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Synthesis of some new substituted imines from aldehydes and ketones derived from quinolinic acid

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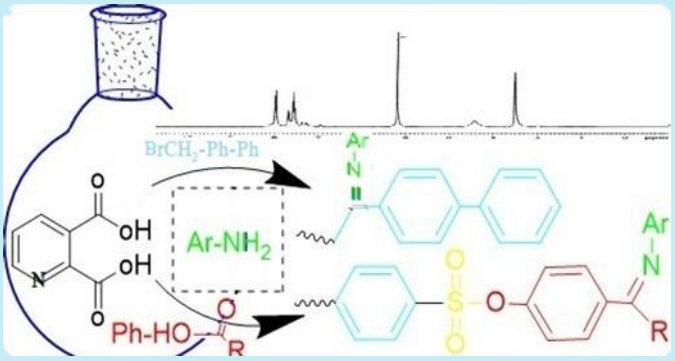
ABSTRACT: In this paper, some substituted imines compounds have been prepared from quinolinic acid as a starting material. Firstly, the quinolinic acid was treated with acetic anhydride and acetic acid to form furo[3,4-b]pyridine-5,7-dione (1); the resulting compound was heated with urea to form 5H-pyrrolo[3,4-b]pyridine-5,7(6H)-dione (2). After that, it was treated with potassium hydroxide to give potassium 5,7-dioxo-5,7-dihydropyrrolo[3,4-b]pyridin-6-dione, which was directly and easily converted to 6-(2-([1,1'-biphenyl]-4-yl)-2-oxoethyl)-5H-pyrrolo[3,4-b]pyridine-5,7(6H)-dione (3) by the reaction with 1-([1,1'-biphenyl]-4-yl)-2-bromoethan-1-one. Finally, the resultant compound reacted with substituted aniline to give imines (4, 5). Secondly the quinolinic acid converted to 4-(5,7-dioxo-5,7-dihydro-6H-pyrrolo[3,4-b] pyridin-6-yl) benzenesulfonyl chloride according to our previous work, then treated with p-hydroxy acetophenone or p-hydroxy benzaldehyde to form 4-substituted bezyloxy 4-(5,7-dioxo-5,7-dihydro-6H-pyrrolo[3,4-b] pyridine-6-yl) benzenesulfonate (6, 7), which were finally treated with substituted aniline to form new substituted imines (8–12).



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1. Introduction

In recent years, imines have been the millstone of chemist research because they were easy to prepare and get various reactions. They can be prepared simply by the reaction of primary amines with carbonyl compound (usually aldehydes or ketones) in the presence of acid as catalyst. The functional group of these compounds is azomethane group C=N. Due to the electronegativity of the nitrogen atom, this group has four different reactions. Firstly, electrophilic, secondly nucleophilic, thirdly dienophile, and lastly, aza-diene reaction (Fig. 1) (Choudhury and Parvin, 2011).

There is a big similarity between carbonyl and the imine groups. Therefore, their reaction is very similar, but the reactivity of the carbonyl group is higher than the imine group because the electronegativity of oxygen is greater than nitrogen. To increase the reactivity, Lewis's acid was used as a catalyst in imine reactions (Chan *et al.*, 2019).

The imine compounds exhibit a wide range of useful biological activity, such as inflammatory, antimalarial, analgesic, antioxidant, antimicrobial, anthelmintic, antitubercular and anticancer (Fig. 2) (Bashiri *et al.*, 2020; Hania, 2009; Jasril *et al.*, 2020; Kajal *et al.*, 2013; Silva *et al.*, 2016).

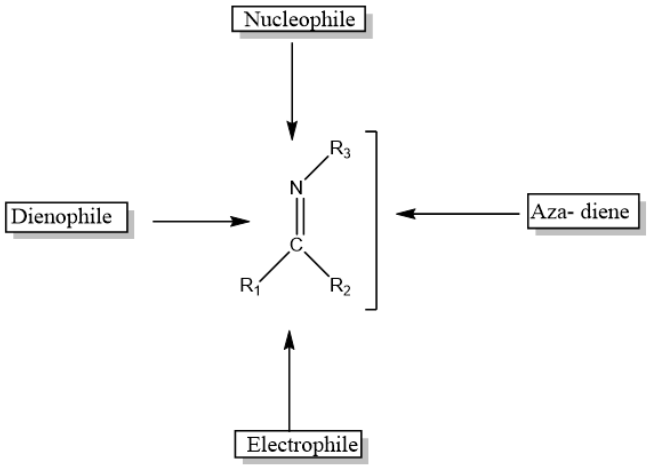

Figure 1. Imine reactivity.
Source: Retrieved from Choudhury and Parvin (2011).

