Nitric Acid Dosing Controller

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The Nitric Acid Dosing Controller (NADC) is an integral part of the Nitrc Acid Dosing Module (NADM) which was developed to ensure reliable and safe dosing of 90% nitric acid (and its mixtures with concentrated sulfuric acid) for use in flow chemistry research, specifically during the development of the furfural nitration platform.

The main ideas behind developing NADM are:

- Improvement of safety and reliability of the system.
- Minimalization of the need for manual manipulation with containers and tubing containing corrosive and reactive acid mixture by system's operators.
- Enabling the remote control and automation.

The NADM is based on a Flom pump (model UI-22-110DC) made of PTFE/PCTFE, offering sufficient chemical resistance for handling 90% nitric acid. The pump is complemented by several additional components, including two electrically actuated valves, a small peristaltic pump and a balance serving as a mass-flow meter. The valves were incorporated into the system to switch between feed containers (water or nitric acid) and to direct the pump's output to either the prime (waste) container or to the reactor system (see figure 1). This design significantly reduces the need for manual handling of the nitric acid container and tubing.

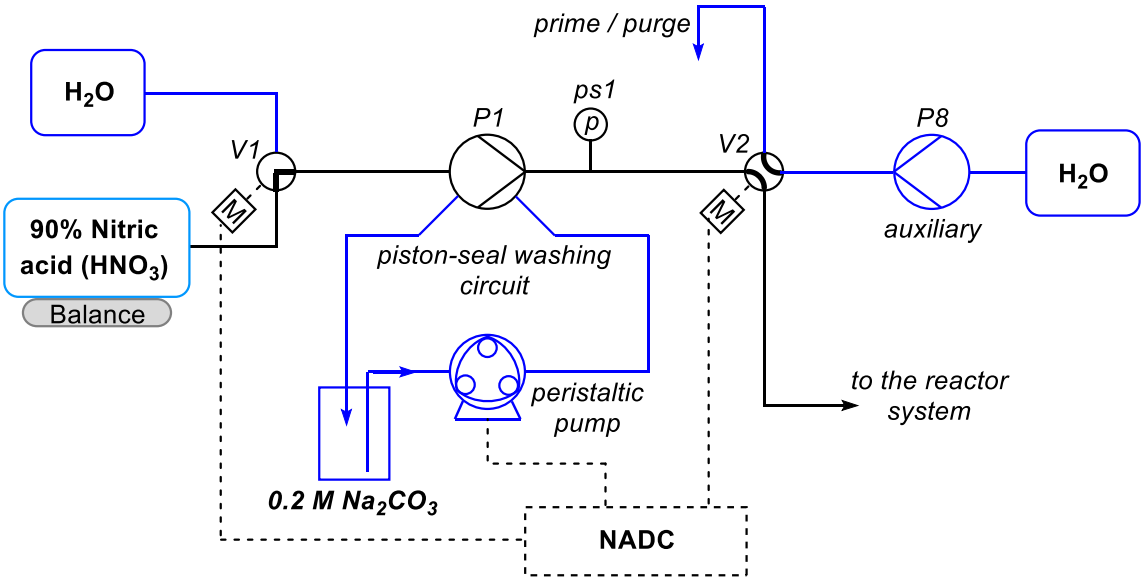

Figure 1. Detailed diagram of Nitric Acid Dosing Module - the setup of chemically resistant plunger pump and auxiliary elements used for delivering 90% nitric acid to the flow nitration setup. Circuits in blue are the auxiliaries, including priming/purging system, and a peristaltic pump for plunger compartment washing.

The peristaltic pump is used to wash the plunger/piston seal compartment of Pump P1 (as shown in Figure 1). Washing of the plunger seal compartment, located behind the main pumping chamber, is a common requirement for HPLC-type pumps, which are typically equipped with dedicated

ports for this purpose. Regular washing prevents the gradual accumulation of the pumped medium, which can lead to plunger seal degradation, corrosion, and in result a reduced pump lifespan or need of replacing seals and plungers. This is especially critical when handling corrosive substances like nitric acid.

