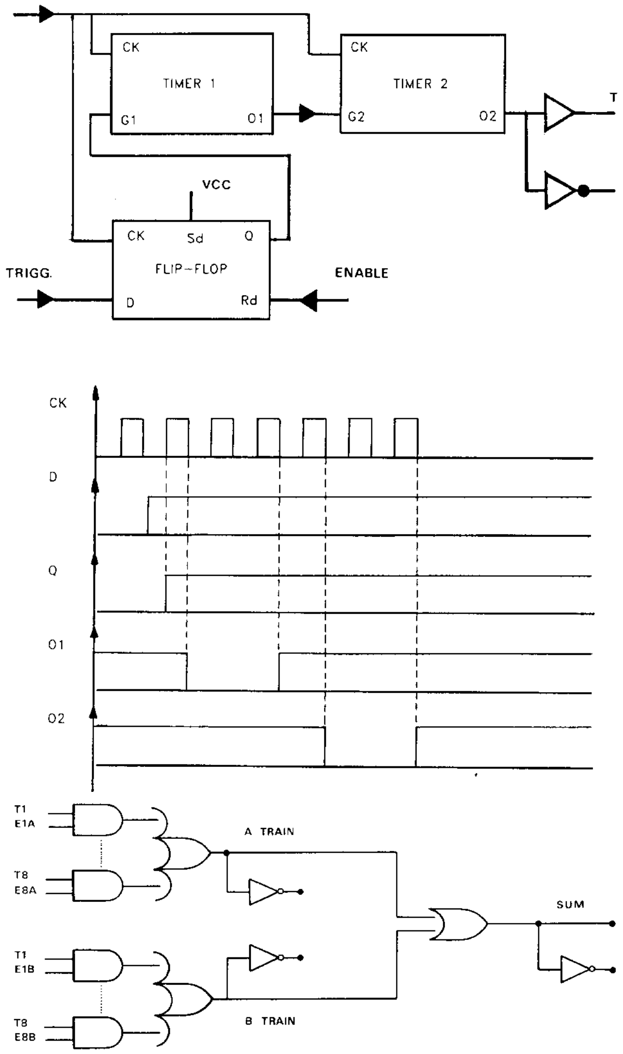


Figure 4. A: pulse generator module and the corresponding timing. B: Pulse summing circuit.

5. Performance of the apparatus

Figure 5a shows the response of the system under a pair of pulses. The recorded signal was taken at the sensing resistor and therefore is directly representative of the current through the magnet. The GTO is shot to assist the beginning of the second pulse. This sequence is commonly used for T_1 (spin-lattice relaxation time) measurements. The low field level in the picture corresponds to a proton Larmor frequency of 300 kHz, while the high level corresponds to 16550 kHz. The relaxation period is amplified in figure 5b, showing the 500 μs the system takes to stabilize the current in the magnet for different values of the relaxation field.

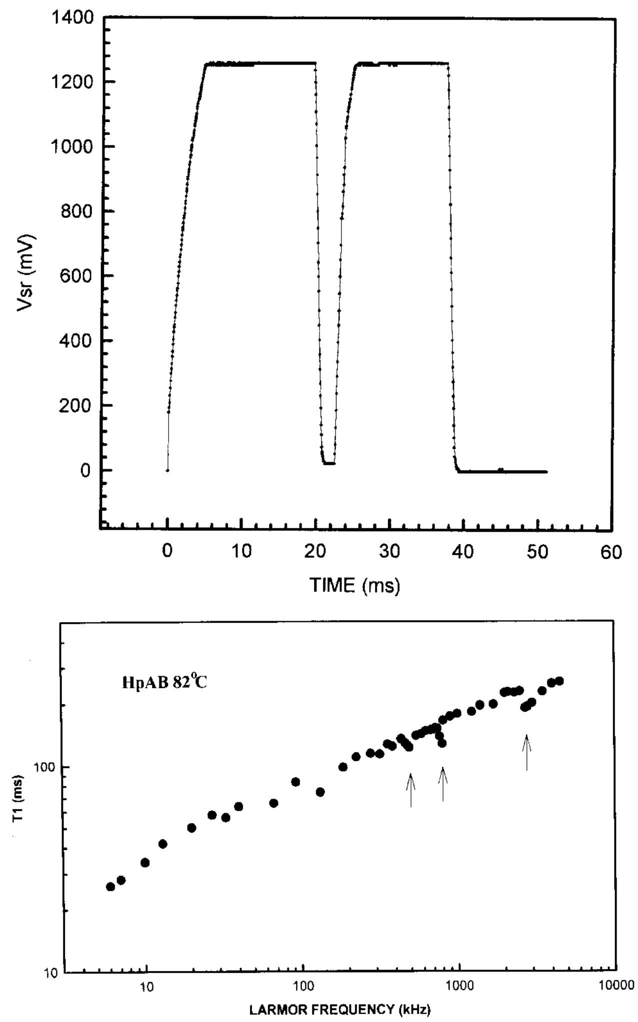


Figure 5. A: Response of the magnet current under a pair of pulses. The GTO thyristor is shot at the begining of the second pulse. B: magnification of the relaxation period showing the recovery time to the low level (about 400 microsecond).

A striking disadvantage in controlling current instead of magnetic field, are the effects originated by magnet mechanical deformations. This phenomenon produces fluctuations in the field, which the electronic control system cannot correct. Magnet deformations arise principally because of thermal dilatation during long polarization pulses and because of the strains present when pulsing the current. To control the field a TL173 hall sensor was used, eliminating completely this problem; anyway, the stability obtained was insufficient. A new field control system is under develop using more sophisticated magnetic field sensors. At the present, this effect is compensated by summing at the input of the control unit a signal proportional to the thermal cycle of the magnet.

