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ARM - Based Automatic Potentiometric Titration System.

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Research Article

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ABSTRACT

Accurate measurement and analysis are very important tasks for many applications ranging from industrial operations, analytical laboratories to biological processes. In modern analytical chemistry, automatic titrators have more prominence than manual interpretations. Various instruments have been developed and proposed in the past to carry out potentiometric titrations. Also there are suppliers available on the international market for automated titration systems. But they are costly with additional features. The present work describes the design and development of a cost-effective, reliable and simple automated potentiometric titration system. Addition of titrant to the analyte is carried out using stepper motor driven peristaltic pump which is designed and developed in the laboratory, incorporating ARM microcontroller, i.e. the whole system is built around an advanced 32 bit, low-power RISC processor architecture ARM7TDMI (LPC2148). The hardware and software are developed in the laboratory using off the shelf components. The software is developed in embedded C using Keil µVision4 software. The aim of the design is to carry out the automatic potentiometric titration for the end point determination of acid base titrations. The developed system is inexpensive, simple and works reliably.

INTRODUCTION

Scientific discoveries are being made every day that are changing the world we live in. From physics to medicine to biology, this contains some crazy innovations. Further advances in computing technology have helped with efficiency in the analytical chemistry laboratory in many ways. The first one is automation and simplification of analytical and synthetic tasks, which includes the use of computer controlled technology and measurement system to improve the accuracy, repeatability and increased utilization of equipment. Recent enhancements in computing technology and low cost capabilities of computing trends have resulted in a new generation of automated analytical instruments. Further the huge development of microelectronics and embedded automation systems changing the way we work, and also making us to be free from tedious tasks.

Automation permits us to carry out different types of experiments such as those requiring acquisition of data, and control of few parameters. Before the introduction of microprocessor automatic systems were not flexible [1]. In the present scenario, most of the automatic systems are controlled by microprocessor. They provide us many powerful and flexible ways of achieving full laboratory automation. Today automation is largely facilitated by the use of personal computers, which are increasingly more powerful, compact and inexpensive. Also they can communicate with scientific instruments and all kinds of devices using standard interfaces and may be programmed using high level languages [2, 3]. Potentiometric titration is one of the analytical techniques that could be automated with microprocessor based system.

For many years, potentiometric titration has been used widely to determine the ionic concentration, analytes in photo processing solutions, equivalence point or pH of a solution etc. In modern analytical chemistry, automatic titrators with potentiometric end point determination are routinely used for acid base titration and other types of titrations. Other applications of potentiometric titration include determination of salt ratio pKa, and ionization state of a drug compound, this is important attribute for drug selection and development. Many authors [4,5,6,7] have successfully implemented potential titrations for different applications like crystallization process of active pharmaceutical ingredients (APIs), acid base titration, and pH of a solution. Titration has also been employed to measure the binding constants between metal cations and small drug molecules [8,9,10]. With a conventional potentiometric titration instrument setup large amount of samples are required for an accurate quantitation or chemical analysis. Other techniques such as NMR tend to be time consuming and cannot be afforded by all laboratories. Also there are suppliers available on the international market for automated titration systems. But they are costly with additional features. Due to this reason, small laboratories cannot afford to procure such costly instruments. Such situations demand low-cost, automatic titrators with simple operation, high performance and improved accuracy.

Auto titrators developed by different authors and commercially available ones are reviewed [11,12,13]. From literature review, it is found that microcontroller based automatic titrators are very rare. Further low-cost, in-house developed stepper motor driven peristaltic pump based automation is rarely found in the literature [14,15,16,17]. Hence it is proposed to design and develop a simple, low-cost automatic potentiometric titration system for end point determination. The present work describes the design, development and fabrication of a novel instrument for automatic potentiometric titration, using a low power 32-bit RISC processor architecture ARM microcontroller LPC2148. This paper is organized as follows. Section II gives the brief introduction to ARM7TDMI based controller LPC2148. Section III describes the hardware and software details of the stepper motor based peristaltic pump for automatic potentiometric titration system followed by the calibration of flow rate of liquid prior to acid base titrations. Conclusion and scope of the future work is discussed in section IV.

