

Efficient Solid-Phase Organic Synthesis of Prochiral Schiff Bases from Asymmetric Ketones on Merrifield Resin

Síntese Orgânica Eficiente em Fase Sólida de Bases de Schiff Pró-quirais a partir de Cetonas Assimétricas sobre Resina de Merrifield

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A solid-phase organic synthesis protocol is presented for the preparation of asymmetric Schiff bases derived from prochiral ketones using functionalized Merrifield resins. Due to the lower electrophilicity of ketones compared to aldehydes, solid-phase imine formation from ketones remains scarcely reported. In this study, 1,2-ethylenediamine was covalently attached to Merrifield resins with chloride loadings of 1.4 mmol/g and 3.5–4.5 mmol/g, followed by condensation with the asymmetric ketones in anhydrous tetrahydrofuran. Reaction conditions were systematically evaluated by varying solvent, temperature (25–70 °C), and reaction time (1–24 h). While reactions at room temperature afforded low yields (20%), reflux at 70 °C for 5 h significantly improved conversions, affording resin-supported prochiral imines in yields ranging from 90 to 99%. The formation of the C=N bond was confirmed by Fourier-transform infrared spectroscopy (1634–1650 cm⁻¹), and fluorescence monitoring indicated that imine formation approached completion within 5 h under reflux conditions. The solid-phase organic synthesis methodology eliminates the need for chromatographic purification, as products were isolated by vacuum filtration followed by drying at 50 °C. This approach provides a practical and reproducible route for the preparation of solid-supported prochiral imines with potential applications as precursors of chiral ligands and functional materials.

Keywords: Schiff bases; solid-phase organic synthesis; prochiral.

1. Introduction

In organic chemistry, the preparation of pure compounds with high yield remains a significant challenge, as traditional purification methods often lead to product loss and reduced yields.¹⁻³ Among the most accessible functional groups for chemical manipulation are primary amines, aldehydes, and asymmetric ketones, which, under appropriate anhydrous conditions, undergo condensation to give imines (Schiff bases), characterized by a C=N bond.⁴⁻⁶ While imine formation from aldehydes and primary amines typically proceeds smoothly under mild conditions, reactions involving asymmetric ketones demand stricter dehydration protocols and often suffer from lower conversions due to steric hindrance and electronic stabilization of the carbonyl group.⁶⁻⁸

Solid-phase organic synthesis (SPOS) is a technique in which soluble reagents in a reaction solvent become covalently or non-covalently attached to a polymeric support.^{9,10} These solid supports vary in chemical composition depending on the nature of the base polymer and the linker. Typically, a reactive leaving group as chloride is displaced by a nucleophile, which becomes covalently bound to the polymeric support. Two of the most widely used polymeric resins in SPOS are Wang¹¹⁻¹⁵ and Merrifield¹²⁻¹⁶ resins. Both of them are composed of polystyrene with crosslinked divinylbenzene; the main distinction lies in the structure of the linker, as the reaction is performed at this section of the polymer. These materials typically feature a defined loading capacity, expressed in millimoles per gram (mmol/g) of reactive sites of the resin.

One of the major challenges in SPOS is the characterization

of the resulting supported compounds, as conventional techniques such as proton and carbon nuclear magnetic resonance (NMR) are often not applicable due to the insolubility of the resin in common solvents. To address this limitation, fluorescence spectroscopy¹⁵⁻¹⁸ and Fourier-transform infrared (FT-IR) spectroscopy¹⁹ have been successfully employed. These techniques are indispensable and widely used to characterize materials prepared by this method. A key advantage of these polymer-supported systems is their absorption in the UV-visible region, which facilitates fluorescence measurements. Additionally, FT-IR spectroscopy can be conveniently used, either with potassium bromide (KBr) pellets or attenuated total reflection (ATR) techniques, to monitor functional group transformations during resin modification.

