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# MIK-I: a portable low-cost platform for automated C–C bond synthesis

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The formation of C–C bonds through automated synthesis platforms aims to accelerate the preparation of molecules, simplify operational processes, and reduce the human time involved in the preparation of molecules. Here, we report different C–C bond formation reactions using a low-cost automated synthesis platform that aims to make automated organic synthesis more affordable. The platform, MIK-I, is capable of performing reactions widely used in organic chemistry, such as Claisen–Schmidt condensations, Suzuki–Miyaura couplings, Knoevenagel condensations, and Baylis–Hillman reactions, with similar yields obtained manually. This result highlights the platform's potential to operate under a basic scheme of synthesis to generate molecules of interest in organic chemistry.

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## Introduction

The C–C bond formation reactions constitute one of the most powerful methods employed in organic chemistry.[1] Developing efficient methodologies to access intricate molecules such as natural products,[2] bioactive molecules,[3] and functional materials[4] is of significance for academic and industrial research.[5] The current demand leads to the proposed novel methodologies that can satisfy on-demand synthesis, including catalytic and organocatalytic processes with operationally simple procedures.[6] Furthermore, the implementation of mild conditions and green chemistry alternatives has become an essential requirement for C–C bond formation.[7]

Although it is possible to find a myriad of C–C bond formation reactions, their preparation is a manual, tedious, and time-consuming process, requiring significant effort.[8] Turning attention to automated synthesis platforms enables parallelization, synthetic reproducibility, and cost reduction.[9] The success of the reactions through automated synthesis platforms depends on several factors, such as the use of building blocks in combination with robust and well-defined chemical reactions, the setup of the machine, and synthesis execution (Fig. 1).[10,11]

Additionally, automation enables precise control over reaction parameters such as drop-by-drop addition, stirring velocity, and reaction temperature, making it useful in optimizing and accelerating the synthesis pace.[12] In this context, Burke and co-workers reported a versatile automated synthesis platform capable of generating C($sp^3$)-rich macrocyclic and polycyclic organic materials and pharmaceuticals through iterative building-block assembly *via* automated C–C bond formation and cyclization reactions[13] and subsequently expanded this block-based strategy to iterative N–C couplings.[14] Complementarily, Aggarwal and co-workers demonstrated that iterative, building-block-based synthesis can also be stereocontrolled, achieving automated C($sp^3$)–C($sp^3$) bond formation under air- and temperature-sensitive conditions.[15] Others have demonstrated the capability of a fully integrated continuous-flow chemistry system to generate C–C bonds by implementing Suzuki cross-coupling and olefination reactions.[16,17] Besides, complex platforms and 3D-printed[18] systems focused on universality and digitization of organic synthesis have demonstrated their ability to execute reactions to form C–C bonds through cross-coupling, Wittig reactions[19–22] and even the reductive coupling of amides.[23] These automated platforms have demonstrated the potential to synthesize

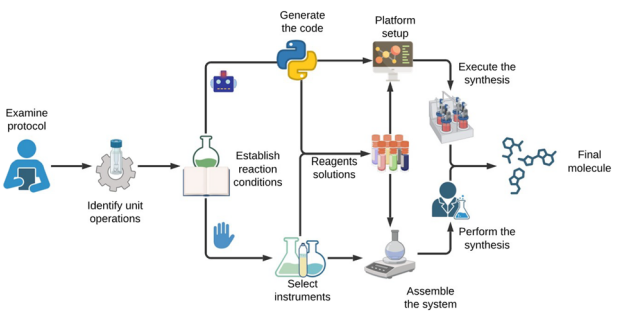


Fig. 1 Conceptual representation of a chemical synthesis process directed towards automation from a traditional approach.