Architecture overview of ARM7TDMI based Microcontroller LPC2148

ARM stands for Advaced RISC Machine developed by ARM Ltd which is most widely used in number of Embedded systems. Today ARM family accounts for approximately 75% of all embedded CPUs making it one of the leading architecture in the world. Previous designs used 8 bit/16 bit devices, but the designers are looking for highly integrated high performance ARM based 32-bit microcontroller. Heart of the design is ARM 32 bit RISC processor, hence brief description was given about its specifications below.

The LPC2148 board consists of ARM7TDMI as its core and it is designed by NSK shown in Fig 1, which is helpful to students to design embedded applications. This kit is benefit for the students in such way that all the possible features of the microcontroller will be easily used by them. ARM7TDMI family has good performance in situations where the energy consumption is critical design goal. LPC2148 has ARM7TDMI as its core is called CPU core. The modules inside are connected by the CPU high performance bus called Advance High performance bus (AHB) and the peripherals are connected by VLSI peripheral bus (VPB) [18, 19].

LPC 2148 has 2 types of memory.

- ✓ Flash memory 512KB
- ✓ Static RAM 40KB

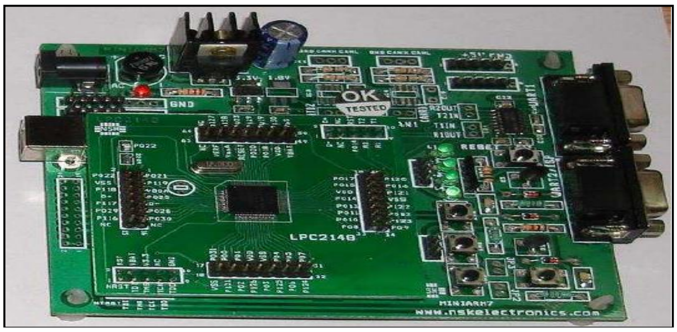

Figure 1: LPC2148 Mini ARM kit (ARM7TDMI)

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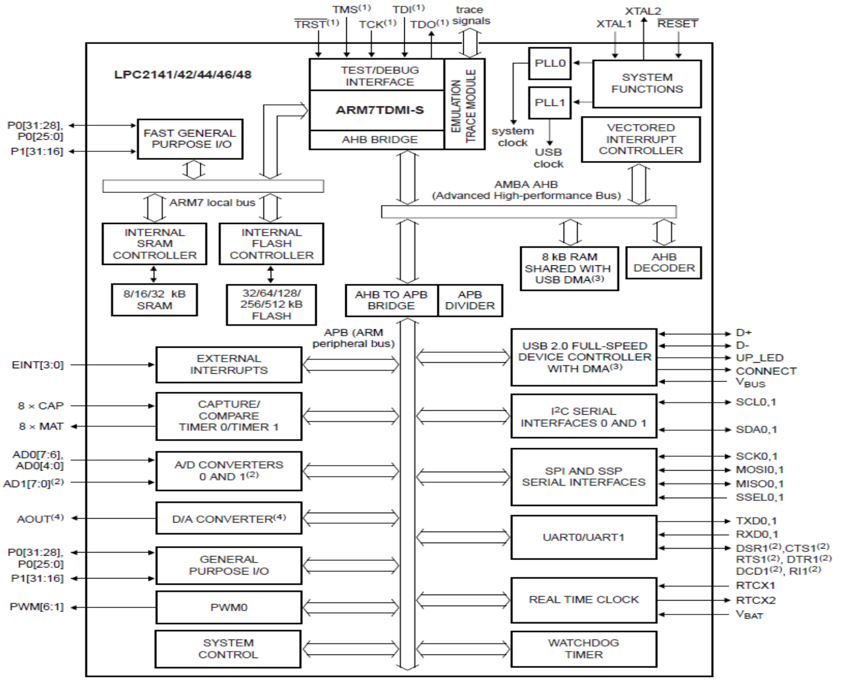

Figure 2: Block diagram of ARM 7TDMI

Hardware and Software details of the automatic Potentiometric Titration System

This part describes the hardware developed for the stepper motor driven peristaltic pump. The software details developed for the calibration of flow rate of liquid is also discussed here. The block diagram of the entire potentiometric titration system is shown in Fig 3.

