ROBOTIC PROCESS AUTOMATION: ON THE CONTINUITY OF APPLICATIONS DEVELOPMENT AT SOLEIL

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 PIPETTING APPLICATION

Liquid handling is a basic laboratory procedure that is $\frac{25}{5}$

Abstract

For some years now SOLEIL has developed and put into operation robotic applications, using 6-axis robotic arms, to automate some of its beamlines and some processes of magnetic measurements. In the last year, SOLEIL has been working on the development of two new robotic applications, having thus continuity in the development of applications using its robotic standard. This paper describes these two new applications that are being developed to automate the injection of liquid samples for the SWING beamline and to automate the mechanical and magnetic adjustment of the modules that compose an insertion device.

INTRODUCTION

SOLEIL is a synchrotron facility witch offers a large variety of experimental techniques and samples environments to its users, with its 29 beamlines and a wide energy range from THz to Hard X-ray. With the upgrade project, SOLEIL II will provide an increase in brilliance and coherence and all the beamlines will benefit from the unique properties of the new accelerator: measures at the nanometer scale, experiences up to 1,000 times more sensitive and up to 10,000 times faster. These properties will open up experimental new possibilities and also new scientific and engineering challenges to address. Automation is then considered an important piece of the upgrade project in order to achieve the previously mentioned properties.

SOLEIL has spent the last few years working on developing robotic skills to be proficient in process automation and it is especially focused on the arm robot-based automation due to the great advantages that 6-axis robots offer. The specialization includes the definition of hardware interfaces between systems, developing a flexible TANGO-based software interface as well as selecting the Stäubli brand [1]. This standardized approach was designed to guarantee proper implementation, easy deployment and efficient interventions and maintenance for technical staff. The last two robotic applications being developed with the SOLEIL robotic standard are here described. The first application aims to automate the injection of liquid samples for BioSAXS analysis of protein solutions at the SWING beamline. Hence, a pick-andplace 6-axis robot is used to automate the process of sample aspiration and sample dispensing into the measurement cell. The second application, is dedicated to the automation of the mechanical and magnetic adjustment of magnetic modules of the future insertion devices (undulator) of SOLEIL II.

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General Experiment Control

PIPETTING APPLICATION

done regularly across many industries: biological and chemical research, diagnostics, drug manufacturing, food quality control, among others [2]. Liquid handling could be defined as the transfer of pre-determined measured volumes of liquid from one container to another. It can be performed manually by using tools called pipettes or by using automated solutions called liquid handling workstations or simply liquid handling robots, with Cartesian robotic systems¹ being the most representatives of automated liquid handling systems commercially available. However these liquid handling robots are useful only for a limited range of applications, since these are devices integrated in self–contained and rigid workstations with a specified and limited workspace [3]. When Cartesian robots do not meet the application needs, arm robotic systems can also be a solution for liquid handling processes if equipped with suitable pipettes.

Pipettes come in all shapes and sizes, with a variety of different technologies incorporated into their design function. Whether mechanical or electronic, they can handle volumes ranging from a few nanoliters up to milliliters. Electronic distribu pipettes are more precise and accurate than the mechanical ones because they use a motor to control piston movement, so they always dispense exactly the volume programmed. $\vec{\xi}$ Pipetting protocols, including volumes and speeds, can also be pre-programmed and saved so that they are executed in the same way every time [4].

On the other hand, the SWING Beamline allows simultaneous Small-Angle X-ray Scattering (SAXS) and Wide-Angle X-ray Scattering measurements (WAXS) in the 5- 16 keV energy range. SWING beamline helps answering numerous questions related to soft condensed matter, conformation of macro-molecules in solution and material sciences [5]. Currently, the beamline has a fully automated combined ৳ system, including an auto-sampler robot and online High-Performance Liquid Chromatography (HPLC), to carry out the⁻ biological Small-Angle X-ray Scattering (BioSAXS) experiunder ments. Both systems are connected to a quartz capillary cell placed within a vacuum chamber, named in this paper also as the measurement cell, see Fig. 1.

