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ABSTRACT

The purpose of this report is to provide a detailed description of modular hardware and software design for the automated synthesis of organic materials, specifically protective coatings for electrode materials and currently under construction at Fraunhofer ISC. The system allows for the physical and software integration of heterogeneous single units comprising standard lab equipment like vessels, rotary evaporators and pumping units, providing maximum flexibility and being scalable. The designed system is planned to be the physical part of the planned BIG-MAP fully autonomous platform capable of integrating computational modelling, materials synthesis, and characterization. Therefore a unified network architecture was designed to allow for seamless machine-to-machine communication via intra- and internet.

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1. Introduction

Automated synthesis of organic molecules dates back more than 60 years. Already in 1965, Merrifield demonstrated the automated synthesis of peptides in a linear reactor array.^[1] In recent years process automation in combination with artificial intelligence (AI) based process control has become state of the art for the synthesis of organic compounds.^[2-4] Bédard et al. proposed a plug-and-play, reconfigurable, continuous-flow chemical synthesis system based on membrane technology where the different reaction modules are integrated as individual boards.^[4] The Cronin Group (University of Glasgow) developed a flow synthesis system including a chemical programming language, the so-called *Chemputer*, where synthesis steps are classified into four main activities and sequentially processed.^[2] The *Chempiler*-Software is made available via GitHub (<https://zenodo.org/record/1481731#.XnnZXXbAhnI>). Yet another example of an integrated synthesis was proposed by Chatterjee et al. with the concept of a radial synthesis of organic molecules.^[5]

Despite their versatility, many of these concepts focus on the synthesis of molecules. Due to the limitations in the reaction and purification chambers, the synthesis of more complex polymers is hardly feasible. Integrated systems working with syringes also have issues with clogging of the reaction chamber and the transfer funnels,^[6] making it difficult to upscale these processes to larger batches in the Liter-scale or beyond.

By utilizing standard lab equipment orchestrated by a one-arm robot, it is intended to overcome these limitations and produce large amounts of pre-polymers and polymers in vessels of up to 1000 ml. Additionally, the system is very flexible in operation and allows for the integration of (nearly) any lab-sized equipment to be integrated.

2. Description of the modular synthesis robotics for the synthesis of organic coating materials

2.1 Concept of hardware design

The development described in this deliverable aims to provide a modular robotic platform that allows the synthesis of optimised organic molecules and electrode coatings. Due to frequently changing formulations and the resulting high number of different laboratory devices and equipment, a basic platform expandable via a modular trolley system are basic prerequisites of the design. This ensures that the hardware can be adapted in case the synthesis specifications change. First, device categories are developed, which are then examined in more detail in section 2.3. These categories are handling of the platform's movement apparatus, the laboratory equipment used and its integration into the platform, protective devices and safety measures, and finally, the system control. Figure 1 shows an illustration of the concept study currently installed in the labs of Fraunhofer ISC.

In brief, a robot arm (Yaskawa HC10DT) is located in the centre of the system. Its range is further increased by mounting it on a linear unit. Functional modules on movable carriages can be connected to the sides of this main construction, thus adapting the range of functions for the desired

processes. These functional units can be a rotary evaporator, dosing stations but also liquid extraction units and other generic and self-developed laboratory equipment. Movable venting hoods allow for individual use and thus effectively protect against the release of gases into the environment.

The manufacturing process for the production of hybrid polymers serves as a basis for the development of the individual components. This makes it possible to define the necessary hardware and software requirements and to validate the system after it has been set up. Within the system boundaries, the influencing factors can be alternated for each individual step. This leads to a high number of combinations of the process parameters and thus to the possibility of optimisation. After successful automation, autonomous processing can be directly implemented using the communication standard developed in parallel. In the future, this system will be an important building block for generating an efficient closed-loop materials discovery process.

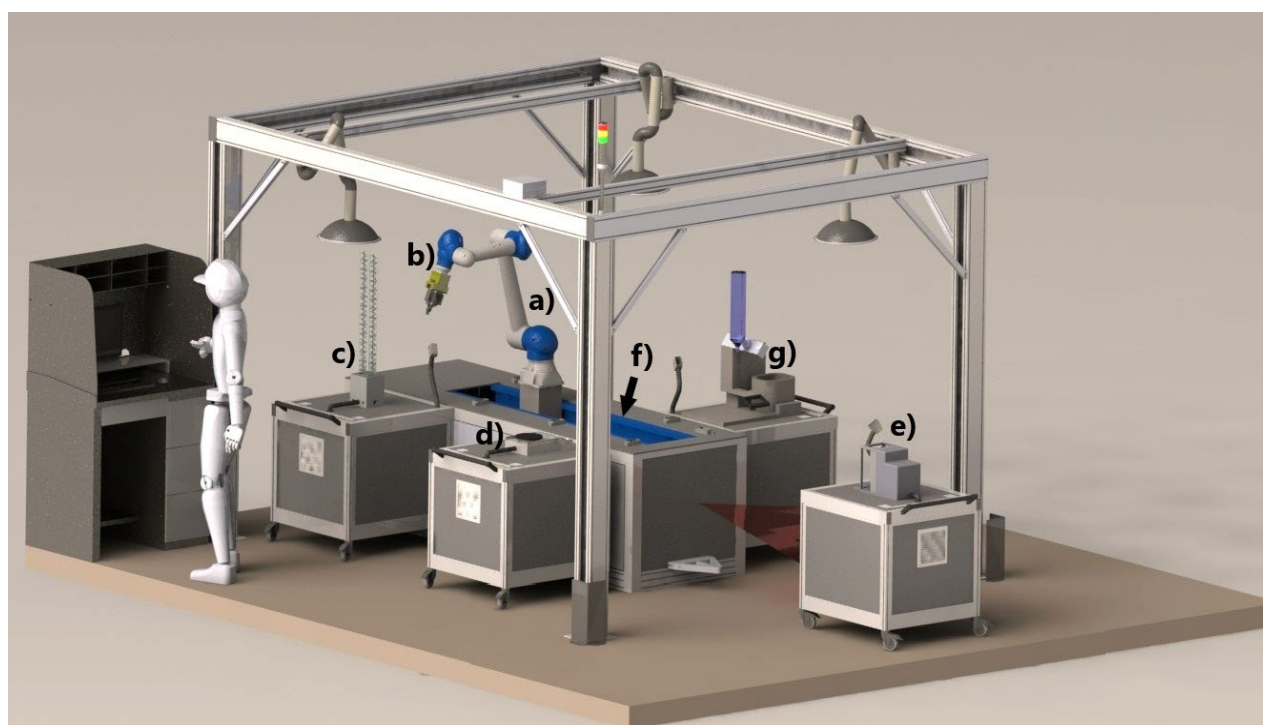


Figure 1. Concept illustration of Fraunhofer ISC setup. Robotic arm (a) installed on a linear axis (f) and equipped with a robotic gripper (b). Liquid-liquid-extraction device (c). Magnetic stirrer with heating plate (d). Precision scale (e). Rotatory evaporator (g).