To automate this process, a small peristaltic pump (Grothen model: G328, 12 V DC, with 2 × 4 mm silicone tubing; typical flow rate: 39 mL·min^{-1}) was implemented. The washing ports of both pump heads were connected in series, leading to the peristaltic pump and a flask containing a sodium carbonate solution (~0.1–0.2 M), using silicone tubing with a 2 mm internal diameter. The peristaltic pump was configured to push the solution through the plunger compartments, after which the output was recirculated back into the same flask (see figure 2).

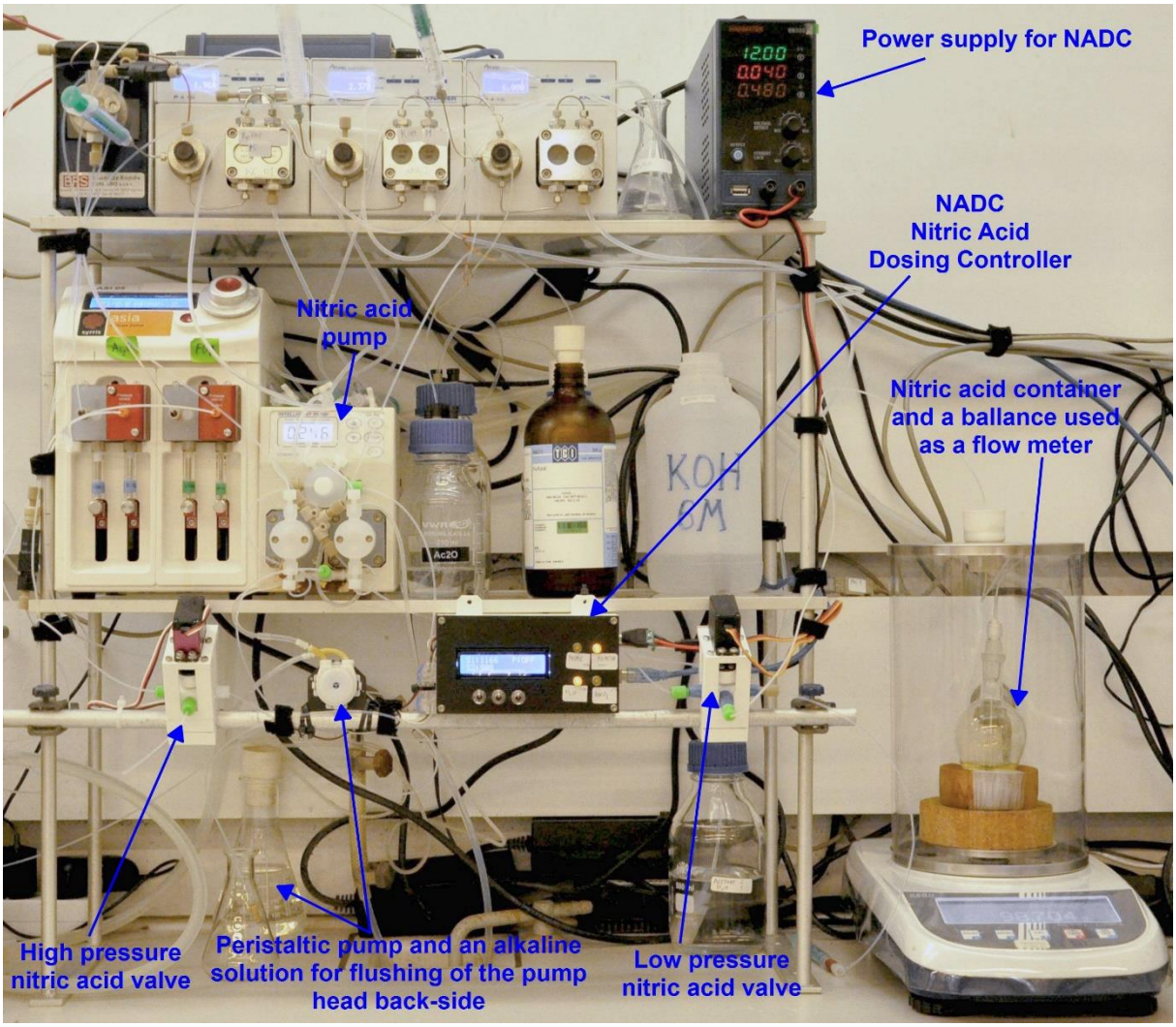

Figure 2. Overview of the Nitric Acid Dosing Module (NADM), showing the electrically actuated valves, peristaltic pump, Nitric Acid Dosing Controller (NADC), the nitric acid pump (Flom), and the balance used for flow-rate monitoring. Other pumps and feed containers being part of the system used during research on the furfural nitration platform are also visible.