This combined system has worked perfectly on the beamline for more than a decade, however, new scientific requirements have been arisen and nowadays the beamline has to be able to inject the liquid samples as close as possible to the measurement cell in order to avoid wasting sample in the tube that connects the auto-sampler robot to the mea-

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¹ A Cartesian robot has three linear joints (or a combination of them) that use the Cartesian coordinate system (X, Y, and Z).

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Figure 1: SWING Beamline Previous Equipments: the HPLC system, the auto-sampler robot and the measurement cell.

surement cell and eliminate potential cross-contamination with disposable tips. The industrial robot with the electronic pipettes then comes to replace the auto-sampler robot, but preserving the HPLC system to be able to continue changing between the two injection systems 2 .

Process Overview

With the combined system (auto-sampler + HPLC) it is possible either to inject sample ready for immediate SAXS analysis or to inject the sample through an HPLC purification pathway, without manual intervention [6]. In the new pipetting application, this switching between systems is preserved using a robotic tool that allows the HPLC system to be connected/disconnected to the measurement cell when required. Since the measurement cell can move, the experience begins with a calibration stage, where the position of the quartz capillary is determined with respect to the robot frame. Hence, the calibration stage must be carried out each time the measurement cell is moved. Once the HPLC system has been disconnected and the measurement cell position has been calibrated, the sample injection process can take place. The whole injection process can be divided into 2 sub-process or steps and it can be repeated as many times as necessary:

- 1. The sample injection step:
	- (a) Pick up the tool with the sample pipette
	- (b) Take a tip from the sample tips rack
	- (c) Aspirate a defined volume of air
	- (d) Aspirate the sample located at the sample store
	- (e) Dispense the sample into the measurement cell
	- (f) Eject the tip
- 2. The cleaning step:
- (a) Pick up the tool with the cleaning pipettes
- (b) Take a tip from the cleaning tips rack, if necessary
- (c) Aspirate at the same time a detergent solution and water
- (d) Dispense the detergent solution into the measurement cell (washing)
- (e) Dispense the water into the measurement cell (rinsing)
- (f) Eject the tip, if necessary

In general, the capillary has to be cleaned after each sample with a detergent solution to clean the capillary walls and prevent parasite scattering due to the capillary fooling. For this same reason, at each time a sample is aspirated using a new tip to avoid cross-contamination. On the contrary, cleaning the measurement cell is carried out with the same detergent and water solutions, therefore it is not necessary to change the pipette tips every time the capillary is cleaned. Because the volumes needed for the sample and cleaning are different, two pipettes with different capacities had to be selected: the sample pipette has a volume range of 5 – 125 μ l, while the cleaning pipettes have a volume range of $50 - 1250 \mu$. Notice that, since cleaning requires first a washing step and then a rinsing step, it was decided to use two pipettes in the same robotic tool to reduce the execution time of the process. The duty cycle of the process is about 2.8 min including the aspiration, injection, measurement and cleaning.

System Overview

The robotic platform, shown in Fig. 2, is based on a Stäubli TX2-60L robot with six degrees of freedom, which is mounted on a mobile chassis and integrates the Cs9 robot controller, the electric rack, the fluid panel and the input/output modules. This robot was chosen for its radius of work of 920 mm and the load capacity sufficient enough to carry the robot end-effectors.

Figure 2: SWING Robotic Liquid Handling Platform.

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The description of how the HPLC system and the auto-sampler robot work and their characteristics are outside the scope of this paper, but they can be consulted in [6].

Figure 3: SWING Robot Environment.

As it can be observed in the Fig. 3 the sample store is placed next to the tips racks and it has 2 racks with a storage capacity of 96 samples each. The sample store was designed with a cover and a refrigeration circuit to avoid sample evaporation. In addition, it was designed in such a way that commercial consumable racks can be easily integrated without having to modify the design if the supplier changes. There are two racks with tips of different sizes, one rack for the samples tips and the other one for the cleaning tips.