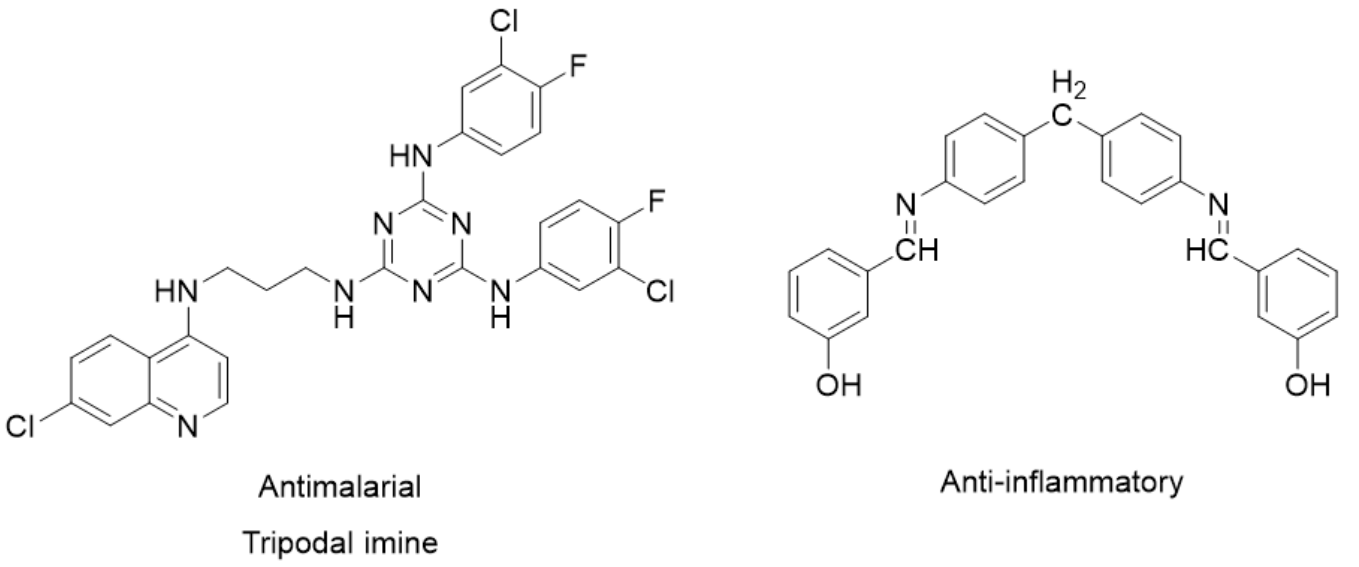

Figure 2. Some of the bioactive imine compounds.
Source: Adapted from Bashiri *et al.*, (2020) and Kajal *et al.*, (2013).

2. Experimental

All chemicals and solvents were purchased from commercially available known sources and used directly without more purification. IR spectra (*V*max in cm^{-1}) were verified utilizing a Shimadzu FT-IR 8400 spectrophotometer with KBr disc. ^{1}H-NMR Bruker at 300 MHz, using dimethyl sulfoxide-d^{6} and tetramethylsilane as a standard.

2.1 Synthesis of 5H-pyrrolo[3,4-b] pyridine-5,7(6H)-dione (2)

Furo [3,4-b] pyridine-5,7-dione (1) was prepared from quinolinic acid (Rapolu *et al.*, 2019) (0.15 mol, 9 g) from anhydride (1) was mixed with (0.3 mol, 2 g) of urea in 100 mL round and heated to (130 to 135 °C) with shaking for (10–20 min) until the reaction was noticed increasing in size. After this point, the product cooled to

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room temperature, then (10 mL) of water was added and mixed to destroy the amide (2); then, filtrate and recrystallisation from ethanol to give white crystal 83% (188–190 °C), IR, 3104, 1725, 1620, 1391 cm^{-1} (Cai, 2012).