2.2 Product portfolio of the automated synthesis system

Due to the modularity of the designed system, it will be capable of synthesizing different material classes, given the required lab-periphery provides an API for machine-machine communication. For the purpose of ramp-up and demonstration, a hybrid polymer is selected as a first use case. Inorganic-organic hybrid polymers are a particular class of materials in which inorganic components are mixed at a molecular level with the polyether domains that are necessary for Li-ion conductivity. The highly disordered structure enhances segmental mobility and promotes conductivity. The

polysiloxane/polyether-based hybrid polymer is obtained by a sol-gel technique involving a network-forming component bearing a polymerizable terminal group and a network-modifier (non-polymerizable component), i.e., a polyether-functionalized silane. The formation of the polysiloxane backbone is dependent on the reaction conditions (e.g., humidity, temperature). Small deviations can lead to the formation of slightly different inorganic networks. The structure of this siloxane backbone has a significant impact on the electrochemical properties of the final polymer electrolyte. Reproducibility of the network formation is thus necessary for the consistent quality of the polymers. Therefore, an automated process is planned, and the transfer of the synthetic steps to an automated procedure is the focus of this work.

2.3 Components for planned platform assembly

The planned platform for the production of organic coatings for battery electrodes will require a range of manufacturers and suppliers. In addition, components such as laboratory equipment must have communication functionalities, i.e., suitable interfaces protocols and APIs of the manufacturer. Via these API devices and peripherals can be controlled with the help of a higher-level programmable logic controller (PLC). In addition, resilient, chemical-resistant and inert materials and construction materials are required to ensure consistent production quality.

Even if the platform is to run fully automated to the greatest possible extent and is able to receive parameters and protocol details, human observation and intervention are part of the system design. Therefore, occupational health and safety measures must be considered and conceptualized in such a way that hazards and risks can be clearly identified or even completely eliminated.

2.3.1 6-axis robot arm

A 6-axis robot arm (MOTOMAN HC10DT Yaskawa Electric Corporation, (Figure 1a, Figure 2) serves as a physical link between individual stations or laboratory devices. It performs the handling of instruments and mirrors the manual movements of a laboratory technician. Its high number of axes is required for the handling of lab equipment and interaction with the peripheral machinery. Even fully extended to a reach of 1350 mm, it can carry a load of up to 10 kg at a very high precision of



Figure 2. Image of the Yaskawa MOTOMAN HC10DT IP67, provided by the manufacturer.

0.1 mm. This ensures high reproducibility of the physical motion sequence. In addition, the collapsing function makes it very suitable in close working environments with human colleagues. The HC10DT works in collaboration and direct vicinity to human operators via trained stopping points requiring a very low threshold (e. g. by hand) only. With the help of the linear traversing axis TS100D (2000 mm track length), the working range is further increased.

2.3.2 Gripper hand

To enable the robot arm to interact with its environment, it requires specific actuators, also called grippers hands. The grippers are either operated by pneumatic systems or can be controlled electrically via serial signals using the IO-Link. Specially designed fingers are attached to the grippers so that they can grip either centrally or in parallel in order to provide suitable geometries for the respective area of application. A variety of gripping tools is also needed for a large number of devices. For this purpose, an adapted gripper changing system is used. With the aid of a gripper change adapter, various modules can be flanged to the robot arm.

2.3.3 Modular laboratory trolleys

In order to offer a modular and flexible structure, mobile and individually loadable trolleys are developed. These have defined dimensions and exact attachment points to be coupled to the base/central platform (Figure 3). Three different sizes (single, double, triple) are available in order to accommodate periphery of different sizes. The frames, surface elements and connector systems, as well as the structure of the frame construction above the platform, are supplied by the manufacturer, Item, who is specialized in construction systems that meet the high standards in industrial chemistry. In order to provide the lab periphery with the required media, the trolleys are equipped with multi-purpose connectors (HARTING Technology Group). This specially designed connector system (see Figure 4) over an interface for the supply voltage (power cable), pneumatic energy (air pressure) and the exchange of data packets (Ethernet). For the protection, preparation band correct connection of the pneumatic system, components of the company Festo SE & Co KG are used.

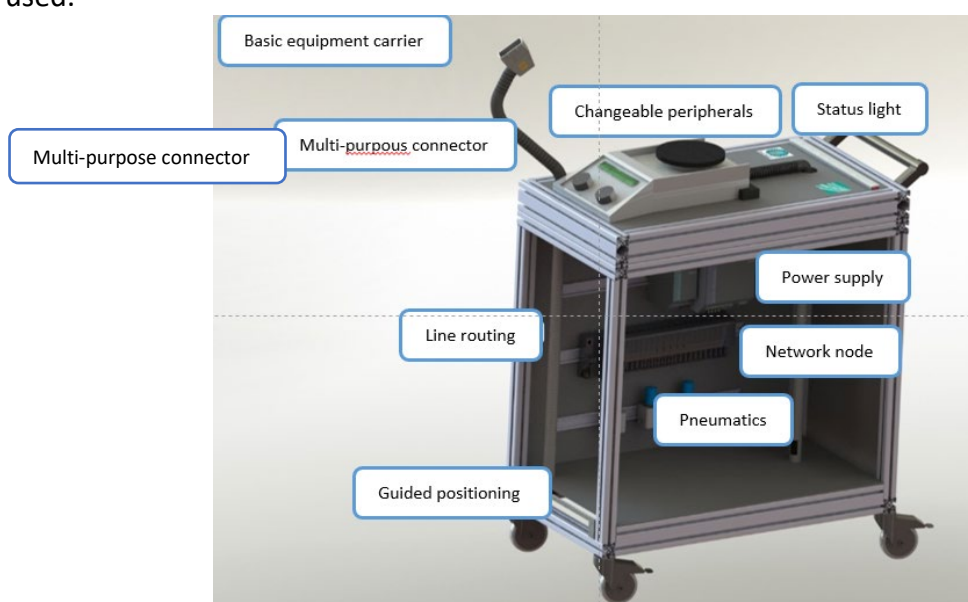


Figure 3. 3 CAD of a modular single-sized trolley to carry a magnetic stirrer.