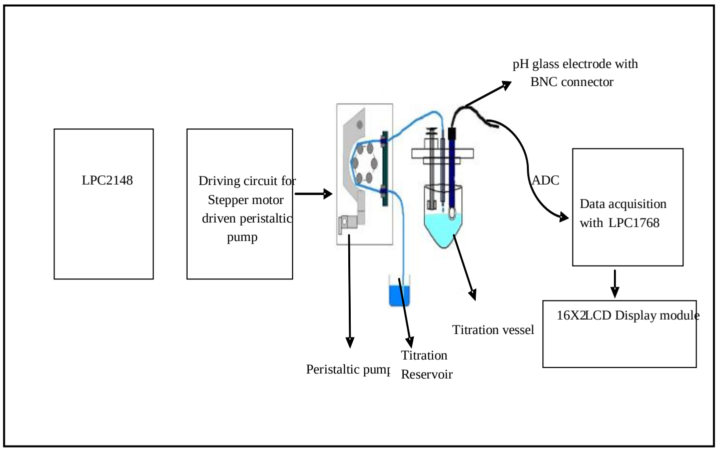

Figure 3: Block diagram of the complete automatic potentiometric titration system

The hardware designed and developed in the laboratory for the complete set up is divided into three parts.
Part A:

- Mechanical arrangement required for the construction of peristaltic pump and its working principle
- Electronic circuit for stepper motor driven peristaltic pump

Part B: Hardware required for the calibration of pH glass electrode
Part C: emf measurement and analysis system using LPC1768 controller unit.

But this paper describes the construction of peristaltic pump using LPC2148 and stepper motor, and also calibration of flow rate of liquid using stepper motor driven peristaltic pump designed in the laboratory i.e. Part A. Explanation of the above part A is described below.

Part A: Mechanical arrangement required for the construction of peristaltic pump and its working principle

Peristaltic pump is a device used to move different types of liquids. It has many applications such as in medical sector, R&D laboratories, Pharmaceuticals, food, chemicals, pump in and pump out with balanced flow etc. Now a days peristaltic pump is designed for various applications requiring dependability and volume of liquid to be pumped. For example in embedded system such as in MEMS technology an embedded peristaltic pump is a part of medical drug delivery system [20]. The construction, working principle and flow characteristics of peristaltic pump was described by many authors [21-23]. The photograph of the designed peristaltic pump system is shown in Fig 4. The parts which are required for the construction of peristaltic pump are the rotor, tube, shoe, pump hosing and base plate. The pump designed in the present work is vertical. It makes use of rollers in the form of bearings. The bearings are fixed on diagonally opposite axis. The bar on which the bearings are fixed is mounted to the shaft of the stepper motor. As stepper motor rotates, the roller which passes over the tube exerts pressure on the tube which squeezes the liquid from the suction (reservoir).

ARM7TDMI CPU operates with a maximum clock frequency of 12 MHz to 60 MHz and operating voltage is 3.3v. ARM7 CPU has a programmer's model. It has sixteen 32-bit General registers, 2 Special purpose registers – Current program status register (CPSR) and Saved program status register (SPSR). It also provides a wide range of peripherals like Two 10-bit ADCs with 14 channels, USB 2.0 full modem interface, Two I2c, Two SPI serial interfaces, Two 32 bit Timers, WDT, PWM unit, RTC with optional battery backup and 45 general purpose I/O pins. Block diagram of ARM7TDMI is shown in Fig 2.

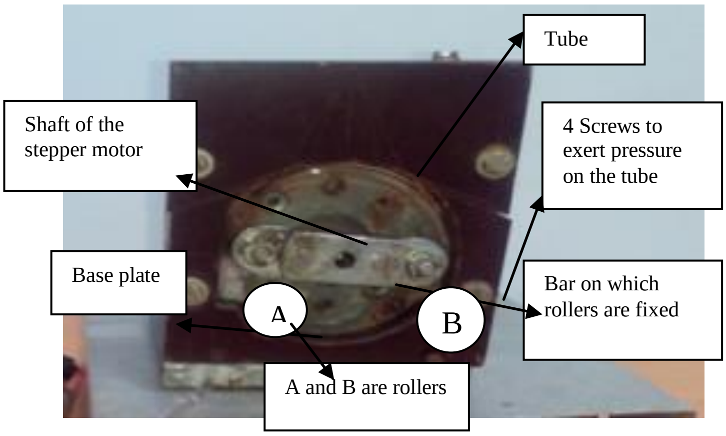

Figure 4: Designed peristaltic pump with important parts

The tubing is continuously squeezed by the rollers which push the liquid in the direction of the revolving rotor. It delivers the liquid to the other end of the tube.