2.2 6-(2-([1,1'-biphenyl]-4-yl)-2-oxoethyl)-5H-pyrrolo[3,4-b] pyridine-5,7(6H)-dione (3)

First step: Imides (0.01 mol, or 1.4 g) were dissolved in 20 mL of absolute ethanol, and the mixture was then heated in a water bath with a clear solid obtained. Ethyl alcohol was added to potassium hydroxide solution of 0.01 mol of KOH in 25 mL of pure ethanol while stirring and letting it cool. The precipitate that formed was then filtered and dried. The resultant compound was used in the next reaction without any further purification; 73% broke down at 230 °C.

In the second step, 0.01 mol (2.7 g) of 4-phenyl phenacyl bromide was dissolved in 25 mL of absolute ethanol in a round-bottomed flask. Then, 0.01 mol (1.8 g) was added. Imide salt, which was prepared in the first step, was added slowly while stirring. The resultant mixture was heated and stirred for 6 h, then cooled to room temperature. The precipitate was filtered, washed with distilled water, dried, and then recrystallized from ethanol to give a brown powder. 60% (120–124 °C), IR, 3104, 1715, 1680, 1676, 1620, 1391 cm^{-1}, 1HNMR δ 8.3–7.3 (m, 12H), 4.9 (s, 2H) (Fig. S1 – Supplementary Material) (Aliabadi *et al.*, 2013).

2.3 General procedure for synthesis of Schiff bases (4–5, 10–12)

For 6 h, 35 mL of absolute ethanol and 2-3 drops of glacial acetic acid were added to 0.01 mol of carbonyl compound and 0.01 mol of primary aromatic amine while stirring at a refluxing temperature. After cooling the reaction mixture to a room temperature, the precipitate was separated from the solvent by filtration, dried, and recrystallized from ethanol Fig. 3 (Altaee and Al-Sabawi, 2021; Yin *et al.*, 2020).

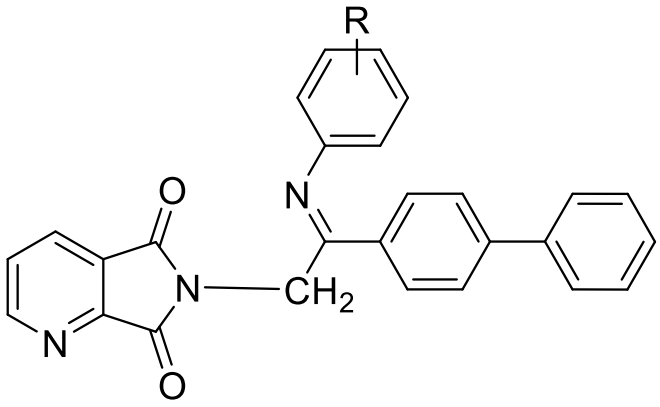

Figure 3. Substituted Schiff bases.

Compound 4 (R = P-hydroxy) is a yellow powder yield in 81% and m. p. 116–117 °C, IR, 3440, 3035, 1728, 1690, 1630 cm^{-1}, ^{1}HNMR δ 8.5 (s, 1H), 8.2–7.3 (m, 16H), 5.0 (s, 2H) Fig. S2 (Supplementary Material).

Compound 5 (R = m-nitro) is a red powder yield in 65% and m. p. 112–114 °C, IR, 2995, 1702, 1621, 1550 cm^{-1}, ^{1}HNMR δ 8.1–7.3 (m, 16H), 5.2 (s, 2H) Fig. S3 (Supplementary Material).

2.4 4-substituted benzyloxy 4-(5,7-dioxo-5,7-dihydro-6H-pyrrolo[3,4-b] pyridin-6-yl) benzenesulfonate (6-7)

A combination of 0.015 mol of 4-hydroxyacetophenone or 4-hydroxybenzaldehyde and 3 mL of pyridine was placed in three-necked flasks with a stirrer and thermostat. The temperature of the combination was lowered to 10 °C by the ice bath that surrounded the flask. During a 20-min period with constant stirring, the phthalimide sulfonyl chloride (Fathi and Al-Jawaheri, 2022) (0.01 mol, 3.2 g) was added gradually. After being refluxed for 2 h, the mixture was cooled to room temperature before being put into cold water and stirred till the oily layer solidified. The resultant solid was filtered, washed with cold, diluted HCl solution, then washed with distilled water before being dried. The desired chemicals were produced by recrystallizing the final product from ethanol Fig. 4 (Soyer *et al.*, 2017).