In recent years, SPOS strategies have been successfully applied to the preparation of imines and Schiff base derivatives supported on polymeric matrices. Several studies have reported the functionalization of Wang and Merrifield resins with primary amines, followed by condensation reactions to obtain resin-bound imines with potential applications in catalysis, metal coordination, and sensing materials. These supported systems offer advantages such as simplified purification, improved reaction control, and facile handling compared to solution-phase methodologies. Despite these advances, most reported approaches focus predominantly on aldehyde-derived imines, while examples involving asymmetric or prochiral ketones remain considerably less explored. There are previous reports of amine-functionalized resins serving as precursors for the solid-phase synthesis that could be employed to prepare imines derived from asymmetric ketones. However, reports on the solid-phase synthesis of prochiral imines using ketones are limited, due to the lower reactivity of ketones toward nucleophiles such as amines, which is an issue that becomes more pronounced when reactions occur on polymeric supports such as Wang or Merrifield resins.^{19,20} The aim of this study is to optimize the reaction conditions, including time and temperature, for the synthesis of new Schiff bases derived from Merrifield resin with loadings of 1.4 mmol g⁻¹ (denoted as **m**) and 3.5–4.5 mmol g⁻¹ (denoted as **M**).

2. Materials and Methods

2.1. Reagents and characterization techniques

All reagents were purchased from a local distributor of Sigma-Aldrich (St. Louis, MO, USA) and were used without further purification unless otherwise stated. Only tetrahydrofuran (THF) was dried over metallic sodium with benzophenone as an indicator. The other reagents were

used without further modification or purification. THF was selected due to its consistent performance under anhydrous conditions. In the following, in all the experimental procedures, the term “equivalent” refers to the molar ratio relative to the amount of reactive functional groups present on the Merrifield resin (loading expressed in mmol/g). This definition is used to determine the stoichiometric amounts of ketone and 1,2-ethylenediamine employed in the coupling reactions with the resin.

FT-IR spectroscopy measurements were performed using a Perkin Elmer Frontier FT-IR/FIR spectrophotometer. Samples were prepared as KBr pellets for the FT-IR analysis. Fluorescence spectroscopy studies were performed using a Perkin Elmer LB-50 spectrofluorometer. Prior to fluorescence measurements, the absorption maximum of the functionalized resin was determined, resulting in a wavelength of 339 nm; thus, this wavelength was selected as the excitation wavelength (λ_{ex} = 339 nm). Emission spectra were recorded in the range of 350–700 nm under flux cell conditions for solid samples.

2.2. Synthesis of chelating resin

The two Merrifield resins employed in this study, denoted as **M** and **m**, differ in their functional group loading. Resin **M** exhibits a higher chloromethyl loading (3.5–4.5 mmol g⁻¹ of resin), whereas resin **m** has a lower loading (1.4 mmol g⁻¹ of resin). This variation is primarily influenced by the crosslinking degree of the resin, which directly affects swelling behavior, reagent diffusion, and the accessibility of reactive sites within the polymeric matrix. The higher loading (**M**) allows for increased functional density, which can be advantageous for maximizing total product per gram of resin. However, the lower-loading resin (**m**) often resulted in better reproducibility and purity of the supported products, possibly due to minimized steric hindrance and more efficient diffusion of reagents into the polymeric matrix.²¹

Both resins were tested in this work under identical conditions to evaluate performance, and the results are discussed accordingly. Before initiating the Schiff base synthesis, the bidentate chelating amine 1,2-ethylenediamine (denoted here as **e**) was coupled to the two different resins **m** and **M**. For this, 100 mg of resin, were first suspended in 5 mL of anhydrous THF with 3 equivalents of K₂CO₃ to neutralize HCl formed in the reaction. Subsequently, 5 equivalents of 1,2-ethylenediamine were added. The reaction mixture was subjected to mechanical stirring for 24 h at room temperature. After completion of the reaction, the product was washed with distilled water (three times using 10 mL of water) to remove excess K₂CO₃, followed by THF washes (twice with 15 mL of THF) to eliminate residual

amine. This resulted in the formation of two chelating resins, denoted here as **Me** and **me**. Volhard titration was performed resulting in a 100% substitution in the resins in one protocol.¹⁶ The scheme for the amine functionalization of **M** and **m** with **e** to obtain **Me** and **me** is presented in Figure 1.

