Sample Preprocessing Platform Based on Dissolution Method and Its Control System Design

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***Abstract*—At present, the sample analysis and detection process in many field such as food and medicine is spent most of the time on the sample preprocessing step. In order to improve the detection efficiency and reduce the preprocessing error, a sample preprocessing platform based on the dissolution method was designed, and its automation and control system were analyzed in detail. The platform automatically prepares solutions, handles batch samples, and ultrasonically extracts mixed solvents. The human-computer interaction interface and software system are designed based on PC and PLC for distributed control of valves, syringe pumps and three-dimensional motion mechanisms. The platform has high degree of automation and wide range of application, and solves the shortcomings of labor force, low efficiency and wide error source brought by manual preparation of solution.**

***Index Terms*—solvent preparation, control system, ultrasonic extraction, structural design**

I. Introduction

Sample analysis is widely used in food inspection, analytical chemistry, drug inspection, pesticide residue, environmental protection and other research fields [1-3]. Modern sample analysis is highly complex, and the concentration requirements of the detected substances are getting lower and lower, and the stability changes at any time, which brings a series of difficulties to the analysis. In particular, the possibility of direct detection after sample collection in various environments is small. As a result, the requirements for sample preparation and preprocessing techniques are increasing.

At present, many sample analyses in various fields require sample preprocessing, and it is a very time consuming, cumbersome process that is easy to introduce analytical error [4]. The dissolution method is a widely used in sample preprocessing technology, which needs to prepare a large amount of solution. At present, the technology of sample preprocessing and its automatic equipment just have short-term development and have many defects at home and abroad, such as manual operation and poor repeatability that bring big probability of error, the large labor intensity and the low efficiency [5, 6].

In the field of modern analytical chemistry, the development of sample preprocessing technology has achieved remarkable results, but there are still many deficiencies in related instruments and automation devices, especially in the production of samples and the preparation of solutions. Aiming at this problem, one sample preprocessing platform is designed in our laboratory that has the functions of quantitative addition of multiple solvents without cross-contamination, ultrasonic extraction [7, 8] and batch processing of samples, which is based on the requirements of the dissolution method and sample preprocessing technology [9]. The sample preprocessing platform has the characteristics of high efficiency, high adaptability and automation control.

II. The Basic Principle of the Platform

The fully automatic sample preprocessing platform based on the dissolution method is a mechatronics automatic control device that can finish automatic preparation of solutions, ultrasonic treatment and batch handling. Its functions include: quantitative configuration of solvent and no cross-contamination, ultrasonic vibration extraction to accelerate solution fusion process, automatic batch handling of samples. The basic principle block diagram of the platform is shown in Fig. 1.

The platform consists of the mechanical structure, the electronic control system and the software control system. The mechanical structure is designed in combination with the electronic control system, mainly including the three-dimensional motion mechanism, the dosing needle holder for adding liquid, ultrasonic extraction device, solution transfer system. The electronic control system consists of a solution transfer module and a solution preparation

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module. The solution transfer module is composed of the syringe pump, the distribution valve, the control circuit board and the pipeline system. The solution preparation module consists of the liquid adding unit, three-dimensional motion system, limit switch, stepping motor and its driver, PLC controller and ultrasonic extraction device.

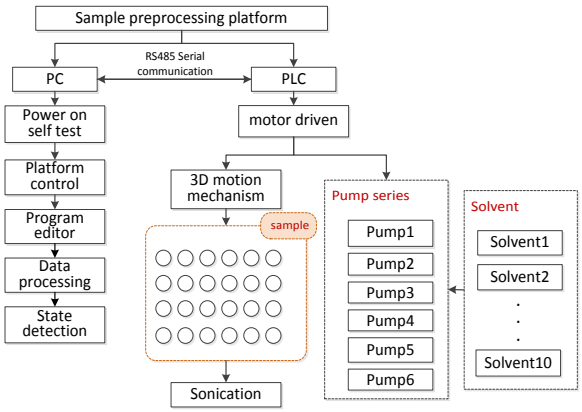


Figure 1. The block diagram of the basic principle of the platform.