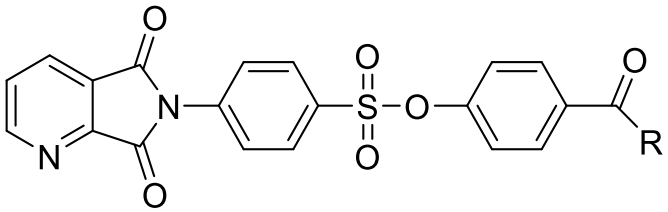

Figure 4. Substituted benzyloxy benzenesulfonate.

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Compound 6 (R=H) is a yellow powder yield in 70% and m. p. 170–172 °C, IR, 3100, 1720, 1690, 1205 cm^{-1}, ^{1}HNMR δ 10.0 (s, 1H), 8.2–7.7 (m, 9H), 7.4 (d, 2H) Fig. S4 (Supplementary Material).

Compound 7 (R=CH_3) is a yellow powder yield in 75% and m. p. 271–272 °C, IR, 3075, 1707, 1680, 1200 cm^{-1}, ^{1}HNMR δ 8.2–7.8 (m, 9H), 7.35 (d, 2H), 2.60 (s, 3H) Fig. S5 (Supplementary Material).

These compounds were prepared according to the general procedure for synthesis of Schiff bases (8–12) Fig. 5.

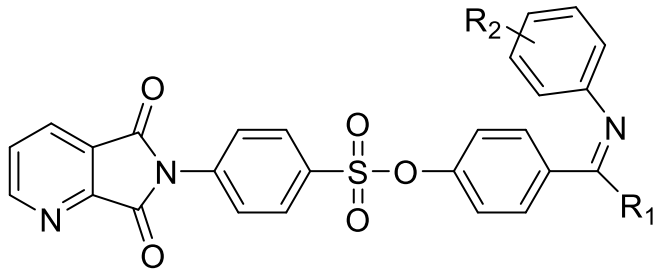

Figure 5. Substituted benzyloxy benzenesulfonate Schiff bases.

Compound 8 (R_1= CH_3/ R_2= O-methyl) is a yellow powder yield in 80% and m. p. 270–271 °C, IR, 3112, 2990, 1728, 1630 cm^{-1}, ^{1}HNMR δ 8.2–7.7 (m, 13H), 7.35 (d, 2H), 2.6 (s, 3H), 2.45 (s, 3H) Fig. S6 (Supplementary Material).

Compound 9 (R_1= CH_3/ R_2= P-methyl) is a light-yellow powder yield in 83% and m. p. 189–190 °C, IR, 3100, 2920, 1720, 1610 cm^{-1}, ^{1}HNMR δ 8.25-7.75 (m, 13H), 7.35 (d, 2H), 2.55 (s, 3H), 2.47 (s, 3H) Fig. S7 (Supplementary Material).

Compound 10 (R_1= CH_3/ R_2= 2-Nitro 4- chloro) is a white powder yield in 62% and m. p. 160–161 °C, IR, 3120, 2950, 1715, 1605 cm^{-1}, ^{1}HNMR δ 8.25–7.63 (m, 11H), 7.33(d, 2H), 2.55(s, 3H) Fig. S8 (Supplementary Material).

Compound 11 (R_1= H/ R_2= P-methyl) is a white powder yield in 90% and m. p. 226–228 °C, IR, 3099, 2925, 1710, 1600 cm^{-1}, ^{1}HNMR δ 8.7 (s,1H), 8.2- 7.7 (m, 11H), 7.55–7.2 (d,d, 4H), 2.05 (s, 3H) Fig. S9 (Supplementary Material).

Compound 12 (R_1= H/ R_2= 2,4-Dichloro) is a yellow powder yield in 83% and m. p. 190–191 °C, IR, 3090, 1725, 1600, 770 cm^{-1}, ^{1}HNMR δ 8.65 (s, 1H), 8.3- 7.7 (m, 13H), 7.6–7.3 (m, 3H) Fig. S10 (Supplementary Material).