2.3.4 Lab periphery

Most of the laboratory devices used on the trolleys are largely standard devices that are commercially available. The key feature that must underlie all of them is a suitable interface for communication with the control system. Serial communication is most likely to be with the RS232



Figure 4. Specifically designed plug and socket for the attachment of the trolleys to the central unit.

standard, and for more advanced equipment, a connection with Ethernet is also an option. A heating and stirring device from the company Heidolph Instruments is used, an R-300 rotary vaporizer (including accessories) from Büchi Labortechnik GmbH, peristaltic pumps from Spetec Gesellschaft für Labor- und Reinraumtechnik mbH, precision scales from Mettler-Toledo, pipettes from Sartorius.

2.3.5 Housing

Even though the robot arm allows for direct interaction during operation, the system is cased in an aluminium frame as an additional measure of safety. Various operating states of the platform are visually signalled by means of attached signal lights, emergency stop switches located at several positions ensure a safe operating stop. Motion sensors in the form of light curtains, which delimit and define working areas, help to protect people and machines. In addition, air vents from Nederman GmbH are integrated, which extract a suitably defined amount of air from critical work processes. This protects people and the environment. In addition, the platform is integrated with regard to the exhaust air in the building services.

2.3.6 PLC

As the main coordinator and orchestrator of the processes in a process step chain, a programmable logic controller (PLC) from Siemens is utilized. The PLC used is a controller of the S7-1500 device class, which is characterized by a large number of connectable modules and originates from decades of successful industrial applications of the Siemens Company. In addition, this device class has the function for establishing its own OPC-UA server, which is described in more detail in chapter 5. Building on this PLC basis, adaptations can be carried out safely and quickly. The controllers are also available with various failsafe modules.



2.4 Software involved

The software programs involved are used for the design of the overall system (Solid Works), programming of the controller/PLC (STEP7/TIA-Portal), teaching and programming of the robot (Inform III). Specific custom developments for data processing are written in Python, e.g., for communication between the OPC-UA server on the PLC side and the middleman server for external communication. Modules are controlled via Raspberry Pi OS environment (e.g., Liquid-Liquid-Extraction device).

Solid Works is a 3D CAD program widely used for mechanical engineering, tooling in medical technology and plant engineering. Especially in the field of design, Solid Works plays its strengths and is now used according to the producer of about 1,600,000 developers and designers in the industry and in the field of education around the world. The benefits of using Solid Works for small and medium-sized businesses include speeding up design with absolute accuracy and increasing efficiency.

A suitable programming environment for the development of control systems is offered by the SIMATIC STEP 7 in TIA Portal 7 software developer kit. This implements the following programming languages standardized:

- FBD - Function Block Diagram (formerly FBD - Function Plan)
- KOP - Ladder Diagram (English LD or LAD for Ladder Diagram)
- AWL - Statement List
- S7 SCL (Structured Control Language) Structured Text - a structured high-level language
- S7 Graph (graphically programmable sequential function chart)
- S7 HiGraph, graphical programming via state graphs
- S7 CFC (Continuous Function Chart)

Programs in the instruction list rather correspond to classical assembler programming. Together with SCL, it belongs to the text-based programming languages. All other programming tools are graphical programming interfaces. The linchpin of all operations here is an accumulator, which can work with any data type. All representation forms in the program are created in so-called functions (without state storage) and function blocks (with state storage). This kind of structured programming increases clarity and simplifies future program changes. The reusable blocks also reduce the effort for subsequent projects. Engineering tools for diagnostics and simulation, as well as for parameterization of simple or complex control loops, are available as further options. Simatic controllers programmed with STEP 7 access the same database as Simatic operating and monitoring devices. This consistency, which Siemens calls Totally Integrated Automation, results in advantages for users of Simatic devices for different tasks.

Python is a universal, commonly interpreted, high-level programming language. It promotes an easy-to-read, concise programming style. For example, blocks are structured using indentations rather than curly braces. Python supports several programming paradigms, such as object-oriented, aspect-oriented, and functional programming. It also provides dynamic typing. Like many dynamic languages, Python is often used as a scripting language. The language features an open, community-based development model supported by the non-profit Python Software Foundation, which maintains the definition of the language in the reference implementation CPython.

2.5 Process flow diagram

In order to automate a complex process such as the synthesis of organic polymers of the ORMOCER® family, the first step is to translate the Standard Operating Procedure (SOP) into a flow chart. This allows system requirements to be defined and clearly discussed across disciplines.