The FT-IR spectra of the precursor Merrifield resins (**M** and **m**) and the corresponding amine-functionalized materials (the chelating resins **Me** and **me**) are provided in the Supporting Information for direct comparison. Upon amine functionalization, the characteristic C–Cl stretching band of the chloromethylated resin significantly decreases, while new N–H stretching bands appear, consistent with the substitution of chloride sites by the ethylenediamine moiety. Although FT-IR analysis does not allow strict quantitative determination of complete conversion, the observed spectral changes support efficient amine incorporation under the reported conditions.^{16–19}

2.3. Schiff base synthesis derived from chelating resins and 2-fluorobenzaldehyde

After preparing the amine functionalized resins (the chelating resins **Me** and **me**), the optimal conditions for imine formation derived from an aldehyde were determined by reaction at different times: 1, 3, 5, 7, 9, and 24 h at room temperature. The reaction, schematized in Figure 2, was performed as indicated in the following lines. First, 1 equivalent (100 mg) of the chelating resin, **Me** or **me**, was stirred in 10 mL of anhydrous THF for 5 min to allow swelling. Subsequently, 5 equivalents of 2-fluorobenzaldehyde were added as the aldehyde substrate, along with 3 equivalents of sodium sulfate (Na_2SO_4) to facilitate water removal and drive the equilibrium toward imine formation. After the studied reaction time, the product was washed and filtered under vacuum using 10 mL of anhydrous THF (twice) followed by 10 mL of distilled

water (twice). The solid was filtered under vacuum and dried overnight in an oven at 50 °C prior to the spectroscopic analysis.

2.4. Efficient approach of Schiff base preparation derived from chelating resins and asymmetric ketones

Once the protocol for imine synthesis from 2-fluorobenzaldehyde was established, the experimental conditions for synthesizing imines starting from ketones were optimized as well. For this purpose, an amount of the chelating resin, **Me** or **me**, corresponding to 1.0 equivalent of amine groups, was suspended in 10 mL of anhydrous THF and reacted with 5 equivalents of the following prochiral ketones: methyl phenyl ketone (**a**), methyl isobutyl ketone (**b**), 4-methoxyacetophenone (**c**), and 4-nitroacetophenone (**d**); additionally, 3 equivalents of sodium sulfate (Na_2SO_4) were used as a desiccant. The reaction progress was monitored by solid-state fluorescence spectroscopy. The fluorescence emission spectra were recorded ($\lambda_{\text{ex}} = 339 \text{ nm}$; emission range 350–700 nm) at each selected reaction time (1, 3, 5, 7, 9, and 24 h), and the decrease in emission intensity associated with imine formation was used to evaluate reaction progress. Once the reaction was completed, a yellow solid product was obtained, which was washed and filtered under vacuum using 10 mL of anhydrous THF (twice) 10 mL of distilled water (twice). The solid was then dried overnight in an oven at 50 °C for subsequent spectroscopic analysis. In this way, eight different prochiral imines derived from **Me** and **me** using the mentioned prochiral ketones were obtained: **Mea**, **mea**, **Meb**, **meb**, **Mec**, **mec**, **Med** and **med**. The reaction scheme illustrating the synthesis of these prochiral imines is shown in Figure 3. The previous reaction methodology was performed at different temperatures to evaluate the effect of this parameter: 25, 40, 50, 60, and 70 °C.

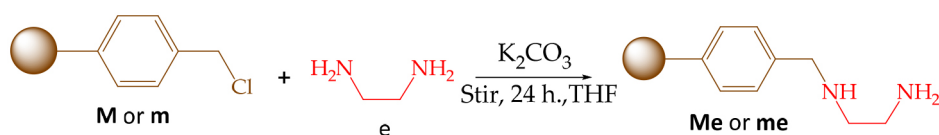


Figure 1. Scheme for the amine functionalization of the Merrifield resins **M** and **m** with **e** to obtain the chelating resins **Me** and **me**¹⁶

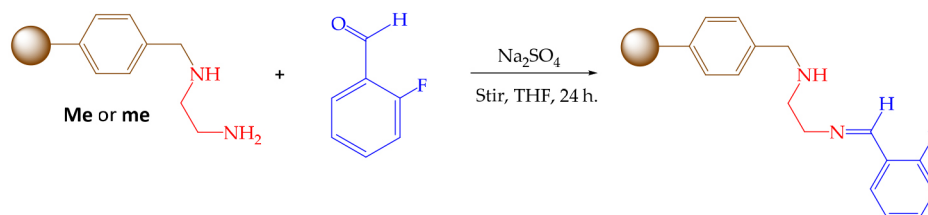


Figure 2. Scheme for the imine (Schiff base) synthesis from the reaction of chelating resin **Me** and **me** with 2-fluorobenzaldehyde