The users compile sample information and solvent preparation scheme by operating the PC interactive interface. The distributed control model of PC and PLC is adopted when the platform works, and they exchanges data through serial communication protocol. The three-dimensional motion system drives the liquid adding unit to achieve accurate positioning operation and complete the transfer and preparation of the solution. In addition, the platform can automatically cleaning and ultrasonic extraction the mixed solvent to complete the solution configuration and prepare for sample testing. It has the characteristics of high efficiency, high precision and automatic control.

In order to eliminate cross-contamination between solvents, improve the accuracy of the solution configuration and speed up the processing efficiency, the process flow of the sample preprocessing platform was designed for the function of automatically cleaning and calibrating the solvent volume. It mainly includes the following steps:

1) Cleaning operation: Cleaning the platform through the washing liquid, removing the residual solvent in the pipeline system of the platform and the syringe to avoid cross-contamination;

2) Rinsing operation: Filling all the inlet and outlet pipes with solvent to eliminate bubbles and other impurities that may exist in the pipe and ensure accurate solvent volume;

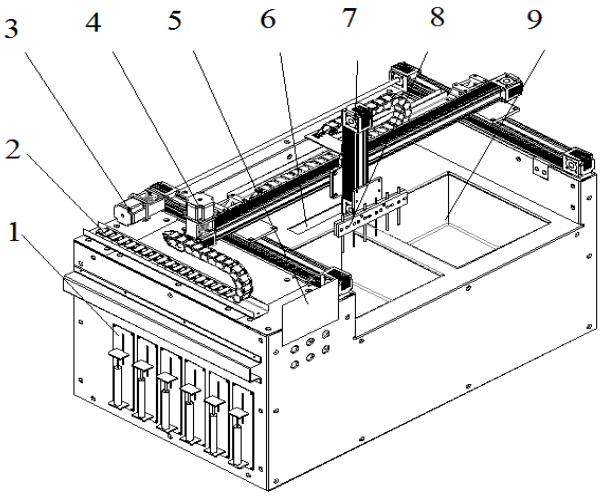
3) Solvent preparation operation: The dosing needle is driven by the three-dimensional motion system, to add the required solvent to the sample tube. Through the control system, the sample sequence is positioned to realize batch addition processing of the sample;

4) Ultrasonic treatment: Set the specific power and time for sonication, so that a variety of solutions can be quickly fused with the sample, and the mixed solution can be layered and extracted.

III. Mechanical Structure

The mechanical design technical indicators of the platform include: 1) having 96 sample positions and automatically formulating 10 solvents; 2) ultrasonically oscillating the sample mixed with various solvents; 3) design idea of mechatronics.

According to the mechanical structure of the electronic control system design platform. The mechanical structure mainly consists of a frame, a base, a series of syringe pump brackets, a three-dimensional motion mechanism, an ultrasonic extraction device, and an electronic control system. The overall structural dimensions are 1139mm×732mm×820mm. The 3D assembly drawing is shown in figure 2. The three-dimensional motion linear sliding table is mounted on the bracket, and an ultrasonic extraction device is arranged under the movement mechanism. The solution injection pump is disposed on the left side of the platform, and the needle tube for adding the solution is installed at the end of the Z-axis linear sliding table. Behind the rack is equipped with electronic components of the electronic control system.



1-Syringe pump bracket; 2-Drag chain; 3-Y-axis motor; 4-x-axis motor; 5-touch Screen; 6-Waste tank; 7-Z-axis motor; 8-Needle tube bracket; 9-Ultrasonic extraction device

Figure 2. Overall mechanical structure assembly drawing.

The three-dimensional motion structure is mainly composed of a two-dimensional side sliding table and a vertical sliding table. Taking into account the carrying capacity and smoothness of the mobile platform, the linear slide adopts the W45-15 model. The linear slide is driven by a timing belt. The type of timing belt is 3GT, which makes more compact between the timing belt and the timing pulley, and the transmission efficiency is higher. The 86 stepping motor can provide high torque and high speed power. In order to enable the linear slides on both sides in the Y-axis direction to operate synchronously, a synchronous lead screw is mounted at the end of the slide table, controlled by the same stepper motor.