According to theory, the flow rate depends on the following parameters:

- Internal diameter of the tube (higher flow rate with larger inside diameter)
- RPM of the pump head

Theoretical flow rate (ml/min) = V*L*N*RPM

Where,

V = volume of occluded tubing; L = Tubing length that will be occluded by pump rollers; N= Number of rollers on the rotor; RPM = rpm of the pump head

Certain advantages of peristaltic pump in automation of titrant are:

- Control of flow rate is simple
- Simplicity and suitability of operation
- Convenience in exchange of titrant

Electronic circuit for the stepper motor driven peristaltic pump

The block diagram of the stepper motor driven peristaltic pump is shown in Fig 5, and its schematic circuit is shown in Fig 6. An electrical drive circuit developed in the present work provides the requisite amount of current to the windings of the stepper motor. This is accomplished by using power transistors TIP 142 (T1-T4) to supply the necessary current for activating the coils. The components used for interfacing are listed below.

Components list: Stepper motor

ICs (1-4): MCT2E-Opto Isolator [23]
T1-T4 : TIP142 –Darlington Power Transistor [24]
D1 to D4: 1N4004-Diodes
Resistors: R1-R4: 220Ω; R5-R8: 1kΩ

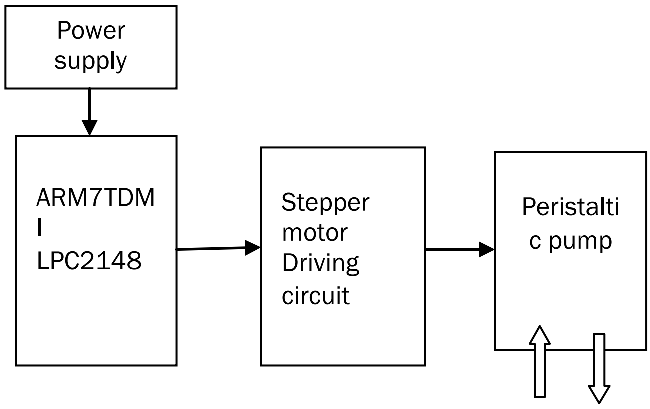

Figure 5: Block diagram of the stepper motor driven peristaltic pump

Stepper motors are characterized by high torque and are capable of handling large loads with precise movements. Presently used stepper motor is a unipolar one. Its important features are: step angle is 1.8˚ per step and rated voltage is 12V and it draws typical current of 1.6 A.