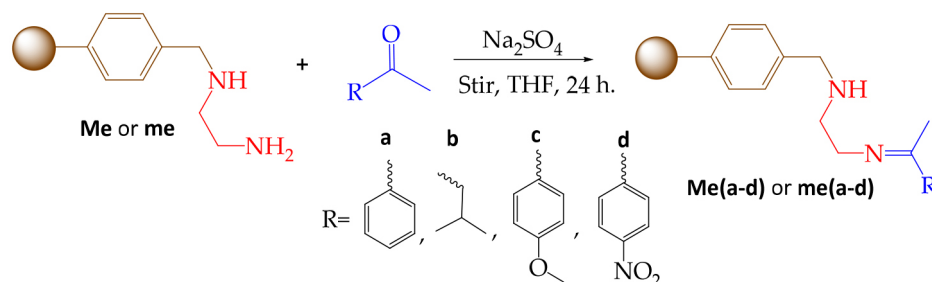


Figure 3. Scheme of the imine (Schiff base) production by solid-phase organic synthesis from the reaction of chelating resin **Me** and **me** with different prochiral ketones

3. Results and Discussion

3.1. Determination of optimal conditions

Once the synthesis method of the Schiff base from the chelating resin and 2-fluorobenzaldehyde was established, the reaction was found to proceed efficiently, reaching completion after 8 h of stirring. The formation of the Schiff base derived from 2-fluorobenzaldehyde was confirmed by FT-IR spectroscopy, as observed in Figure 4, through the appearance of the characteristic C=N stretching band of imine at 1643 cm^{-1} and the complete disappearance of the primary amine N–H stretching upon imine formation at $3500\text{--}3350\text{ cm}^{-1}$, indicating completion of the reaction. On the other hand, the reactions derived from ketones are not completed at 25°C . This behavior is consistent with the higher electrophilicity of aldehydes compared to ketones in condensation reactions. Since reactions derived from ketones performed at room temperature (25°C) afforded low conversion (20%), the temperature was systematically increased to 40, 50, 60, and 70°C . Optimal conversion was achieved under reflux conditions at 70°C . This tendency is in agreement with the well-documented lower reactivity of ketones relative to aldehydes toward nucleophilic attack by primary amines in Schiff base formation.^{22,23} The

reaction yields with the mentioned ketones were calculated gravimetrically by weighing the dried products. The eight imines coupled to resin (**Mea**, **mea**, **Meb**, **meb**, **Mec**, **mec**, **Med** and **med**) were obtained in better yields, ranging between 90 and 99%, as presented in Table 1. It should be noted that gravimetric yield determination in solid-phase synthesis may be affected by residual solvent or physically adsorbed species. In this work, the resins were thoroughly washed and dried prior to weighing, and the formation of the imine functionality was independently confirmed by FT-IR spectroscopy. Therefore, the reported values should be considered as apparent gravimetric yields of the functionalized resin.

In addition to THF (the solvent originally included in methodology), dichloromethane (DCM) and *N,N*-dimethylformamide (DMF) were evaluated under identical reaction conditions during the optimization study. Although all three solvents enabled imine formation, THF provided more consistent performance under strictly anhydrous conditions and was, therefore, selected for subsequent experiments. The differences observed among the tested solvents may be rationalized considering the swelling behavior of polystyrene-based resins. It is well established that solvents such as THF, DCM, and DMF can penetrate the polymeric matrix, promoting internal