The ultrasonic extraction device is mainly composed of an ultrasonic pool, an ultrasonic transducer and its circuit system. A sample tube rack can be placed inside the

ultrasonic pool for a total of 6×8×2 sample positions. When ultrasonically extracting a sample, it is necessary to inject an appropriate amount of an aqueous solution into the ultrasonic pool. The syringe pump is mounted on the bracket on the left side of the platform and connected to the dosing needle through a pipe. The dosing needle is above the ultrasonic extraction device and mounted on the z-axis. The proximity switch sensor is mounted on the three-dimensional slide to accurately position the sample. The pipe and the sensor circuit are packaged inside the drag chain, which makes the whole platform more tidier and safer.

The electronic control system mainly includes a touch screen, an industrial computer, the PLC main controller, the drives, the sensors, the relays, the power source and other electronic components, which are respectively installed on the interior of the electrical control cabinet.

IV. Control System

The sample preparation platform has two control modes: manual and automatic. The human-machine interaction interface is provided with a debugging module, which can manually control the sample pre-processing platform. The role of manual control is to debug hardware and software systems, handle faults, or invoke local functions of the platform. The local functions include initialization of the syringe pump, initialization of the 3D motion system, ultrasonic layered extraction of mixed solvents, and cleaning of the platform. Automatic control is the main control method of the sample preparation platform. The user edits the pre-processing scheme and sets related parameters on the human-computer interaction interface, and then starts the button, and the platform can automatically complete the equipment cleaning, solvent transfer, mechanical transmission, and ultrasonic treatment.

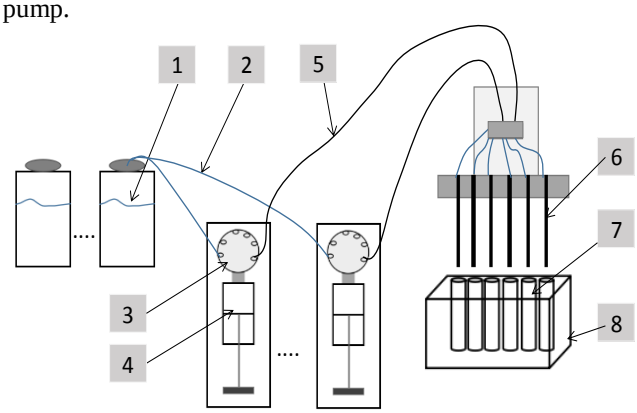
A. Fluid Transfer Control System

The basic principle of solvent transfer is shown in Fig. 3. The core control is the syringe pump, which integrates a distribution valve, a syringe, a distribution valve drive motor, a syringe drive motor and its circuit board. It has the advantages of integration and high precision. The dispensing valve consists of 11 inlet valve ports and 1 outlet valve port for automatic transfer of multiple solvents. The platform automatically adds a variety of solvents to the sample tube and ultrasonically oscillates the mixed solvent sample to complete the solvent preparation. The solvent bottle, the syringe pump and the dosing needle are connected by the pipe, and the sample is processed in batches to prepare a solution.

The syringe pump used in the platform has the advantages of high precision and integration. There are many valve ports in the distribution valve, which are equipped with a variety of solvents. After testing, the capacity of the syringe pump ranges from 50μL to 50ml, and the syringe pump motor has two modes: in the normal mode, 0-6000 step corresponds to the volume of the syringe pump; in micro-step mode, 0-48000 steps correspond to the volume of the syringe pump volume. That can achieve high throughput and ensure its volume accuracy. It is more intuitive and easy to send pulse commands with the way of steps. The fluid transfer system mainly includes high-precision syringe pump, dispensing valve, solvent tank, dosing needles mounted on a three-dimensional motion system, and a piping system. The syringe pump and the distribution valve are controlled by LabVIEW and PLC, and the pulse signal is sent to drive the distribution valve to open the valve port and the syringe pump to extract the solvent, and the solvent corresponds to the valve port. The speed of the syringe pump can be adjusted to achieve a better running speed and stability. The relationship between the number of steps and the volume of solvent in two different modes of the motor of the high-precision syringe pump is:

$$X = \frac{N * V_1}{V} \tag{1}$$

Where X is the number of motor driven piston steps, V is the maximum capacity of the syringe pump, V_1 is the required solvent volume, and N is the total number of steps corresponding to the full volume of the syringe pump.