The valves used in the NADM are detailed in another section of this repository, titled "Electrically-Actuated-Valves-for-Flow-Chemistry" Specifically, the document "4Way_DIBA_PTFE_LP_HP_valve_actuation_V1.pdf" provides comprehensive information, along with the associated folders containing 3D models for creation of 3D-printed elements.

Nitric Acid Dosing Controller – description of electronics

The Nitric Acid Dosing Controller (NADC) is based on an Arduino Uno R3 clone board, which controls two electrically actuated valves (detailed in other section of this repository, "Electrically-Actuated-Valves-for-Flow-Chemistry") and a peristaltic pump used for continuous washing the nitric acid pump's plunger compartment.

Manual switches and LED indicators

The controller features manual input through three monostable rocker switches, which control the states of both valves and the peristaltic pump. These switches operate in conjunction with 10 kΩ resistors—when the switch is open, the Arduino's input pin reads 0 V, and when closed, it brings the input to +5 V (refer to the schematic in Figure 3). To clearly indicate the status of the valves, a set of four yellow LEDs was added, and an LCD display connected via I^2C provides additional information, such as the precise valve positions and the operating state of the peristaltic pump.

Servo motor control

The servo motors controlling the valves are powered by a step-down DC-DC converter (based on the LM2596 module), providing a stable 6 V DC output from 12 V DC input. This DC-DC converter need to be adjusted prior to use with the servo, to provide voltage withing the range suitable for the servo.

The Arduino generates control pulses via its digital outputs to manage the servo motor positions. The pulse length determines the servo's rotation angle. When using the servoX.writeMicroseconds(value); command, the value typically ranges between 500 and 2500. In this project, 550 µs resulted in a 0° rotation, while 2550 µs produced a maximum rotation of ~270°. The specific values should be tested for each servo-valve combination to ensure the correct alignment between servo rotation and valve states. In our case, pulse values of 550 µs and 1166 µs were used to set the servo from 0° to 90°.

The servo motor pulse settings are located in the Arduino code, in the top section under the constant value declarations.

Peristaltic pump (12 V DC motor) control

The peristaltic pump is controlled through a MOSFET module (based on the FR120N), which controls the 12 V DC (see figure 4). To extend the life of the silicone tubing and pump mechanical parts, the peristaltic pump operates periodically, with approximately 1 second of pumping followed by a 4-second pause. To mitigate electrical noise generated by the pump's motor, a reverse-polarity diode and three ceramic 10 nF capacitors were installed: one capacitor and the diode between the motor power leads, and one capacitor between each lead and the motor metal casing (capacitor leads were soldered to the motor's casing).

Power supply

The entire system operates from 12 V DC, which is within the allowed input voltage range for Arduino Uno. The 12 V supply is connected to the Arduino's power socket and additional wires are soldered directly to adequate pads on the Arduino Uno's PCB (power socket leads) to distribute the 12 V to the MOSFET module controlling the peristaltic pump motor and to the DC-DC step down regulator providing 6 V DC for servo motors. If a peristaltic pump with a different input voltage requirement is used, the circuit must be adapted accordingly to provide adequate voltage. Step-down or step-up DC-DC converters can be applied to provide the necessary voltage if required.

Information on assembly

The connections between electronic components were made by soldering to ensure robust and reliable contacts. For interconnections, color-coded wires from ribbon cables or high-quality Ethernet cables (FTP/UTP 4-pair) were used. Although "gold-pin" connectors are an option, issues with loose electrical contacts were encountered. Some connections were made using a universal PCB matrix board. Resistors for the LED indicators and switches were soldered directly to the component leads and connected to the corresponding Arduino pins using longer wires, providing flexibility when handling the open enclosure.