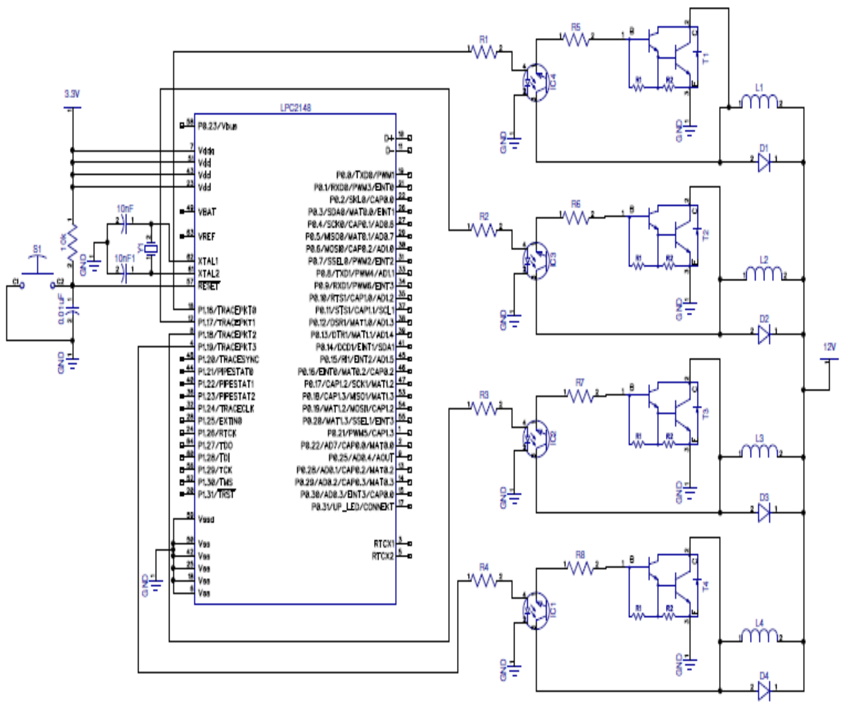

Figure 6: Schematic of the stepper motor driving circuit

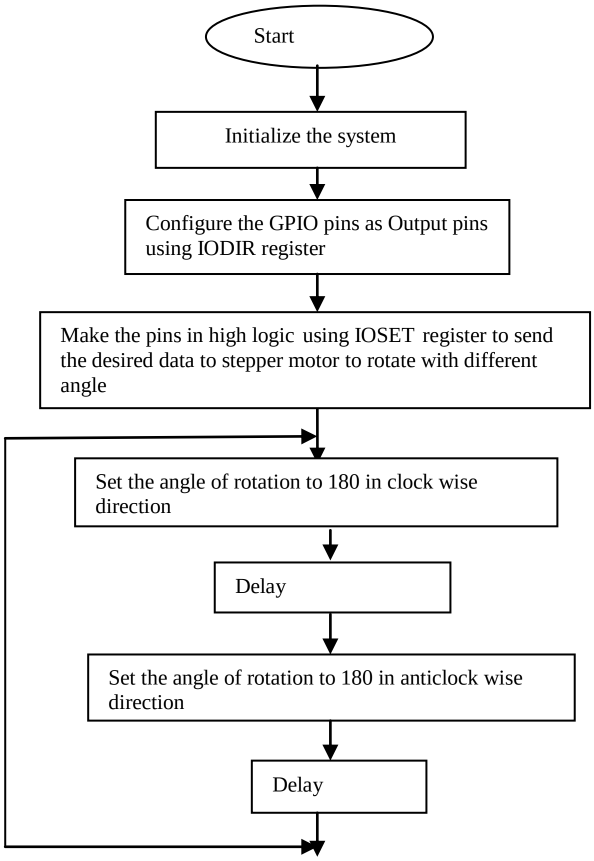

Figure 6: Flow chart to rotate the stepper motor both in clockwise and anticlock wise direction

The output pins P1.16-P1.19 of LPC2148 are connected to the Optoisolators (MCT2E) via 1kΩ resistors. MCT2E consists of a LED-Phototransistor combination integrated on the silicon and sealed hermetically. When the LED is activated by a high signal from the controller, light from LED falls on the base of the on-chip transistor, which makes it to conduct. This arrangement will protect the microcontroller from high current driving circuit by opto-isolation. The emitter of the on-chip transistor is connected to the base of the power transistor via 1kΩ resistor, which controls the current flowing through the base of the transistor: TIP142. The collectors of the transistors: MCTE and TIP142 are tied together and connected to the stepper motor windings. Diodes (D1, D2, D3, D4) are connected across the windings to suppress the back EMF and hence works as freewheeling diodes. M1, M2, M3, M4 represents the windings of the stepper motor. A high logic at any of the pins enables the on chip LED which in turn energizes the corresponding coil. A low logic disables the on chip LED which leads to de-energization of the corresponding coil. Thus the four windings are energized in a sequential manner to rotate the shaft of the stepper motor. Photograph of the developed system is shown in Fig 7.

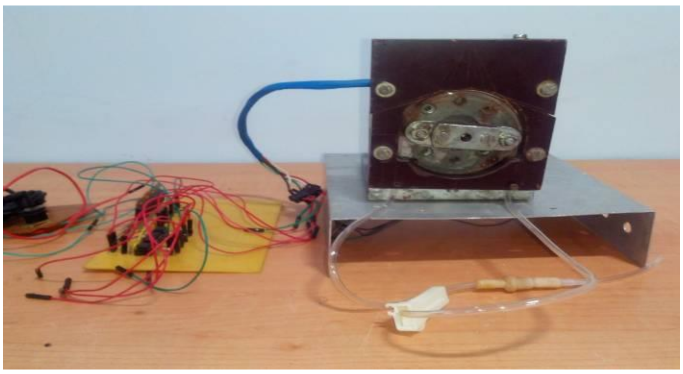

Figure 7: Stepper motor driven peristaltic pump with the PCB developed for driving the motor