Proton Larmor frequency dependence of nuclear magnetic spin-lattice relaxation time T_1, has been successfully used for the study of dynamical processes in liquid crystals and biological systems. If the molecule contains quadrupolar nuclei, an anomalous minimum appears in the $T_1(\nu)$ dispersion curve, because of an effective cross-relaxation between the quadrupole nuclei and the protons. This phenomenon is called "quadrupole dip".

In order to check the general performance of the spectrometer, $T_1(\nu)$ was measured in bovine serum albumin (BSA) hidrated 15 % by weight at 300^oK. A liquid crystal (HpAB) at 355 ^oK was also studied. Measurements in BSA were carried out in the frequency region of the quadrupole dip reported by other authors [19,22,23] (see figure 6). The dip found agree with data published in reference [22].

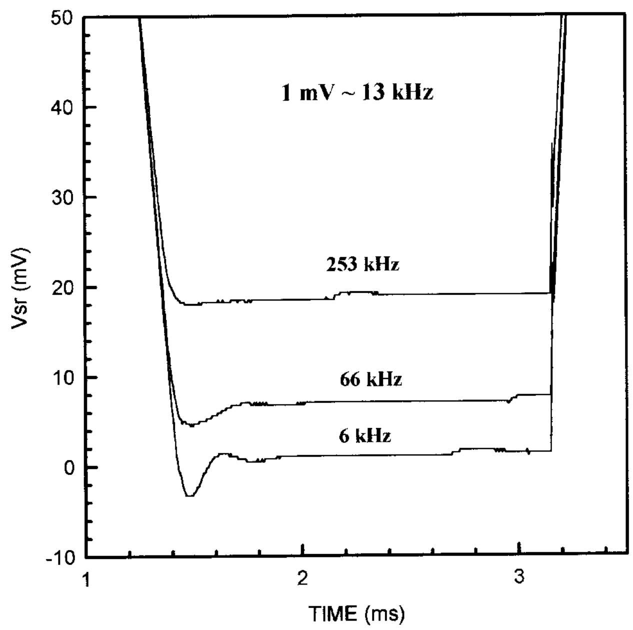


Figure 6. Hidrated BSA ^{14}N quadrupole dip in the proton NMR T_1 frequency dispersion. The dip is coincident with data reported by other authors[19,22,23].

HpAB (4.4'-Bis-Heptyloxy-azoxy-benzol) is a thermotropic liquid crystal in a smectic mesophase at 355^oK. $T_1(\nu)$ was previously studied at Stuttgart using field cycling NMR [24-26]. In addition in our group, we made experiments of the Larmor frequency dispersion of the dipolar relaxation $T_{1D}(\nu)$ [27]. Figure 7 shows $T_1(\nu)$ data from 6 kHz to 5 MHz, indicating ^{14}N-^1H quadrupole dips by arrows. Present measurements agree with reference [26], but exists a slight discrepancy

Table I

B_{max} [T]	t_{on} [μs]	t_{off} [μs]	t_r [μs]	P_{peak} [MW]	$(\Delta B(t)/B)$
0.5	500	700	1300	0.5	$<10^{-4}$

- B_{max}: maximum flux density (100A magnet current).
- t_{on}: minimum switching-on time with GTO assistance (500V) for a field flux density of 0.41T.
- t_{off}: switching off time from 0.41T to zero.
- t_r: minimum restoring time after switching needed to acquire NMR signal.
- P_{peak}: maximum power in the magnet circuit during switching on from zero to 0.5T and 500V GTO assistance.
- $\Delta B(t)/B$: temporal stability of the field.

with the width and frequency position of quadrupole dips reported in reference [24]. The new measurements (Zollino's and ours) were done under better magnetic field regulation conditions than reference [24]. A poor magnetic field stability will originate broader dips because of the Larmor frequency sweep during the cross-relaxation. Frequency position of dips also can be strongly dependent on temperature.

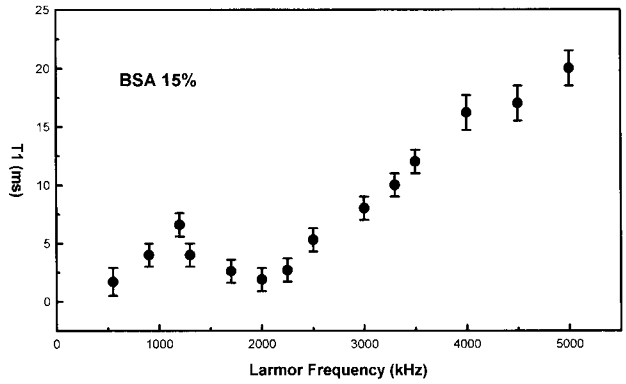


Figure 7. Spin-lattice frequency dispersion in the smectic mesophase of HpAB. Quadrupole dips were detected at similar frequencies reported by other authors.

Dips frequency and shape in HpAB are under study using field-cycling NMR in a similar way than was previously done with conventional NMR in a chlorinated liquid crystal [28]. Results obtained suggest important physical consequences to be described in a forthcoming paper. Table I resumes general technical information of the spectrometer.

6. Concluding remarks

The developed control system allows to control the magnetic field in a simpler way than other fast field cycling machines that uses two series PID amplifiers. Moreover, the finishing of the spectrometer is easier than using series configuration. Preliminary measurements presented in this work agree with previous results reported by other authors.

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