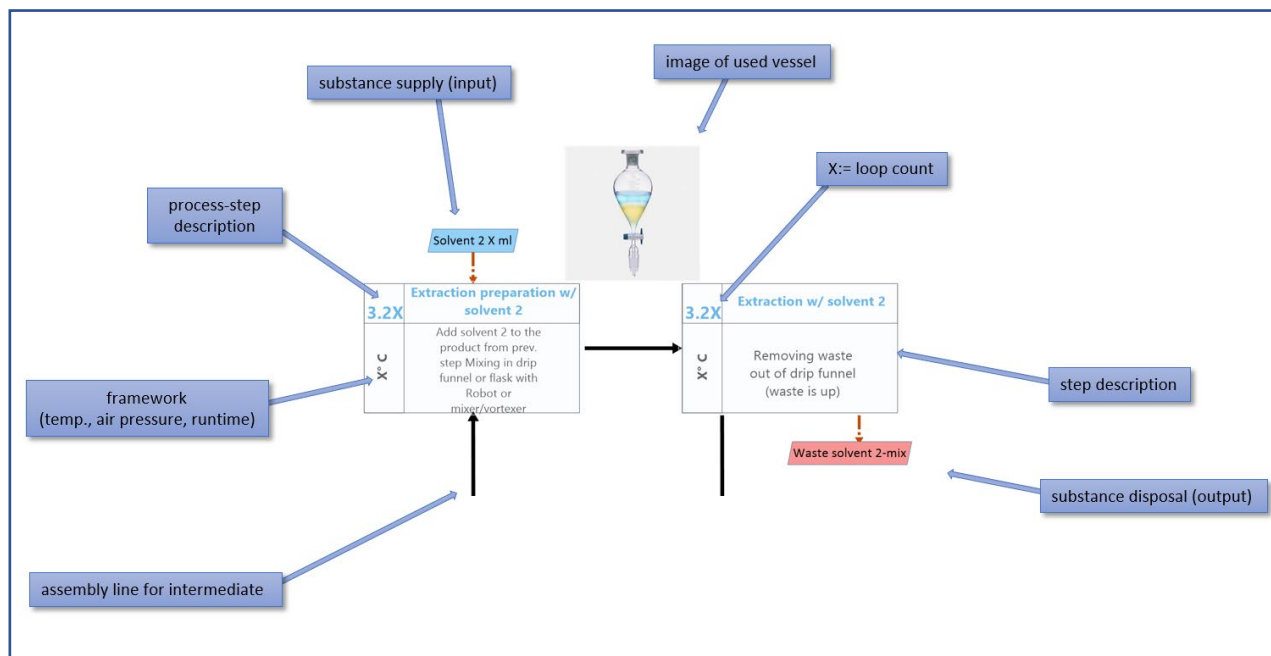


Figure 5. Excerpt of the complete flow chart of the synthesis process.

Figure 5 shows an excerpt of the complete flow chart. To explain the entire process chain, two related process steps are explained in detail. The individual process step is displayed as a box with a variety of parameters. Each step is clearly defined by its consecutive number and title. The parameters of the framework conditions are also defined for each step. The blue and red parallelograms indicate the substances to be added or removed. The reaction vessel to be used can be clearly identified by schematic representation. Subsequently, the different processes are connected with black arrows, which indicate the flow direction of the intermediate products.

3. Hardware operations

3.1 Transport and loading tasks

To cope with the different transport and transfer tasks, different solutions are developed. To implement fine dispensing, up to two different Sartorius pipettes are used. These are controllable to handle volumes with 200 μl up to 5000 μl . The robot grips the pipette with the aid of the correct gripper and can pipette a defined quantity of liquid from a storage vessel with the command from the PLC. From there, the robotized arm can load the chemical as desired. Larger volumes are pumped through a hose system to the desired vessels and laboratory instruments with the aid of peristaltic pumps. The pumps are volume-controlled and can also be linked into a control circuit with the precision scale for even more precise respective high-resolution dosing. The storage of larger stock solutions or solvents and reactants is also realized in the mobile trolleys. These can be filled and closed by laboratory technicians and coupled to the platform.

3.2 Individual processes

Individual processes performed by the robotic platform include:

- **Mixing (Magnetic stirrer)**
The mixing module is built using a commercial magnetic stirrer (Heidolph) remotely controlled through a serial to Ethernet converter; the mixer is placed on top of the modular laboratory trolley so that the robot arm can access it. The mixer receives settings and commands from the central PLC.
- **Separation (Rotary Evaporator)**
The rotary evaporator module is built using a commercial rotary evaporator module remotely controlled through a serial to Ethernet converter and connected to the central PLC. The evaporator is placed on top of the module cart so that it can be accessed by the robot arm.
- **Sampling and testing**
Sampling and testing are performed using pressurized flasks and valves (Festo). As the valves located at the exit of the flasks open, the positive pressure provided by a pneumatic system pushes the liquid through the tubing and to their respective destinations. This keeps the chemicals from coming in contact with other parts of the system that could contaminate them.
- **Extraction (Liquid-liquid extraction LLE)**
For the multiple liquid-liquid extraction steps, a device that automatically mixes solvent and feed detects the interface formed by the liquids and separates them is designed. The main hardware components can be observed in the following Figure 6.

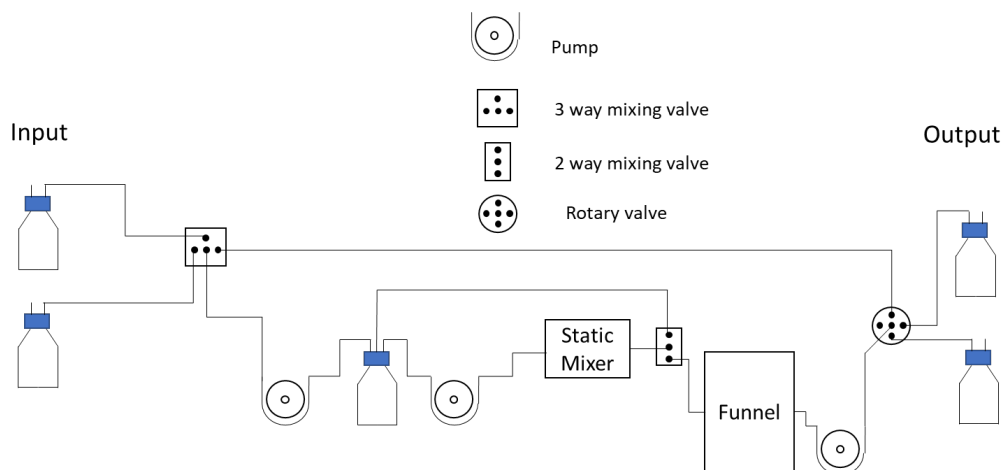


Figure 6. Schematic diagram of the liquid-liquid extraction device.

Components include:

- Three-way mixing solenoid valves (Biochem fluidics – PTFE body)
- Input vessels (Borosilicate with PTFE connection caps)
- Diaphragm pumps (KNF Liquiport, PTFE Body)
- Two-way mixing solenoid valves (Biochem fluidics – PTFE body)
- Mixing stage

- Detection stage: In funnel detection with light sensor
- Dosing pump (Spetec OEM dosing pump with chemical resistant tubing)
- Electric rotary valve (Biochem fluidics – PTFE body)
- Output vessels (Borosilicate with PTFE connections)
- Controller boards (Raspberry Pi 4, Arduino Uno, Arduino Mega)
- Cable connections
- Tubing connections

All components are connected by inert PTFE tubing and the valve and diaphragm pump bodies are also made of PTFE, providing chemical resistance to the solvents used. The dosing pump tubing is also selected to be chemically resistant and inert.