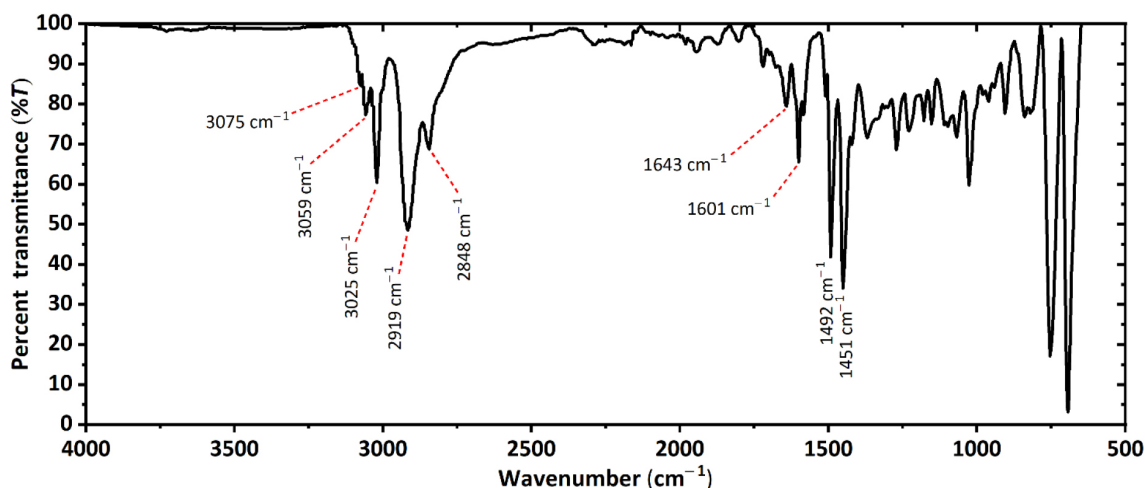


Figure 4. Fourier-transform infrared spectrum of the imine formed from **me** with 2-fluorobenzaldehyde

Table 1. Yields and positions of the Fourier-transform infrared signal corresponding to the C=N stretching band for the prochiral Schiff bases obtained by solid-phase organic synthesis

Resin	Color	Substituent	Yield (%)	C=N Stretching (cm ⁻¹)
mea	yellow	phenyl	99	1635
Mea	pale yellow	phenyl	98	1635
meb	pale yellow	isobutyl	90	1656
Meb	pale yellow	isobutyl	90	1654
mec	yellow	4-methoxyphenyl	95	1634
Mec	yellow	4-methoxyphenyl	90	1639
med	brown	4-nitrophenyl	99	1638
Med	brown	4-nitrophenyl	96	1640

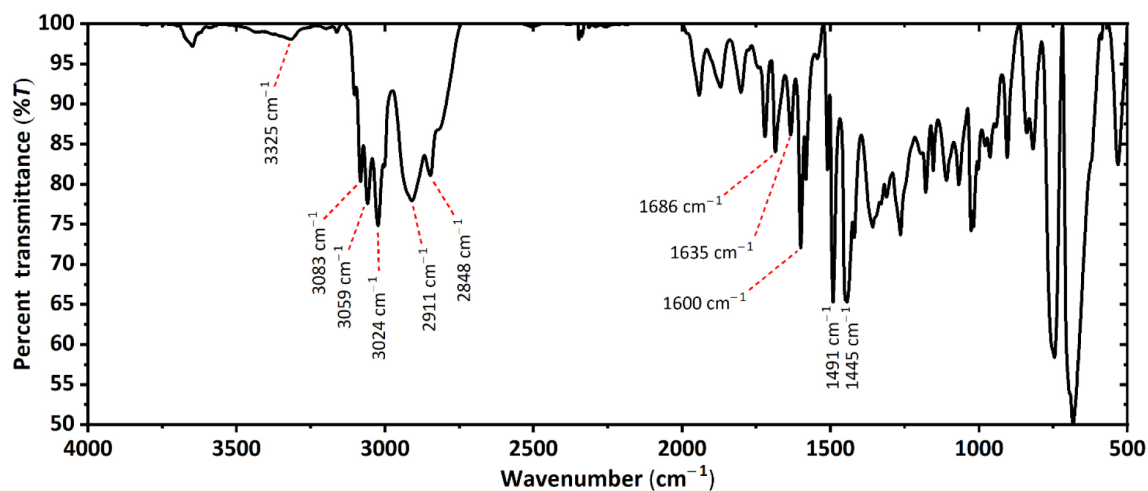
Yields and FT-IR signals correspond to the average of three independent reactions performed under optimized conditions (THF, 70 °C, 5 h).

expansion and increasing the accessibility of reactive sites, a phenomenon known as resin swelling.²³ Enhanced swelling improves reagent diffusion within the support and facilitates imine formation. Among the synthesized compounds, one of the best yields was obtained for **med** (99%) and **Med** (96%). This result is attributed to the electrophilic character of ketone **d** due to the presence of an electron-withdrawing nitro group, which promotes efficient imine formation. A comparable, though slightly lower yield, was observed for **mec** (95%) and **Mec** (90%), which contain a methoxy substituent. Notable changes in the appearance of the polymeric material were observed depending on the substituents of the aromatic ring. For instance, **Mea** and **mea** such as **Mec** and **mec** exhibited a yellow coloration. Photographs of the supported materials are provided in the Supplementary Information (Figure S1), while **Med** and **med** developed a more intense orange-brown color. These changes contrasted with the original light-yellow color of the chelating resin, which became increasingly intense upon product formation. The second-highest yield (90%) was obtained for **meb** and **Meb**, which are the least reactive of

the four, since, being aliphatic, its reactivity to the nitrogen nucleophile in the reaction decreases further.