3. Results and discussion

The synthesis of new compounds is a millstone in the field of organic chemistry and is the first step for the invention of new things that could improve our life. Quinolinic acid was used as the available starting material in this paper to convert to anhydride (1) (Fig. 6) using the dehydration agent acetic anhydride. The resultant compound (1) in 85% is regarded as a known compound and is confirmed by its physical properties as a beige solid powder and m.p. = 139–140 °C. The IR spectra gave strong signals at 3043 and 1765 cm^{-1} belonging to the C-H aromatic and carbonyl groups, respectively (Sigma Aldrich CAS Number 699-98-0).

The next step was to convert the anhydride (1) to pyrrole (2) by heating the anhydride with urea. The resultant compound was confirmed by its physical properties as a white crystal yield in 83%, m.p. = 188–190 °C. The IR spectra gave in cm^{-1} at 3104 for C-H aromatic, 1725 for carbonyl, 1620 for N-H.

Compound (3) was prepared in two steps. Firstly, compound (2) was treated with alcoholic potassium hydroxide to form organic salt to increase the nucleophilicity to react with 4-phenyl phenacyl bromide via one step SN2 mechanism (Fig. 6). The resultant compound was confirmed by physical properties as a brown powder resulting in 60% (m.p. 120–124 °C), IR 3104 cm^{-1} for aromatic C-H, 1715 cm^{-1} for amid carbonyl, 1680 cm^{-1} for ketonic. In addition, ^{1}HNMR δ chart gave signals at 8.21–7.32 (m, 12) and at 4.95 singlet belongs to two protons for CH_2 (Maimaris *et al.*, 2022).

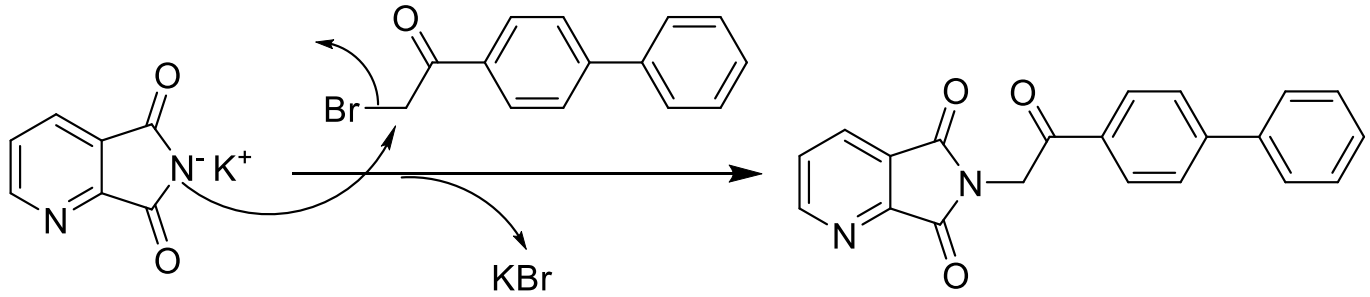

Figure 6. Mechanism of SN2 Reaction.

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Intermediate compounds were prepared according to our past paper (Fathi and Al-Jawaheri, 2022). Compounds (6-7) were prepared from phthalimide sulfonyl chloride by replacing the chlorin with the phenolic group from p-hydroxy benzaldehyde or p-hydroxy acetophenone throw SN2 mechanism. The resultant compounds were confirmed by their physical properties, for (6) was a yellow powder yield in 70%, melted at 170–171°C, IR 3100 cm^{-1} for C-H aromatic, 1720 and 1690 cm^{-1} for carbonyls, ^{1}HNMR δ chart have clear unique signals at 10 ppm singlet for aldehyde proton, 8.2–7.7 multiplet and at 7.4 doublet for aromatic protons. Compound (7) was a yellow powder yield in 75% and m.p. in 271–272°C, IR 3075 cm^{-1} for aromatic C-H, 1707 and 1680 cm^{-1} for carbonyls, ^{1}HNMR δ signals confirmed the structure at 8.2–7.8 ppm multiplet and at 7.35 doublet for aromatic protons and at 2.60 singlet belongs to three protons of CH_3 (Fadlelmoula *et al.*, 2022).