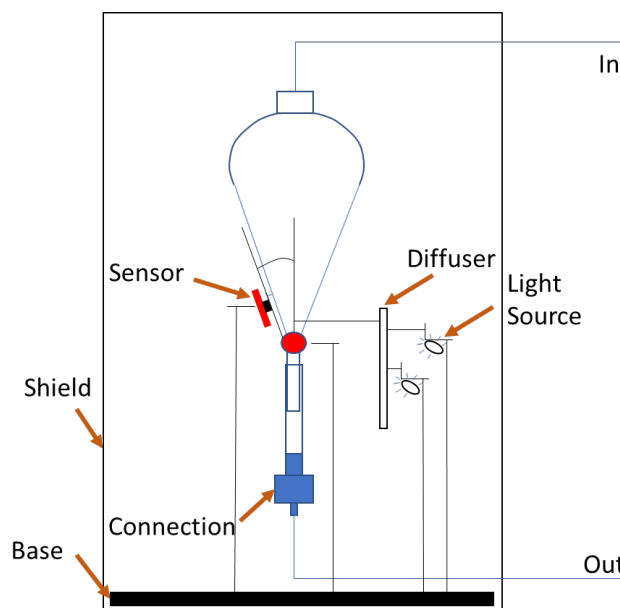


Figure 7. Schematic of the liquid-liquid extractor.

The detection stage (Figure 7) relies on a light sensor capable of detecting light in different wavelengths of the module is composed as follows:

The detection stage consists of:

- 1 Separatory vessel (Borosilicate 2L separation funnel with PTFE connection)
- 1 Light source (Custom LED)
- External light shielding (Custom)
- 1 Near-infrared light sensor (SparkFun Triad Spectroscopy Sensor - AS7265x)
- 1 Flow conductivity probe on the outlet (Custom)
- Tubing connections for the funnel inlet and outlet

Finally, the mixing stage (Figure 8) is composed of a mixing element capable of dispersing the solvent in the feed by reducing its droplet size and thus increasing the surface area between the two liquids. This is achieved by using a pumped static mixer design, in which mixing elements inside a tube shear liquids and make them come in contact with each other.

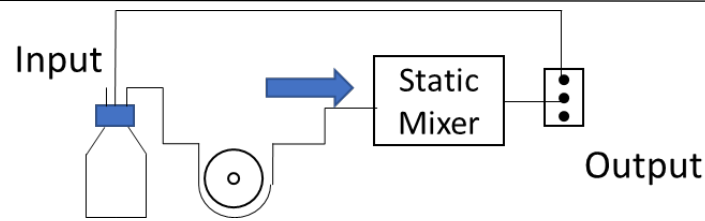


Figure 8. Schematic of the mixing stage.

4. Low-level software architecture

4.1 Robot cell and the robot

For the programming of the robot cell, including the robot, controller, and other peripherals, the units of the platform must be considered separately. Thus, the complex movement patterns of the arm and its position in space are initially taught independently of the planned PLC. The teach pendant can be used to move to the individual points in space, program loops and jobs, trigger outputs such as control signals, create individual coordinate systems and implement similar functions. The teach pendant is connected to the YRC1000 robot controller, which processes data and commands fed in by the user interface. The wired connection to the PLC is established via a fixed Ethernet protocol. In addition to the hardware implementation, the software modules are also programmed and parameterized within the environment of the TIA portal. Here, the step chains for the process are entered and visualized by means of Sequencer Programming with GRAPH (Figure 9). Each of the steps has special transfer conditions, which have freely selectable states so that the next event block with commands can be executed (Figure 10).