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different types of molecules by combining specific chemistry with machine learning algorithms to yield optimization. However, their accessibility to the scientific community is usually limited due to high costs, the particular materials required for their specialized design, and the engineering involved in their setup.[24,25] This has motivated many research groups to develop affordable, flexible, and low-cost automated platforms for implementation in laboratories for research purposes and to introduce chemists to automation and digitization of chemistry.[26,27]

In this line of thought, we present MIK-I, a portable and low-cost automated synthesis platform designed to perform a range of C–C bond-forming reactions under reproducible and scalable conditions. MIK-I can execute reactions remotely thanks to communication *via* Wi-Fi. To prove the advantage of MIK-I, we decided to execute C–C bond-forming reactions widely used in organic chemistry: Claisen–Schmidt condensations, Suzuki–Miyaura couplings, Knoevenagel condensations, and Baylis–Hillman reactions. All the reactions were carried out in triplicate using the machine to ensure repeatability and reproducibility. Also, MIK-I provides a friendly graphical user interface (GUI) to expand the accessibility of automated synthesis and support its broader integration into academic and research-driven applications.

## Results and discussion

### MIK-I as a low-cost automated synthesis platform

MIK-I, our automated synthesis platform, consists of four main components: (i) a microcomputer (Raspberry Pi 4), (ii) an electronic system, (iii) a series of peristaltic pumps adapted with polytetrafluoroethylene (PTFE) tubing or silicone tubing, and (iv) an integrated friendly graphical user interface (GUI) that allows executing automated organic synthesis without prior programming knowledge. An overview of MIK-I is shown in Fig. 2.

MIK-I works in time intervals controlled by a Raspberry Pi. This system provides an accessible programming interface for managing the platform's components and provides a Wi-Fi connection, which makes it ideal for executing reactions remotely. The electronic system of the platform is primarily comprised of a GPIO extension cable that connects the Raspberry Pi to a breadboard. The breadboard allows the pins to be distributed to a four-channel relay module, an H-bridge module for switching on/off the peristaltic pumps, and a motor for the stirring system. Jumpers connect these modules to the breadboard (see the SI). In the low-cost line of platforms, we decided to use peristaltic pumps as the liquid handling system because one peristaltic pump ($10) is four hundred times cheaper than any commercial syringe pump ($4000). Furthermore, once calibrated, the pumps have the potential to be as precise and efficient as syringe pumps.[28,29] MIK-I operates in a flow/batch system; therefore, all the reagents must be in solutions. Hence, it is necessary to set up the workflow of the pumps, with the flow of the solutions being the unique variable to modify. In this case, to improve the accuracy of reagent addition and minimize flow fluctuations, the entire system is pressurized using a nitrogen balloon. This setup significantly stabilizes the pumping process and improves reproducibility between experiments (SI). All reactions presented in this work have been performed under this pressurized configuration, ensuring consistent reagent delivery and reproducible conditions for all experiments. Although the automation is limited to reagent pumping and stirring control at room temperature, it is sufficient for MIK-I to successfully carry out all the reactions reported in this work. The Raspberry Pi, electronic system, and peristaltic pumps are mounted on a 15.4 × 9.6 × 6.7 in polymethyl methacrylate (PMMA) case (Fig. 2c). All modules of stock reagent solutions can be connected to a 20 mL reactor *via* silicone tubes, which are adapted with needles (end-to-end) for better catch and release of the reagents (Fig. 2).

To provide a more intuitive and user-friendly control, we have designed a graphical user interface (GUI). The GUI has been built using the standard interface tkinter, an importable Python library.[30] The GUI (Fig. 3) is friendly, enabling users to

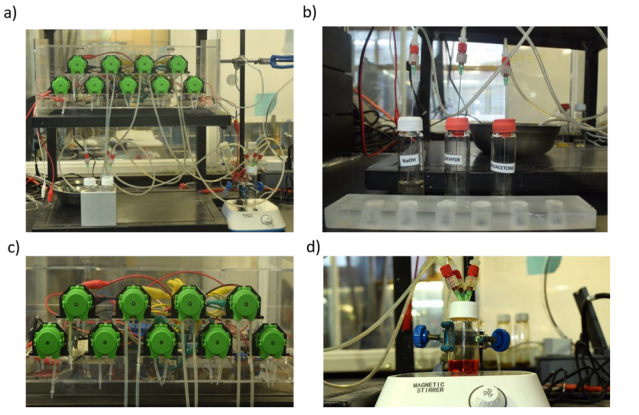


Fig. 2 a) Pictures of MIK-I, a low-cost automated synthesis workflow platform capable of building C–C bonds. b) Solution reagent vials. c) Peristaltic pumps controlled by a Raspberry Pi. d) Reaction vial.