The final step was to form a Schiff base by choosing different primary amines to react with compound (3) to form (4, 5) and compounds (6, 7) to form (8–12). The mechanism of the reaction shows six steps in which the proton of the acid has been attacked by the electron pair of the carbonyl oxygen to increase the electrophilicity of the carbonyl of the carbon and form the oxonium ion. At this point, the electron pair of the amine nitrogen will be ready to attack the carbon of the carbonyl to form the zwitterion. The next steps included the release and addition of the proton, then dehydration (Fig. 7) (Choudhury and Parvin, 2011).

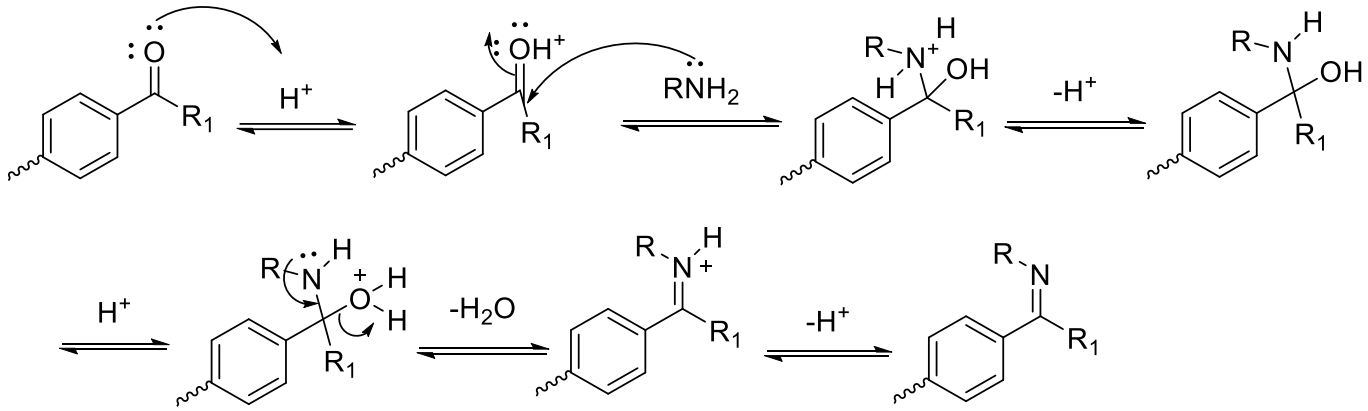

Figure 7. Mechanism of Schiff bases formations.

The structure of the resultant compounds (4, 5) was confirmed by physical properties, yellow powder and red powder yield 81% and 65%, m.p. 116–117 °C and 112–114 °C, respectively. Also, spectral data showed IR cm^{-1} 3440 belonging to the hydroxyl group in (4), 3112, 2995 for C-H aromatic, 1728, 1702 for the carbonyl group, 1690, 1621 for the C=N group, ^{1}HNMR δ ppm showed signals, 8.5 singlet for OH in (4), 8.2–7.3 and 8.1–7.3 multiplet for aromatic protons, 5.0, 5.2 singlet for CH_2 protons, respectively.

Compounds (8–12): Every compound has a unique ^{1}HNMR δ spectrum. For (8) the spectral data presented signals at 2.6 and 2.45 singlet for CH_3 protons, while (9) showed signals at 2.55, 2.47 singlet for CH_3 protons; for (10) at 2.55 singlet for CH_3 protons, for (11) and (12) at 8.7 singlet belonging to the proton attached to the C=N group and 2.05 for CH_3 protons. Finally, for (12) at 8.65 singlet belonging to the proton attached to the C=N group. (Fig. 8), find the attached Supplementary Material.