3.2. Spectroscopic analysis

Characterization was carried out by FT-IR spectroscopy, where the characteristic imine C=N stretching band should be observed in the 1650–1634 cm⁻¹ region depending on the substituent in the prochiral Schiff base. For example, the formation of the imine **mea** was confirmed by the appearance of the C=N signal at 1635 cm⁻¹, as shown in Figure 5, which is consistent with the positions of the same signal observed in the other obtained prochiral Schiff bases (presented in the Supplementary Material). The positions of the C=N stretching band for the eight prochiral Schiff bases obtained by solid-phase organic synthesis are also presented in Table 1. The minor variations observed in the C=N band position can be attributed to substituent effects as well as conjugation and matrix influences that are inherent to polymer-supported systems. In comparison with the precursor resin (**me**), the different spectra of the

**Figure 5.** Fourier-transform infrared spectrum of the imine **mea**

prochiral Schiff bases (Supplementary Material) clearly shows the disappearance of the primary amine N–H features ($3500\text{--}3350\text{ cm}^{-1}$) involved in condensation. However, a residual N–H stretching band, located at 3325 cm^{-1} for the **mea** example (Figure 5), which corresponds to the secondary amine of the ethylenediamine spacer covalently anchored to the polymeric support, remains in the recorded spectra. The spectra were also examined in the carbonyl region ($1680\text{--}1715\text{ cm}^{-1}$), where aromatic and aliphatic ketones typically exhibit a strong, sharp C=O stretching band. If physically adsorbed ketones are present in significant amounts within the resin, detectable absorption in the spectra would be expected. However, no signal attributable to residual carbonyl groups, according to the characteristic shape of this signal (strong, sharp) was detected (after the washing and drying procedure). However, FT-IR alone does not allow definitive assessment of purity. It is important to note that aromatic C=C overtones, which appear at $2000\text{--}1665\text{ cm}^{-1}$, do not overlap with the C=N stretching region ($1650\text{--}1634\text{ cm}^{-1}$); additionally, these overtone bands are typically weaker and broader, making them easily distinguishable from the C=N band. In contrast, the imine C=N stretching band is generally more intense and sharper.¹⁹ Therefore, the signal at 1635 cm^{-1} , based on both its higher intensity and more defined band profile, is more consistent with the characteristic C=N stretching vibration of the imine rather than with aromatic overtone contributions. Additionally, reaction progress was independently monitored by fluorescence spectroscopy, providing complementary evidence for imine formation under the optimized conditions.

Fluorescence spectroscopy analysis was performed, and changes in intensity and shifts of the fluorescent band were observed as the reaction progressed at $70\text{ }^{\circ}\text{C}$ at

different reaction times (Supplementary Material). Each of the prepared imines followed the pattern observed in the previously conducted reactions at a reaction time of 24 h (Supplementary Material). In contrast to the chelating resin **me**, which exhibited significant increases in fluorescence intensity, the imine **mea** displayed a different behavior characterized by a decrease in fluorescence intensity over time is consistent with imine formation. The observed changes in fluorescence intensity during the reaction are consistent with the transformation of the amine-functionalized resin into the corresponding imine. Fluorescence spectroscopy is widely employed as a powerful spectroscopic tool to monitor transformations occurring on polymer supports and classical synthesis.^{24,25} Figure 6 illustrates this decline in fluorescence intensity during the transformation from amine to imine at reaction times of 1, 3, 5, 7, 9, and 24 h under reflux at $70\text{ }^{\circ}\text{C}$. Fluorescence spectroscopy monitoring of the imine formation on the functionalized resins revealed that the most significant changes in fluorescence intensity occurred within the first 5 h of reaction at $70\text{ }^{\circ}\text{C}$. Beyond this point, variations in both intensity and band position were minimal, suggesting that the reaction approaches completion early in the process. Notably, while the chelating resin **me** exhibited increased fluorescence upon imine formation, the imine **mea** showed a decreasing trend, consistent with the consumption of the amine moiety. Considering the negligible spectral changes after 5 h, extending the reaction time to 24 h provides no substantial benefit. Therefore, a 5-h reaction time is optimal, allowing for reduced energy consumption and increased throughput without compromising product quality. It should be noted that, unlike Wang resin which contains an acid-labile linker allowing cleavage and isolation of the product with a hydroxyl group, the Merrifield resin employed in this study lacks such a spacer. Consequently, detachment and subsequent structural