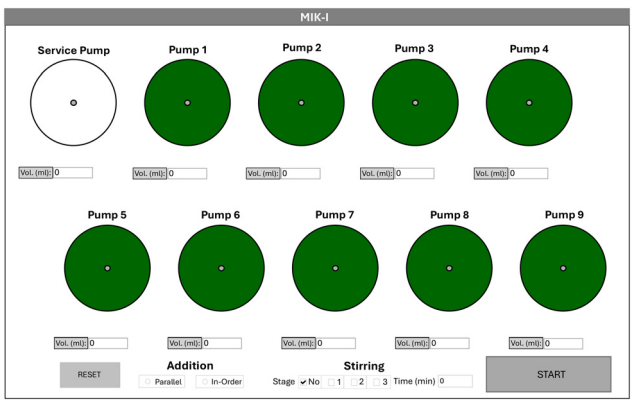


Fig. 3 MIK-I's graphical user interface (GUI) simulates the frontal face of the machine.


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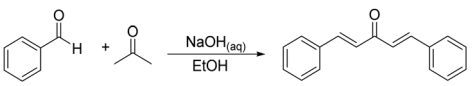
interact with MIK-I without the need to memorize commands or type them. In the SI, there is a guide and a video of the GUI being used and executed. Furthermore, we provide a guide that describes the installation and construction of MIK-I, including a list of components and the total cost.

MIK-I can perform C–C bond-forming reactions such as Claisen–Schmidt condensation, Knoevenagel condensation, Suzuki–Miyaura coupling, and Morita–Baylis–Hillman reaction under normal conditions of humidity and pressure. In this case, the initial synthetic conditions have been translated into Python code to be executed on MIK-I, the automated platform. The platform can carry out reactions with different substrates, producing reproducible results. These observations suggest that low-cost automation can reliably perform basic synthetic operations, providing a practical approach for routine organic chemistry experiments.

## Automated C–C bond-forming reaction approach

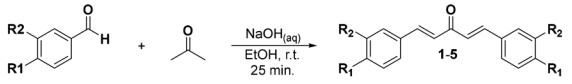
**Claisen–Schmidt condensation.** The Claisen–Schmidt condensation is an extremely useful reaction for the synthesis of diverse molecules inspired by natural products and characterized by the introduction of C═C bonds.[31] Also, the propanone (C═C–C═O) fragment present in the molecules generated during Claisen–Schmidt reactions extends the conjugated system, contributing to the stability of the molecule.[32] This structural characteristic, in combination with the lone-pair electron resonance effect of the oxygen, can enhance light absorption, making the Claisen–Schmidt products ideal candidates to be applied as functional materials in the field of photoelectronics.[33] In this context, we have synthesized five derivatives of the classic dibenzylideneacetone (Scheme 1 and Table 1) using the automated platform MIK-I (see the SI).

The procedure starts when an aqueous NaOH solution is transferred to the reaction vial, followed by an ethanol solution of the corresponding aldehyde, and the reaction mixture is stirred for 5 minutes. Then, 250 mL of water at room temperature is transferred from the water bottle to the crystallizing dish containing the reaction vial using the service pump (a peristaltic pump exclusive to water). The use of room-temperature water helps prevent a rapid increase in temperature. Then, an ethanol solution of acetone is added while maintaining the system at room temperature. Afterward, the reaction mixture is stirred for 25 minutes, during which a yellow precipitate forms. Finally, the product is manually collected by vacuum filtration and washed with water to yield the final yellow product. Detailed spectroscopic characterization of all Claisen–Schmidt products is included in the SI.