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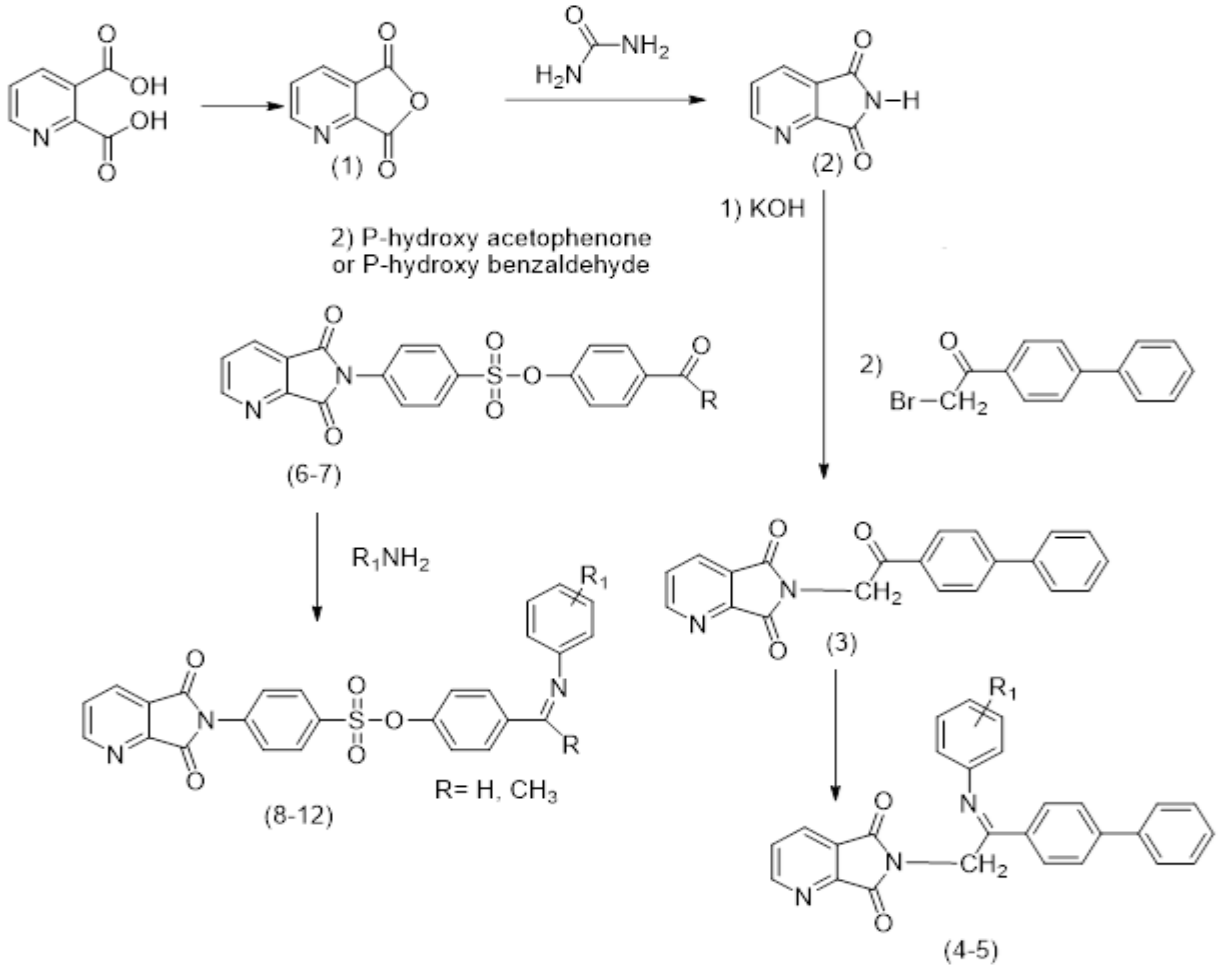

Figure 8. Scheme of the reactions.

4. Conclusions

In this study, we have successfully prepared several new substituted compounds derived from substituted nicotinic acid to enhance the library of organic chemistry. These compounds contain carbon and nitrogen double bond as an imine functional group, which can be used as starting material in many fields, such as organometal compounds.

Authors' contribution

Conceptualization: Fathi, A.; Al-Jawaheri, Y.
Data curation: Fathi, A.
Formal Analysis: Ismaeel, S.
Funding acquisition: Not applicable.
Investigation: Al-Jawaheri, Y.
Methodology: Fathi, A.; Al-Jawaheri, Y.
Project administration: Fathi, A.; Al-Jawaheri, Y.
Resources: Ismaeel, S.
Software: Not applicable.
Supervision: Fathi, A.
Validation: Fathi, A.; Al-Jawaheri, Y.; Ismaeel, S.
Visualization: Ismaeel, S.
Writing – original draft: Al-Jawaheri, Y.
Writing – review & editing: Fathi, A.; Al-Jawaheri, Y.