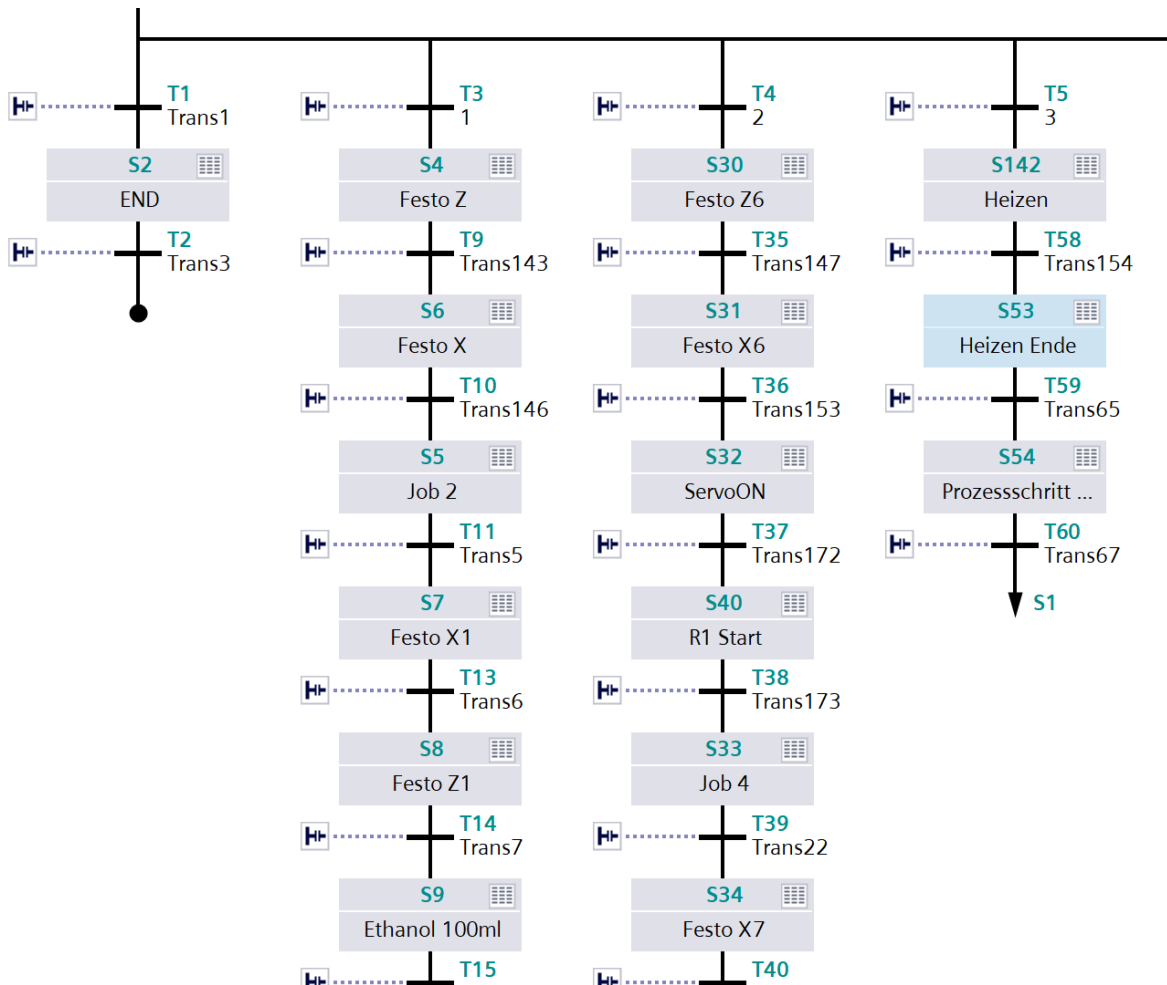


Figure 9. Section of a process step chain from the TIA Portal.

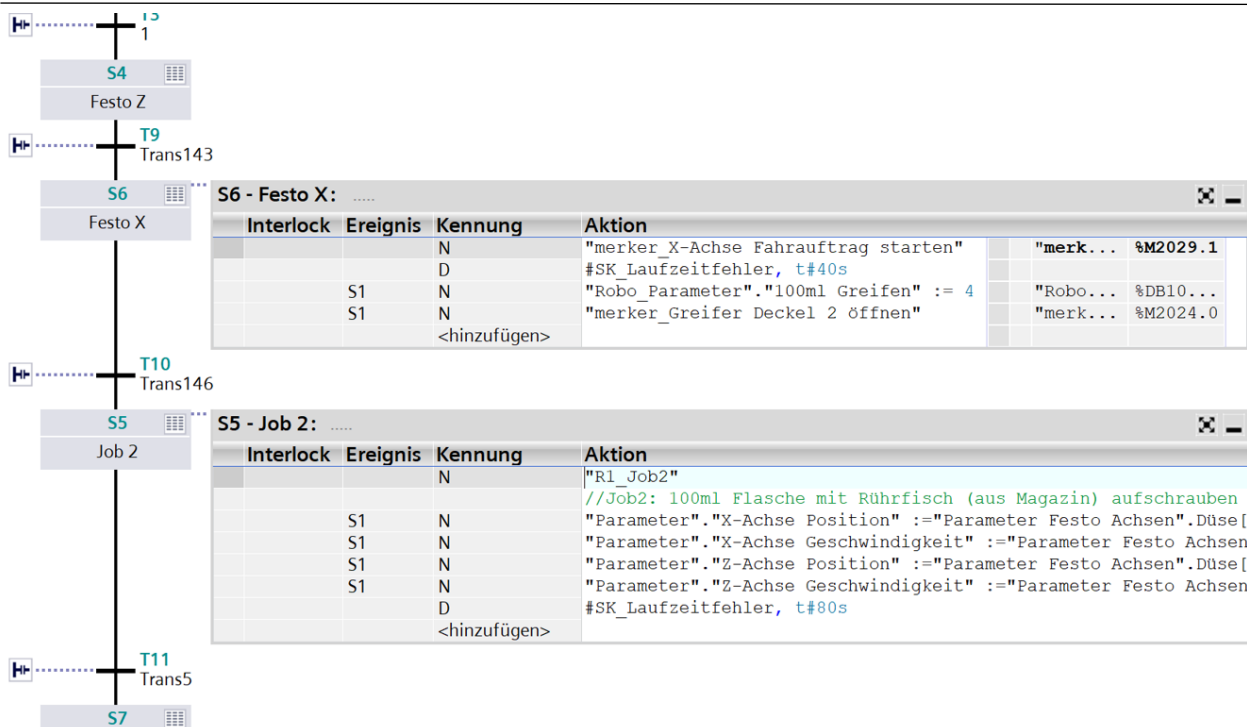


Figure 10. Section of a process step chain from the TIA Portal with open event blocks.

The events called in the block are references to the electronically connected modules of the PLC. As a result, complex function blocks within the controller are addressed and execute their assigned tasks. These references and connections must be suitably programmed to the API of the respective addressed hardware. Instead of programmable blocks that coordinate peripherals directly connected to the PLC, blocks that communicate with the robot controller jobs are also set. This way, defined routines and jobs of the robot controller can be used in the step chain. In the meantime, other programs that are of relevance for certain safety aspects run in a continuous loop, thus ensuring that the entire system is not ready to start in the event of missing inputs.

4.2 Liquid-liquid-extractor

The device is controlled by a central board that implements the OPC-UA server and connects to the OPC-UA software layer. Depending on the type of request to the OPC-UA server, the central board (Raspberry Pi 4) sends commands to two peripheral boards (Arduino Uno and Mega) through serial communication (UART) in order to activate a pump or valve or read a sensor. The communication is implemented in a Main-peripheral architecture, in which the peripheral boards only respond to the commands issued by the mainboard. Commands include a header and information about the parameters of the actuators; peripheral responses include acknowledgments and the sensor data in case of reading a sensor, specific commands include:

- Opening or closing a mixing valve
- Changing the port of the electric rotary valve
- Starting/stopping a pump with a set speed
- Changing the speed of a pump
- Reading the light sensor using serial communication
- Reading the conductivity sensor

The liquid-liquid extraction device control diagram is shown in Figure 11.

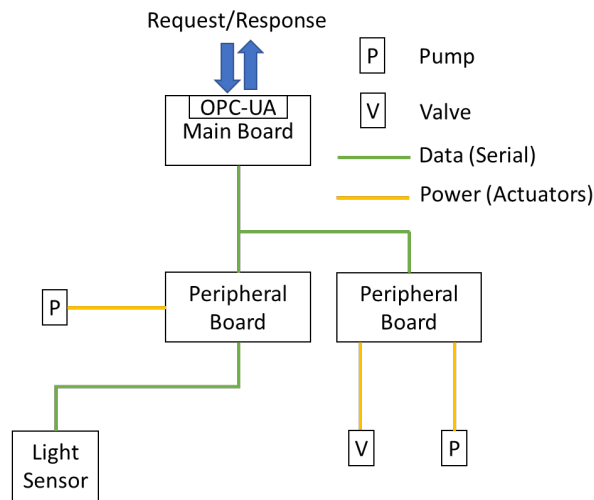


Figure 11. Liquid-liquid extraction device control diagram.