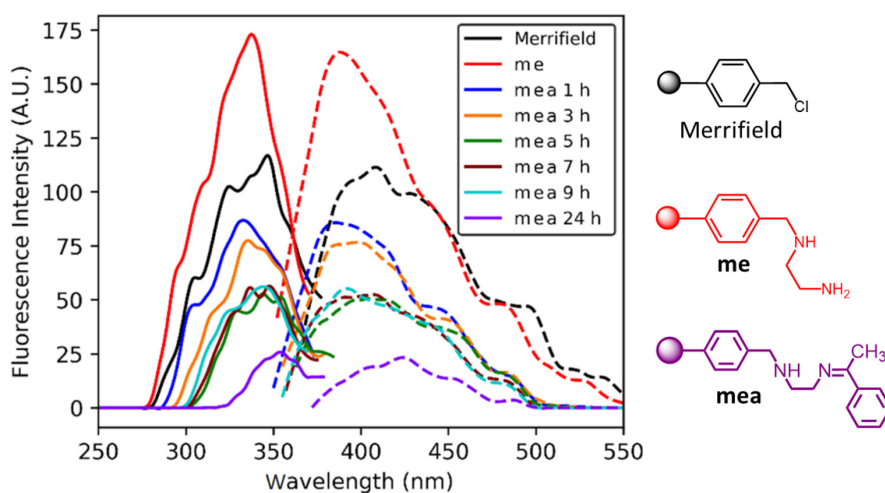


Figure 6. Fluorescence emission spectra of **mea** obtained at $\lambda_{\text{ex}} = 339\text{ nm}$ (emission range $350\text{--}700\text{ nm}$). Reaction performed under reflux at $70\text{ }^{\circ}\text{C}$ at different reaction times. Solid line corresponds to excitation and dash line to emission

characterization of the Schiff base via NMR and MS were not feasible within the scope of this study.

4. Conclusion

This work describes an optimized solid-phase organic synthesis (SPOS) protocol for the preparation of resin-supported prochiral imines derived from asymmetric ketones on Merrifield resins with chloromethyl loadings of 1.4 mmol g⁻¹ and 3.5–4.5 mmol g⁻¹. Reaction conditions were systematically evaluated by varying the solvent, temperature, and reaction time to identify suitable parameters for efficient imine formation from asymmetric ketones. Among the solvents tested (THF, DCM, and DMF), THF provided consistent and reproducible performance under anhydrous conditions and was therefore selected for subsequent reactions. Temperature screening demonstrated that reflux at 70 °C significantly improved conversion compared to reactions performed at room temperature. Four prochiral imines were successfully prepared on each resin support. Resin **m** exhibited consistent spectroscopic profiles and reproducible yields. Reaction progress was effectively monitored by fluorescence and FT-IR spectroscopy, enabling determination of the optimal reaction time. Minimal spectral changes were observed after 5 h at 70 °C, indicating that imine formation was essentially complete within this timeframe. Thus, a 5-h reaction time represents a practical balance between efficiency and resource consumption. The developed methodology provides a reliable and reproducible approach for the preparation of solid-supported prochiral imines and establishes a foundation for future studies involving chiral ligands, coordination systems, or functional materials based on polymer-supported imine platforms.

Supplementary Information

Supplementary Information containing FT-IR spectra, fluorescence spectra, and additional experimental details is available free of charge at the journal website <https://rvq.s bq.org.br/pdf/AR2026-5117-MS>.

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Declaration on the Use of AI Writing Tools

The authors declare that AI-assisted tools were used exclusively for language editing and improvement of clarity. All scientific content, experimental design, data analysis, and conclusions were developed and critically verified by the authors.

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