All electronic components are housed in a custom-designed 3D-printed enclosure, identical to those used for the pressure and temperature measurement boxes (PTMBs). Additional holes were drilled for the switches and LED indicators, with diameters matching the components. Communication between the NADC and a computer is facilitated through the Arduino's USB interface (using a serial protocol), enabling remote control and monitoring of the valve and pump statuses. For safety reasons, manual control is prioritized over remote operations.

See figures 5 – 8 for additional information on the assembly.

Serial communication

The Arduino program ("NADC_Arduino_soft_v2_6.ino" in this repository) allows both manual control via three monostable rocker switches and serial communication with a PC at a baud rate of 115200. Commands are sent as ASCII strings followed by a newline (\n). The new line character is normally not displayed when tests are performed via Serial Monitor in Arduino IDE environment (pressing "Enter" key sends the command with "\n" character).

Example commands:

"*1 550*\n" – moves Servo 1 to position A (0°)

"*2 1166*\n" – moves Servo 2 to position B (90°)

"*3 0*\n" – turns off the peristaltic pump

"*3 1*\n" – turns on the peristaltic pump

"*4*\n" – requests device status

When requested ("4\n" sent), the device will respond via serial communication with the information on current status:

"*S1: YYYY; S2: YYYY; P: Y*"

Where YYYY represents the pulse width in microseconds for each servo, and Y is 0 (pump OFF) or 1 (pump ON). Example response: "*S1: 1166; S2: 1166; P: 1*" (both servos at 1166 µs, pump ON).

List of components:

Name	Type	Supplier
Arduino Uno Rev.3	ATmega328 based	AZ-Delivery
2x16 LCD module (with I2C interface)	HD44780 type with I2C-FC113 adapter	AZ-Delivery
MOSFET module	FR120N based (max 9.4 A, 100 V)	Amazon
DC-DC Step-Down Voltage Converter Module	LM2596, DC-DC buck converter, 3-40V input, 1.5-35V output, max 3 A; set to 6V	AZ-Delivery
Toggle Switch	Panel Mount, On-(On), SPDT, Solder Terminal (RS: 734-7117)	RS-Online
Yellow LED indicator	1903X00X Series Yellow Panel Mount Indicator, 2.1V, 6mm Mounting Hole Size	RS-Online
Diode	BA159	Amazon
Ceramic capacitors	10 nF	Amazon
Screwless terminals	WAGO 233 Series PCB Terminal Block, 2.54mm Pitch (RS: 181-4636)	RS-Online
Universal PCB matrix board	Single Sided Matrix Board FR4 1mm Holes; no. 457-0755	RS-Online
Servo-motor	DS3225MG-270 with 25 kg·cm torque and 270° rotation	Amazon
Servo-motor	D3009 with 9 kg·cm torque and 300° rotation	Amazon
Peristaltic pump	Grothen model: G328, 12 V DC, with 2 x 4 mm silicone tube; typical flowrate: 39 mL·min-1	Amazon