The mainboard and peripheral board programming is done using a combination of Notepad++ and the Arduino IDE (1.8.13), and the Python language and the Arduino language.

5. Communication protocols

5.1 Description of the OPC-UA software layer

5.1.1 OPC-UA and a single deployment

The software infrastructure enables machine-to-machine communication between BIG-MAP partners allowing automated experiments and information exchange. The OPC-UA¹ protocol² is used as a common standard to organize such communication between partners. Available certified³ and non-certified⁴ software development kit SDKs⁵ can be used to implement necessary clients and servers. Having OPC-UA as a common way of communication means that each partner, who would like to interface with the BIG-MAP/WP4 data infrastructure, is expected to operate an OPC-UA server that relays the information from and to connected devices, sensors, and actuators. Thus, allowing for one partner to read data and execute commands on the equipment of the other partner.

¹ <https://opcfoundation.org/about/opc-technologies/opc-ua/>

² <https://reference.opcfoundation.org>

³ <https://www.unified-automation.com/products.html>

⁴ <https://github.com/FreeOpcUa>

⁵ Software Development Kit

To demonstrate the use of OPC-UA servers and clients, several code repositories⁶ have been created with more detailed explanations for each use case. Figure 12 shows a deployment diagram of an OPC-UA application to a partner's server.

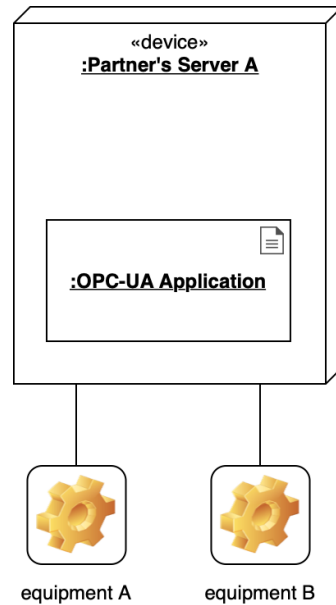


Figure 12. The deployment diagram of an OPC-UA application on a partner's server.

5.1.2 Connecting multiple OPC-UA servers into an infrastructure

While deploying an OPC-UA server in a partner's local network is a straightforward task using any SDK, combining partners' OPC-UA servers into a wider network across multiple local networks requires an additional effort. Thus, to allow inter-partner communication, we introduced a reverse proxy for each partner, which connects to one global proxy server located at IPv4 193.40.22.28 with the domain name of *bigmap.ims.ut.ee*. The global proxy server operates several transport protocols: HTTPS, TCP, UDP, STCP, HTTP. It has a dashboard at <https://bigmap.ims.ut.ee> secured by a login name and password, which allows monitoring of current connections to the global server. Because the global proxy server serves as a point of connection for all partners' servers, and each server takes one port on the server, and the number of ports on a computer is limited, each partner has a pre-allocated set of ports⁷ that it can use.

For a reverse proxy, we use FRP⁸ open-source software. A more detailed description of getting started with the tool and setting up a partner's local proxy is available at the *WP4 Infrastructure* repository⁹.

Figure 13 shows a deployment example for multiple partners with different purposes. Partners A and B serve and consume data using the global proxy UT BIG-MAP server. Partner C only consumes data provided by other partners. Blocks colored in green represent proxy-related artifacts; their only

⁶ <https://github.com/BIG-MAP/wp4-infrastructure>

⁷ <https://github.com/BIG-MAP/wp4-infrastructure#ports-pre-allocation>

⁸ <https://github.com/fatedier/frp>

⁹ <https://github.com/BIG-MAP/wp4-infrastructure#frp-software>

purpose is to relay requests across different partners. Blue components are OPC-UA servers that are responsible for equipment control and monitoring.