**Scheme 1** General synthesis of dibenzylideneacetone.

**Table 1** Comparison of yields obtained *via* the manual procedure and automated synthesis using MIK-I

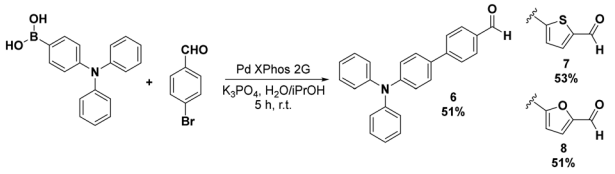



| | *R* | | Yield | | | |
|---|---|---|---|---|---|---|
| | | | | MIK-I | | |
| Molecule | $R_1$ | $R_2$ | Manually | 1° | 2° | 3° |
| **1** | –H | –H | 80% | 78% | 81% | 78% |
| **2** | –H | –F | 65% | 66% | 64% | 64% |
| **3** | –F | –Me | 70% | 71% | 70% | 71% |
| **4** | –SMe | –H | 40% | 39% | 41% | 41% |
| **5** | –OMe | –H | 80% | 78% | 78% | 79% |

The slight variations in yields obtained using MIK-I can be associated with the speed of acetone addition, a step that requires slow (drop-by-drop) delivery. While MIK-I reproduces yields comparable to manual synthesis, the precision of slow reagent addition remains limited compared to commercial syringe pumps. In this case, the issue is resolved by diluting acetone with ethanol, which allows the pump to operate at its normal frequency and ensures the success of the reaction. It is worth mentioning that once the stock solutions are prepared, MIK-I requires about two minutes to prepare and execute each reaction and can repeat the process as many times as needed, consistently yielding the same results (see the video of the reaction in the SI).

**Suzuki–Miyaura coupling.** Suzuki–Miyaura couplings are characterized as one of the most used methods for the formation of C–C bonds.[33,34] The versatility of the Suzuki–Miyaura coupling has made it the perfect candidate for automated synthesis processes.[35,36] Likewise, second-generation palladium complexes facilitate homogeneous catalysis, guaranteeing its execution in almost any automated synthesis platform.[37] Suzuki–Miyaura coupling using MIK-I is performed under soft conditions. The reactions start by mixing a solution of 4-(diphenylamino)phenylboronic acid and a solution of the corresponding bromoaldehyde with a fresh stock solution of Pd XPhos 2G, followed by the addition of an aqueous solution of $K_3PO_4$ as the base (Scheme 2). The reactions are carried out at room temperature, and after 5 h, a precipitate is observed. The solid can be collected by vacuum filtration to afford the corresponding compound (Table 2). Detailed spectroscopic characterization of products is included in the SI.

Although **6**, **7**, and **8** have been reported by others,[38–40] we obtained them remotely using the automated platform (MIK-




**Scheme 2** General procedure for Suzuki–Miyaura coupling in MIK-I.

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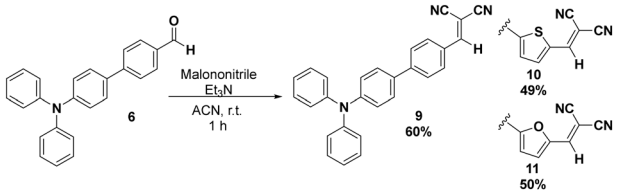
**Table 2** Comparison of yields obtained *via* the manual procedure and automated Suzuki–Miyaura coupling using MIK-I

| | Yield | | | |
|---|---|---|---|---|
| | | MIK-I | | |
| Molecule | Manually | 1° | 2° | 3° |
| **6** | 53% | 52% | 52% | 50% |
| **7** | 55% | 54% | 53% | 53% |
| **8** | 50% | 51% | 51% | 50% |

I), achieving yields comparable to those obtained manually. This result demonstrates the versatility and scope of the platform for routine use in organic synthesis (see the video of the reaction in the SI).

**Knoevenagel condensation.** Knoevenagel condensation, well-known for C–C bond formation, is a reaction that occurs *via* the nucleophilic addition of an active methylene and a carbonyl group of aldehydes or ketones.[41] Structurally, the molecules obtained through Knoevenagel condensation contain alkylidene- or benzylidene-dicarbonyl fragments, a feature that is exploited in polymer production and materials science.[42]

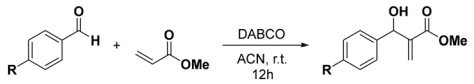
In this context, we synthesized small chromophores derived from **6**, **7**, and **8**, obtained previously through automatized Suzuki-Miyaura coupling (Scheme 2). Automatized Knoevenagel condensation starts when an ACN solution of a corresponding aldehyde and an ACN solution of malononitrile are mixed under basic conditions ($Et_3N$ solution). The reaction mixture is stirred at room temperature for 1 h, forming a precipitate (see video of reaction SI). The final product can be collected by vacuum filtration to afford the corresponding compounds **9** (60%), **10** (49%), and **11** (50%) as shown in Scheme 3. A comparison of yields obtained manually, and automated process are summarized in Table 3. Detailed spectroscopic characterization is included in the SI.