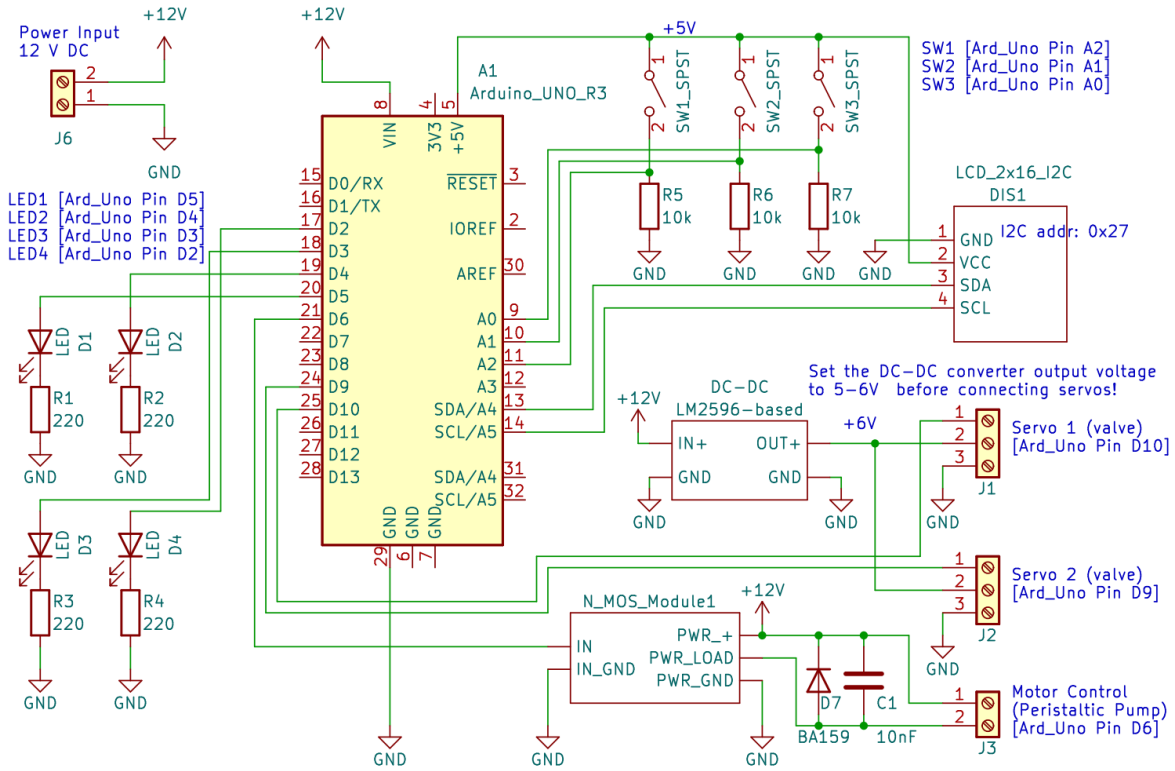

Figure 3. Schematic of the NADC electronic circuit.

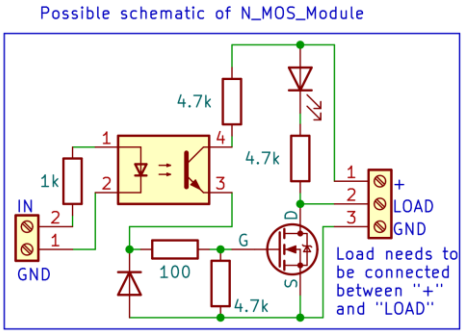

Figure 4. A simplified schematic of the commercial MOSFET module, illustrating the correct wiring for connecting it to the motor (load). The control signal from Arduino is connected to "IN" port, and controls the gate "G" of the MOSFET via optocoupler.