Thus, the rack for the sample tips has a capacity of 384 tips, and the rack with the cleaning tips has a capacity of 96. The containers where the detergent solution and water are stored were also specially designed for this application. Both containers have a capacity of 500 milliliters and a cover to prevent the contamination of the solutions. The cover evidently has a round opening where the tips of the pipettes passes through to aspirate the solutions. A simple level sensor (On/Off) was added to both containers to indicate when the liquid volume is low. In order to remove the tips from the pipettes, a pneumatic gripper is used.

A mechanical interface to adapt the electronic pipettes (VIAFLO) had to be developed so that they could be used by the robot. The electronic pipette brand was chosen in such a way that this adaptation was as simple as possible: once the pipette has been programmed in advance, the robot only "has to press" a single button to execute the program. To accomplish this, the robot's tool is equipped with a electromagnet which is activated when it receives the command from the robot. As indicated in the previous subsection, two robotic tools with two different volume ranges had to be developed: one tool with one pipette for the samples (VIAFLO 125 μ l) **General**

and another tool with two pipettes (VIAFLO 1250 μ l) for cleaning the measurement cell, see Fig. 4.

The measurement cell is mounted on motorized stages with 3 degrees of freedom (XYZ translation), thus the robot must be capable of determining the position of the cell before proceeding to inject the samples. Furthermore, since the robot can be moved between one experience to another (is placed on mobile table), a robust calibration process had to be implemented. Calibration consists of measuring the position of the measurement cell with respect to the robot frame, with the help of a robot's calibration tool. This tool, $\frac{1}{2}$ see Fig. 5, is equipped with a laser sensor (Keyence IL-300) a monochrome camera (Keyence CA200) and a red round multi-angle light (Keyence CA-DRR8M). The laser sensor is used to calculate the depth (X-axis) while the camera is used to calculate the horizontal and vertical displacement (YZ-axis) of the measurement cell. The image processing to calculate the displacement was carried out using the camera controller (Keyence CV-X320F). This controller has a set of image processing algorithms that are relatively easy to implement.

In our case, the position calibration is carried out as follows: first the robot takes the calibration tool and places it Θ in front of the measurement cell. Then, with the laser sensor measures the distance between the laser and the measurement cell to update the X-axis position. Next, a photo is \overrightarrow{S} taken and with an algorithm of shape recognition the distaken and with an algorithm of shape recognition, the displacement of the centroid with respect to a reference image is calculated. Hence, the YZ position of the measurement cell is updated.

Figure 5: Robot Calibration Tool.

Figure 6: Robot HPLC Connecting Tool.

Finally, the last tool that was developed for the application serves to connect and disconnect the HPLC system. It is mainly composed by a stepper motor and a pneumatic gripper that allows screwing and unscrewing the tube that connects the HPLC system with the measurement cell, see Fig. 6. To connect the HPLC system to the measurement cell, once the robot is in the correct position, it begins to screw the tube, by rotating the motor at a constant speed and a given time, then it opens the clamp to be able to move away. To disconnect the HPLC System, the robot closes the clamp to take the tube, following by the rotation of the tube (on the opposite direction) until it unscrews. The HPLC connecting tool is then positioned in its tool holder.

ROBOTIC MAGNETIC MEASURING BENCH FOR INSERTION DEVICES

The purpose of this robotic application is to perform the magnetic and mechanical correction of the future magnetic modules that will compose some of the insertion devices of SOLEIL II. The robotic development is made for the CPMU12 prototype developed in the frame of the Work Package 6 of LEAPS-INNOV program. For this prototype, the previous module system holding magnets and poles [7] and [8] has been replaced by a supermodules accommodating several magnets and poles.