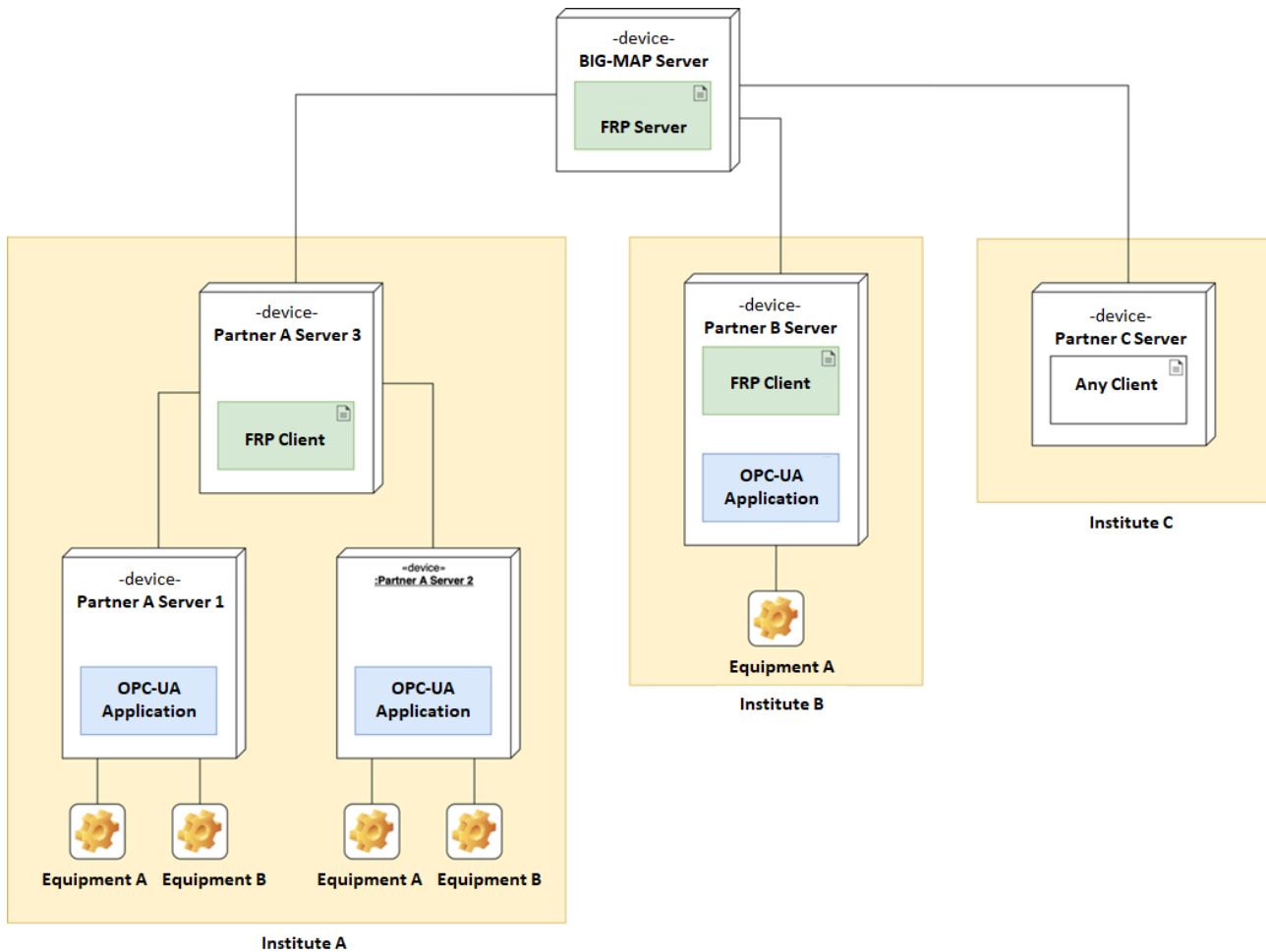


Figure 13. The BIG-MAP/WP4 Infrastructure deployment diagram across multiple partners

Besides OPC-UA, the current architecture also leaves a way of introducing other ways of communication between machines. For example, Partner A can introduce a wrapper JSON-server around its OPC-UA servers, providing an additional post-processing functionality or just to provide another interface for Partner C, who cannot conform to the OPC-UA protocol. This gives us a way of enabling machine-to-machine automated communication based on OPC-UA but still making it possible to interface with any other external data server or consumer. In the case of the robotic synthesis platform, an OPC-UA pre-processing server is used to check requests and manage routines sent to the physical device OPC-UA servers.

5.2 Network architecture

The heterogeneous modules of the robot cell are connected to the PLC via a central switch (Figure 14). So-called interface modules of the respective carriages are realized via the connection with the help of the switch. These form their own subunits of the central controller and control the respective devices on the trolleys (e.g., heating stirrer, dosing unit, liquid-liquid extraction unit, etc.). The interface modules have either additional Ethernet interfaces or specially selected switching modules such as motor control, digital or analogue I/O connections. This creates a hierarchical layered

structure that orchestrates and manages individual programs and commands. With this form of switching network, individual trolleys with their laboratory equipment can be modularly expanded, processed, or replaced.

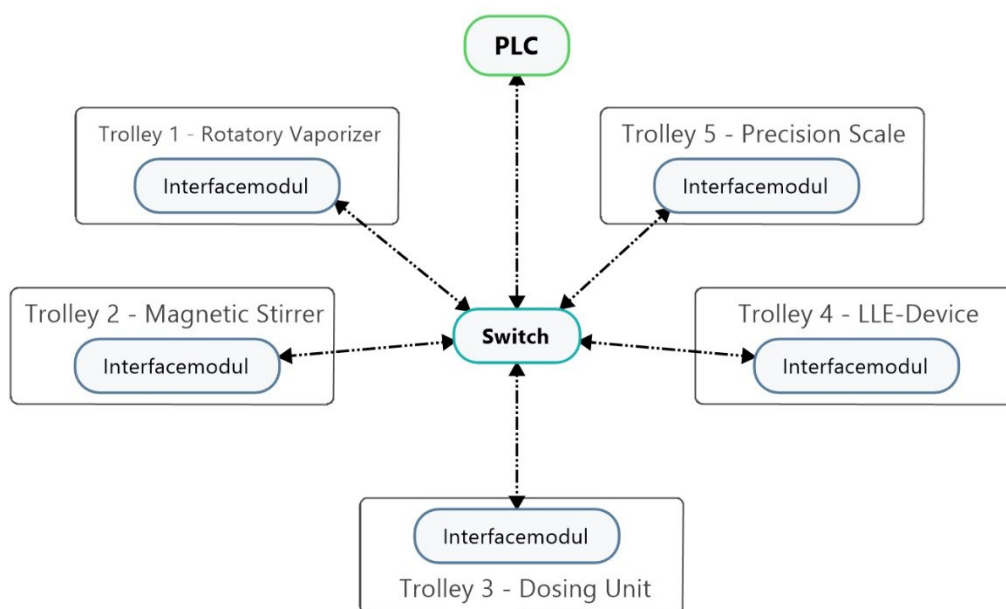


Figure 14. Schematic of the heterogeneous modules connected via central switch to the PLC.

6. Summary

This document presents the design of an autonomous robotic organic synthesis system; the system is composed of modules that perform specific operations or contain specific devices that can be attached to a central unit portraying a robotic arm mounted in a linear axis unit. The finished system will demonstrate the process of synthesizing coatings for battery interfaces in which polyether-based hybrid polymers will be used. The manual process flow diagram and the relevant parameters have been established beforehand.

Based on these, the main operations of the robot system include transport and handling of vessels and thus materials with the robot to the different modules, which perform operations like mixing, using a magnetic mixer, separation, using a rotary evaporator, sampling, and liquid-liquid extraction, using a custom-designed liquid-liquid module.

The platform uses a two-layer software architecture in order to implement it into the BIG-MAP infrastructure. A network of OPC-UA servers, accessible from the internet through a reverse proxy scheme, is used on the top layer. Based on the requests received by these servers, low-level actions and sensor readings are performed by the main controllers. Two main controllers are used: a PLC-based controller, to which different hardware components of the modules are connected, and in the case of the liquid-liquid extraction module, a Raspberry Pi is used as the main controller board.



Both main controllers run OPC-UA servers and control their peripheral boards and hardware using serial communication.

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