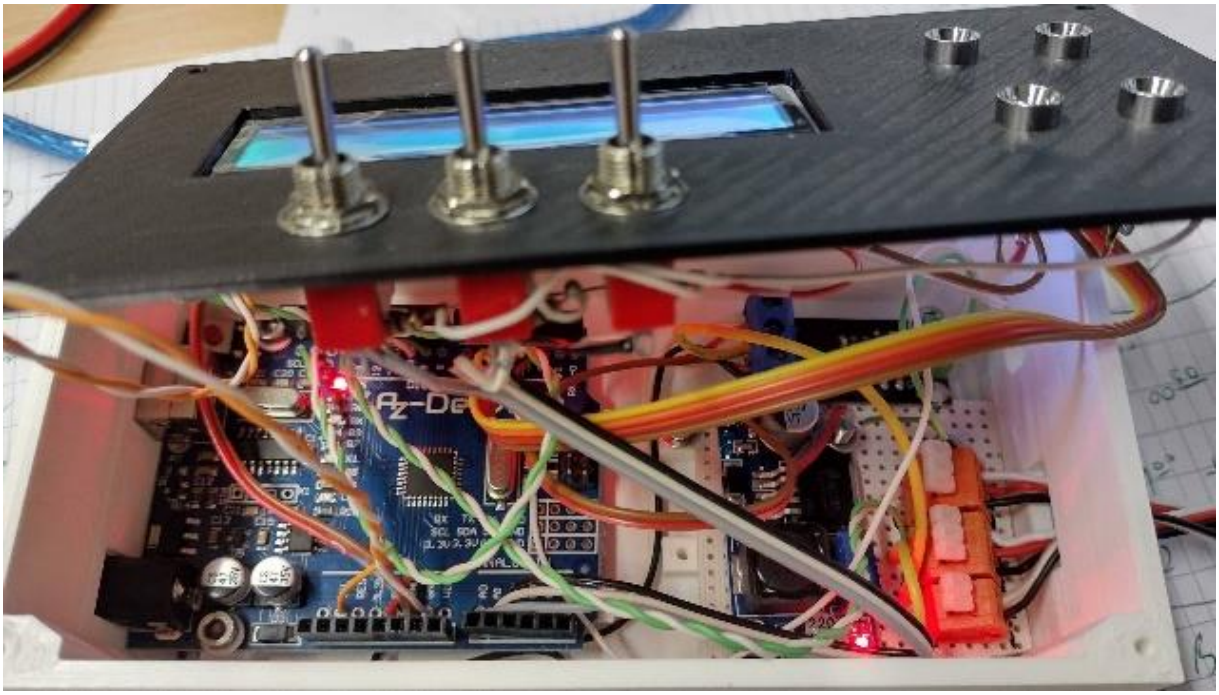

Figure 5. Photograph of the NADC before closing its housing.

Figure 6. View of the front panel of NADC during first tests. Later, during the use, 4 LEDs visible on the right side were labelled with adequate stickers providing information on the state of the valves (inlet and outlet of the pump).