In order to achieve the mechanical adjustment, the robot uses a tool called "screwdriver", see Fig. 7a. The tool is composed by 3 laser sensors Keyence IL-S065 and a screwdriver especially designed for this application composed by: a stepper motor with a resolution of 1.8° per step with a factor 5 reducer, i.e. the final resolution is 0.36° per step, and a screwdriver bit for M1x5 hex socket head bolt. The screwdriver bit is easily removable and it is mounted on a vertical compliance to detect the correct placement of the tip in the screw head bolt. The laser sensors are used to compare the difference in altitude between a pole and the adjacent magnets, see Fig. 7b. Thus one laser sensor measures the altitude of the pole and the other two measure the magnets altitude on each side of the pole at the same time, except for the first and last half poles where only the height of the previous magnet is used.

(a) Robot Screwdriver Tool.

(b) Magnetic Module Figure 7: Magnetic Measuring Bench Components.

The robot measures each of the poles and magnets on the right side of the module, and then does the same measures on the other side, in such a way that at the end, the average of the difference in altitude (at the center of the module) can be obtained for each pole. To reduce robot positioning errors in the Z-axis (altitude), before starting measurements on each side, the laser sensors are calibrated at a previously determined height using a reference piece. Once the altitude difference has been determined, the robot uses the tool's screwdriver to screw or unscrew the poles depending on the difference in altitudes until it is within a tolerance of \pm 10 μ m. It should be noted that this is an iterative process: when the height of the module is adjusted by screwing or unscrewing the pole on one side, the opposite side of the pole becomes unadjusted.

The results of the first mechanical adjustment tests carried out are presented in the Fig. 8.

Figure 8: Mechanical Adjustment Results.

The second functionality developed for this application consists of taking another tool and perform a magnetic measurement with the Hall probe that is mounted on the robot's tool. To execute this functionality, the robot moves parallel to the central axis of the module at a distance from it (Z-axis) that can be adjusted as required (from 1 mm to 5 mm), see Fig. 9. After the magnetic measurement is done, an offset is calculated, and using the same principle of mechanical correction, an adjustment of the poles height is accomplished until the peaks of the magnetic field of all poles are less than 1 %.

Figure 9: Robot Equipped with the Hall Probe Tool.

CONCLUSION

These last two robotic applications developed at SOLEIL are currently in commissioning and it will continue the following months. Up to now, both applications have shown good performance results in the different stages of commissioning, but there is still some automation development to be done at a higher level and tests to need be carried out to verify the robustness of each of the applications.

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REFERENCES

- [1] Y.-M. Abiven et al., "Robotizing SOLEIL Beamlines to Improve Experiments Automation", in *Proc. ICALEPCS'19*, New York, NY, USA, Oct. 2019, pp. 183–186. doi:10.18429/JACoW-ICALEPCS2019-MOPHA001.
- [2] I. Semac *et al.*, "Pipetting Performances by Means of the Andrew Robot", https://www. andrewalliance.com/wp-content/uploads/2017/08/ Pipetting-performances-of-the-Andrew-Robot-A4. pdf.
- [3] H. Fleischer et al., "Analytical Measurements and Efficient Process Generation Using a Dual–Arm Robot Equipped with Electronic Pipettes", *Energies*, vol. 11, no. 10, p. 2567, Sep. 2018. doi:10.3390/en11102567
- [4] Integra, https://www.integra-biosciences.com
- [5] SWING Beamline, https://www.synchrotron-soleil. fr/en/beamlines/swing
- [6] A. Thureau, P. Roblin and J. Pérez, "BioSAXS on the SWING beamline at Synchrotron SOLEIL", *J. Appl. Crystallogr.*, vol. 54, no. 6, pp. 1698-1710, Nov. 2021. doi:10.1107/S1600576721008736
- [7] C. Benabderrahmane *et al*, "Development and operation of a Pr2Fe14B based cryogenic permanent magnet undula-tor for a high spatial resolution x-ray beam line", *Phys. Rev. Accel. Beams*, vol. 20, p. 033201, Mar. 2017. doi:10.1103/PhysRevAccelBeams.20.033201
- [8] M. Valléau *et al*, "Development of Cryogenic Permanent Magnet Undulators at SOLEIL", *Synchrotron Radiat. News*, vol. 31, no. 3, pp. 42–47, May 2018. doi:10.1080/08940886.2018.1460175