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Software details

The present system is implemented by developing a suitable embedded C program using KEIL μ Vision4 software. This is an integrated development environment (IDE) with embedded C/C++ compiler for ARM which supports simulation and debugging interface. The software developed is compiled and uploaded to the flash memory using Flash utility (PHILIPS). The software developed to rotate the stepper motor with different angles at different speeds to squeeze the liquid from the reservoir or beaker is given in terms of flow chart as shown in Fig 6.

Calibration of flow rate of liquid using peristaltic pump

The overall accuracy and precision of the volume of titrant delivered by the automated stepper motor driven peristaltic pump are important parameters. The aliquots of water delivered by the peristaltic pump for different volumes at different stepping angles over a length of time are collected. Fig shows the photograph of the experimental set up for calibration of peristaltic pump. The volume of liquid collected for different stepping angles are computed and is used in the computation of determining the point of equivalence.

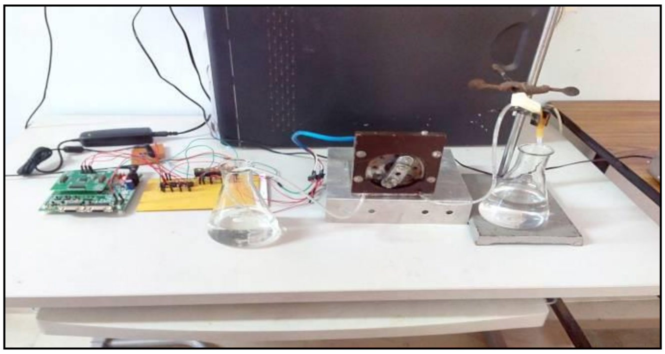


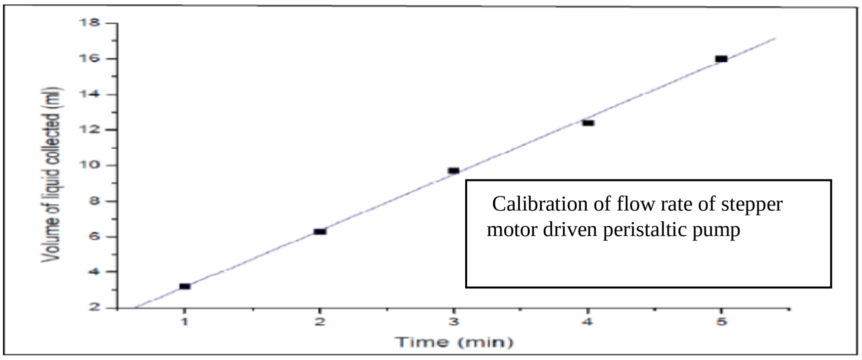
Figure 8: LPC2148 and Stepper motor based Peristaltic pump

Experiments were conducted to check the reproducibility of the delivered liquid. It is in good agreement with the previous readings over a length of time. The obtained values are shown in Table I and are linear fitted on a graph. The graphical representation is shown in Fig 7. The complete set up of peristaltic pump using LPC148 and stepper motor designed in the laboratory to squeeze the liquid from one end of the reservoir to the other end is shown in Fig 8.

Table 4.1 Data for the calibration of flow rate of liquid using Peristaltic pump

S. No	Time (min)	Volume of liquid Collected (ml) at different diameters of tube connected to the peristaltic pump	
1	1	1.8	3.2
2	2	3.54	6.3
3	3	5.3	9.7
4	4	7.1	12.4
5	5	8.5	16.3

Figure 7: Graphical representation of volume of liquid collected per time



CONCLUSION

An instrument is designed an inexpensive peristaltic pump using stepper motor and LPC2148 microcontroller for automatic potentiometric titration. The mechanical arrangement required for the construction and the design of peristaltic pump is presented in this paper. Further experiments were conducted to check the reproducibility of the delivered liquid. It is in good agreement with the previous readings. The hardware and software designed developed in the present work have to be tested for potentiometric titrations of different combinations of acid-base titrations which is a part of future study.

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