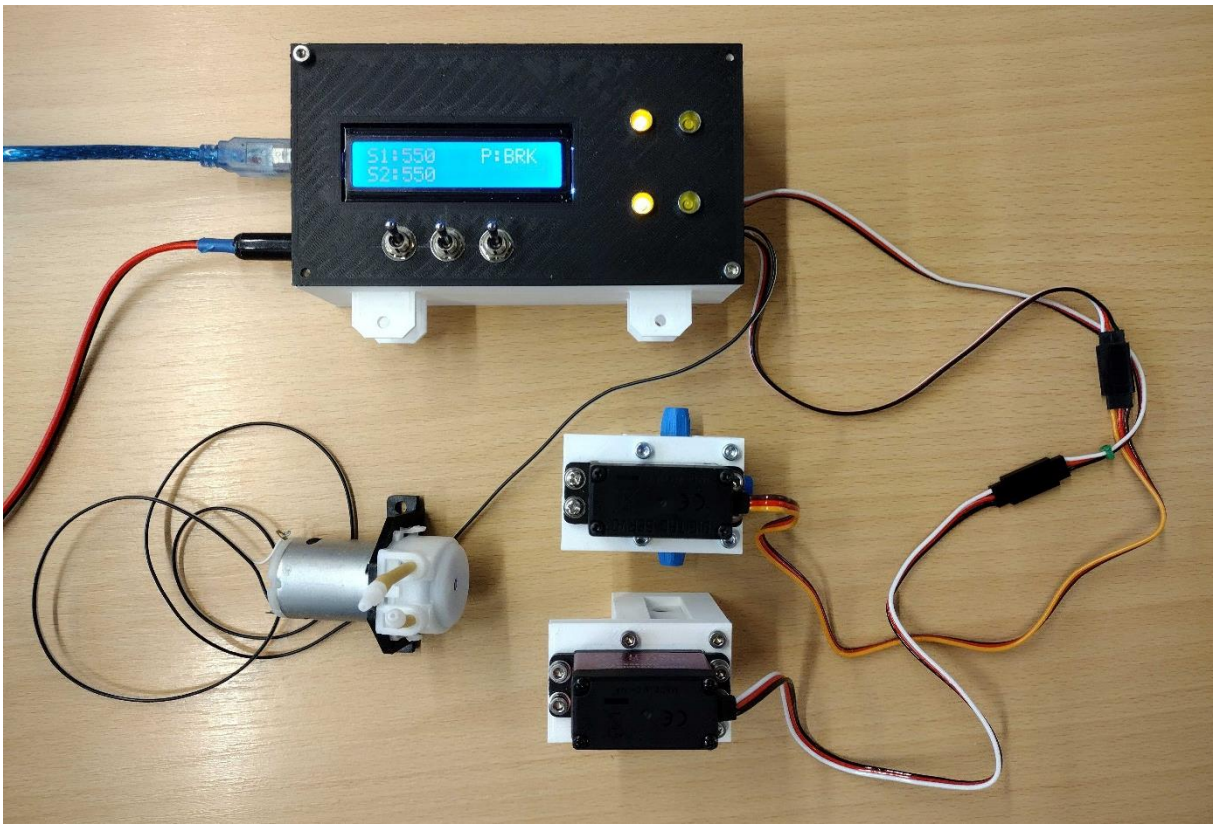

Figure 7. NADC during tests with both servo motors (modified DIBA valves) and the small peristaltic pump connected.

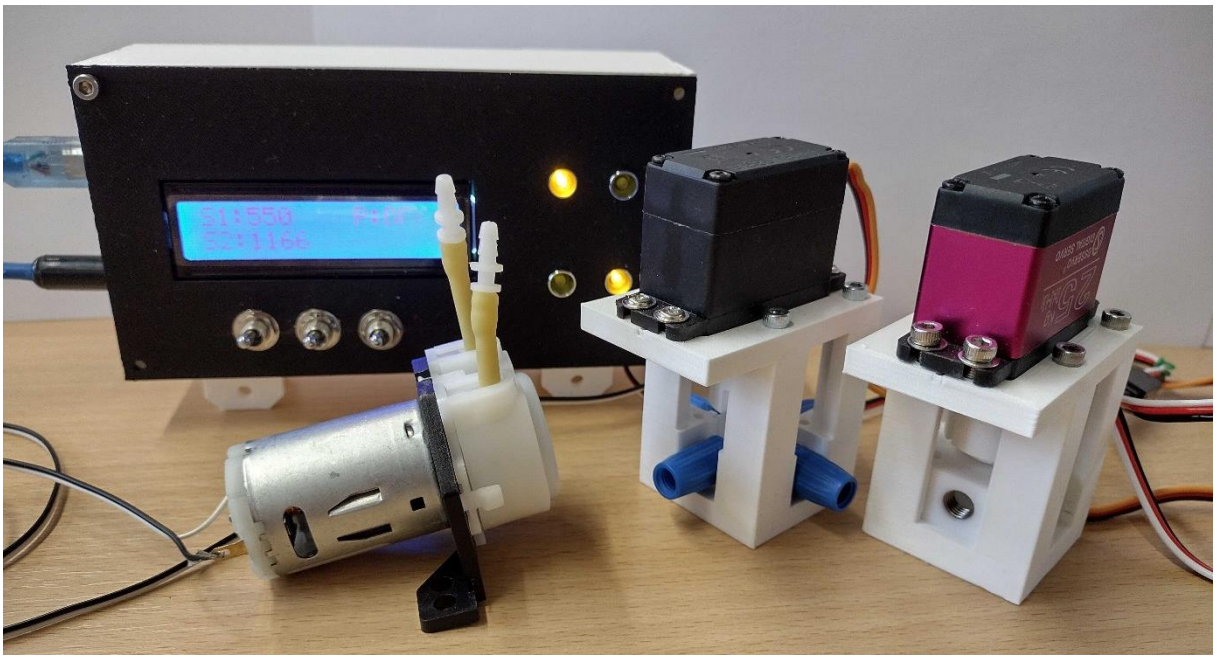

Figure 8. Overview of the NADC with valves and peristaltic pump before installation in NADM.

Acknowledgements

This research was supported by the U.S. Food and Drug Administration under the FDA BAA-22-00123 program, Award Number 75F40122C00192. The authors acknowledge the University of Liège and the "Fonds de la Recherche Scientifique de Belgique (F.R.S.-FNRS)" (Incentive grant for scientific research MIS under grant No F453020F, JCMM).