1-Solvent; 2-Entering pipeline; 3-Distribution valve;
4-Injection pump; 5-Add solution pipeline; 6-Dosing needle;
7-Test tube rack; 8-Ultrasonic extraction

Figure 3. Basic principles of preprocessing platform solution addition.

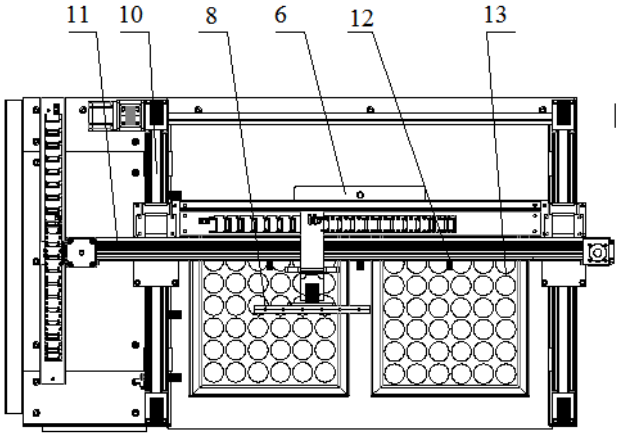
The entire process of solution transfer and formulation is automated through PC and PLC dual control systems. The injection pump is divided into four steps: opening the inlet valve - extracting the solvent - opening the outlet valve - discharging the solvent. PC and PLC use the RS485 OEM communication protocol to send instructions to the syringe pump to complete the corresponding mechanical actions. The volume of the extracted solvent is a variable set by the user on the PC interaction interface, and the upper computer software system processes and converts the data, converts the volume variable into an instruction, and directly sends it to the injection pump. The other three operations of opening the dispensing valve port and discharging the solvent have a specific command form, so the syringe pump is directly controlled by the PLC that is responsible for continuously interrogating all the working states of the scanning syringe pump and feeding it back to the PC. The communication mode of the syringe pump is OEM

communication protocol, such as pump initialization command, the ASCII code format is /1ZR; the hexadecimal format is 02 31 31 5A 52 03 09.

B. Transmission Control System

The sample preprocessing platform based on the dissolution method can automatically add a plurality of solvents to the sample tube, and ultrasonically oscillate the mixed solution to fast fuse and extract the sample to be tested. The transmission control system is designed to replace manual operation, improve work efficiency, batches processing samples. That controls the movement of the liquid feeding needle by controlling the three-dimensional motion mechanism which can accurately position the sample sequence and the waste liquid tank to adding a solvent to the designated sample sequence. Therefore, the hardware part of the transmission electronic control system mainly includes PLC, injection pump, two-phase stepping motor, motor driver, relay, and proximity switch.

As shown in figure 4, the number of the dosing needles is six, and is mounted on the Z axis, and the sample position is 96, which is arranged inside the ultrasonic pool with 6×8 form. The user sets the number of samples that need to be processed beforehand on the interactive interface. The software program converts the data into instructions for transmission to the PLC. The PLC interprets the meaning of the instruction, obtains the number of sample sequences, drives the XYZ three-dimensional motion mechanism in the form of a pulse, adds solvent to the specified sample sequence, and completes the function of the automated batch processing sample.



6-Waste tank; 8-Needle tube bracket; 10-Y-axis;
11-X-axis; 12-Sensor; 13-Test tube rack

Figure 4. Transmission system of the sample preprocessing platform.

The platform uses proximity switches to achieve precise positioning and zero setting of the drive system. It can be operated without mechanical contact with moving parts. When the object approaches the sensing surface of the switch to the operating distance, the switch can be operated without mechanical contact and any pressure to drive the DC electrical appliance or provide a control command to the PLC.