**Scheme 3** General synthesis for Knoevenagel condensation obtained using the MIK-I platform.

**Table 3** Comparison of yields obtained *via* the manual procedure and automated Knoevenagel condensation using MIK-I

| | Yield | | | |
|---|---|---|---|---|
| | | MIK-I | | |
| Molecule | Manually | 1° | 2° | 3° |
| **9** | 63% | 60% | 60% | 61% |
| **10** | 49% | 49% | 48% | 49% |
| **11** | 49% | 49% | 50% | 50% |




**Scheme 4** General synthesis for Morita–Baylis–Hillman reactions using MIK-I.

**Table 4** Comparison of the yields obtained *via* the manual procedure and automated Morita–Baylis–Hillman reaction using MIK-I

| | | Yield | | | |
|---|---|---|---|---|---|
| | | | MIK-I | | |
| Molecule | *R* | Manually | 1° | 2° | 3° |
| **12** | $-NO_2$ | 73% | 73% | 73% | 72% |
| **13** | –Cl | 57% | 55% | 54% | 54% |
| **14** | –OMe | 68% | 65% | 65% | 65% |

**Morita–Baylis–Hillman reaction.** As a final example of the C–C bond-forming reaction, we decided to explore the Morita–Baylis–Hillman reaction. This reaction has been established as an important C–C bond-forming reaction between carbonyl-containing compounds and an activated alkene, catalyzed by a nucleophilic Lewis base.[43] The applicability of this reaction lies in the commercial availability of the starting materials, its high atom economy, its lack of a metallic catalyst, and its operation under mild conditions.[44] Furthermore, the resulting Morita–Baylis–Hillman adducts can be used as building blocks for the synthesis of natural products, biologically active compounds,[45] and functional molecules.[46] All the considerations mentioned above make the Morita–Baylis–Hillman reaction a suitable candidate for automating using MIK-I.

The reactions are performed at room temperature, and the chemical transformation begins when a solution of aldehyde in ACN is mixed with a solution of DABCO in ACN, followed by the addition of a solution of methyl acrylate in ACN (Scheme 4). The reaction yield obtained using MIK-I is very similar to that achieved manually (Table 4), highlighting the potential of this platform for the synthesis of building blocks to generate molecules of interest.

## Conclusions

In summary, we demonstrated that MIK-I can synthesize a series of molecules **1–14** through C–C bond-forming reactions with reproducibility and yields comparable to those obtained using manual procedures. The results support the reliability of performing classic C–C reactions through a simplified operational workflow managed through a graphical user interface. Owing to its modular architecture and remote operational capability, MIK-I highlights the potential of user-friendly and cost-effective automation for routine reaction execution. We believe that platforms such as MIK-I will contribute to expanding automated organic synthesis in academic laboratories for high-throughput molecular discovery, functional material design, or biological interest. By lowering financial and technical barriers, this approach provides a scalable and adaptable alternative to current commercial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solutions that may facilitate the adoption of automation in synthetic research laboratories with limited resources.

## Materials and methods

Detailed materials and methods can be found in the SI.

## Author contributions

A. A. G. designed the concept, devised the project, wrote part of the manuscript, and helped run the team. M. A. F. O. designed the automated synthesis platform (MIK-I), E. R. L. L. identified the synthetic targets and carried out all the associated synthesis. R. A. G. M. wrote the manuscript and associated SI with help from M. M. F. L., who contributed to the initial idea and wrote some codes to automate the preliminary synthesis and optimization.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

The code for running the automated synthesis can be found at https://github.com/aag224/MIK-I-Cheap-Platform.

The version of the code employed in this study is Python 3.9.

Supplementary information (SI): MIK-I platform design, communication protocol, automated synthetic procedures, experimental details, and $^{1}$H and $^{13}$C NMR spectra. See DOI: https://doi.org/10.1039/d5re00447k.

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