Three proximity switches are installed on the X-axis of the three-dimensional motion system, which are 1 origin and 2 limit points; 4 proximity switches are mounted on the Y-axis, which are 2 limit points and 2 positioning points; Z-axis mounting 2 proximity switches for 2 limit points. This enables precise movement in the plane and in the vertical direction. The PLC compares the current position with the received position command to obtain the difference in orientation. Then the PLC sends the required pulses to the stepper motor driver, and the proximity switch can verify the sample position.

C. Software System

The software system is designed in accordance with sample preprocessing technology. The multi-functional human-computer interaction interface designed on LabVIEW graphical programming language exchange data with PLC and syringe pump by RS485 serial communication.

The basic principle of the automation control system is shown in Fig. 5. PC and PLC communicate according to RS485 serial port, write communication protocol to transmit data and feedback signals. The PC is responsible for process sample preprocessing information and send instructions to the PLC. The PLC is responsible for continuously scanning the working state of the syringe pump, the feedback information of the proximity switch sensor, the orderly motion of the three-dimensional motion mechanism, and the operation of the ultrasonic extraction device. The distributed control of the syringe pump and distribution valve through PC and PLC has the fastest working efficiency and optimizes the advantages of the pipetting process.

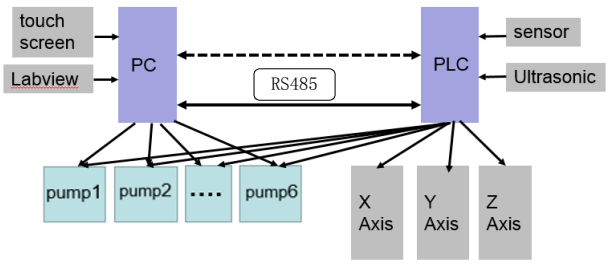


Figure 5. Structure of the distributed control system.

The software system block diagram of the sample preprocessing platform is shown in figure 6, which includes the platform parameter configuration, the compilation of the solution preparation scheme, the host computer monitoring system, and the serial port communication. In addition, the software system has the following functions: (1) Power-on self-test: check the initialization status of the syringe pump and the three-dimensional motion mechanism, and the status of the ultrasonic extraction device, and load the sample preprocessing data information by default. (2) Monitoring: including platform fault, processing progress bar and remaining time. (3) Suspension interrupt and emergency stop: The user can edit another solution preparation scheme during the platform execution process, which is arranged after the currently executed scheme.

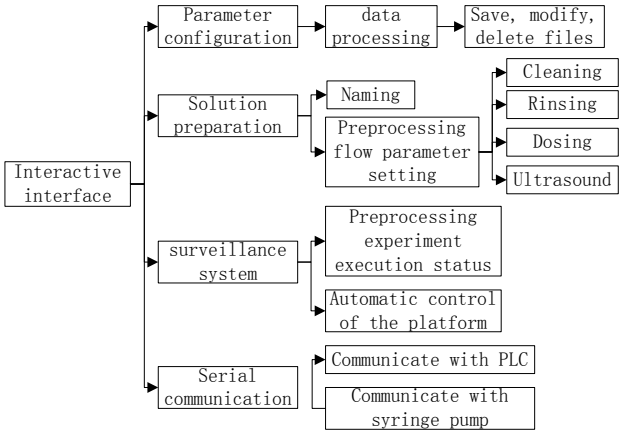


Figure 6. Software framework diagram.

V. Summary

Dissolution is a very extensive sample preprocessing technique that requires extensive solvent preparation. At present, the sample preprocessing technology and its automation instruments at home and abroad have a short development time and have many defects. The sample preprocessing platform designed in our laboratory can be used to prepare 10 solvents and batch processing 96 samples. In addition, the platform features ultrasonic oscillation extraction to accelerate rapid fusion between solvent and sample. The platform has high degree of automation and wide application, which solves the shortcomings of labor force, low efficiency and wide error source brought by manual preparation of solution.

Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Expert Zhiyong Fan puts forward the design requirements of the sample pre-processing platform. Professor Liangen Yang and Professor Tao He are the project instructors, Jingjing Ran is the researcher and wrote this paper, and the other authors are the assistant. All authors had approved the final version.

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