Data availability statement

All data set were generated or analyzed in the current study.

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Supplementary Material

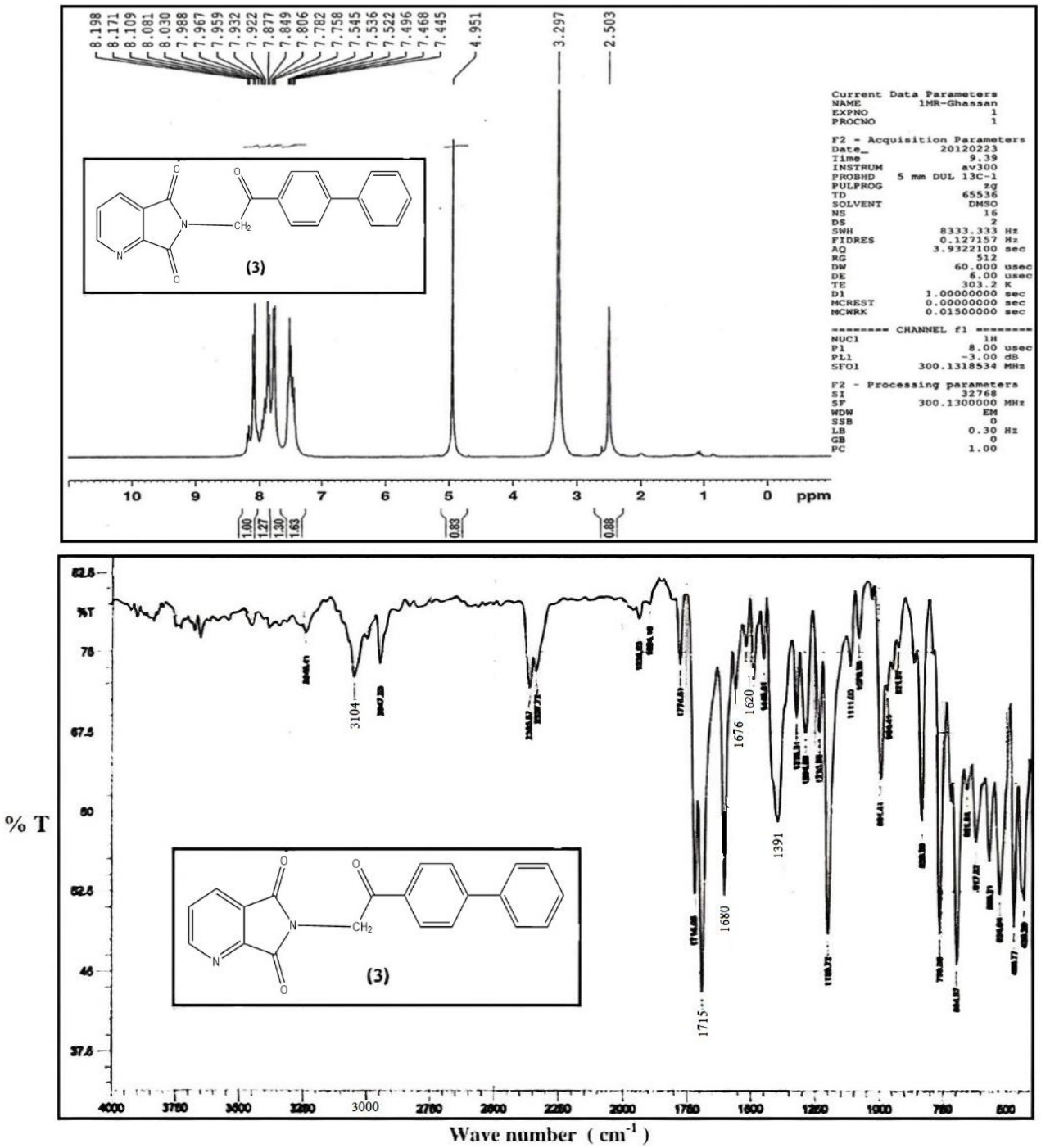

Figure S1. NMR and IR. Charts for compound 3.

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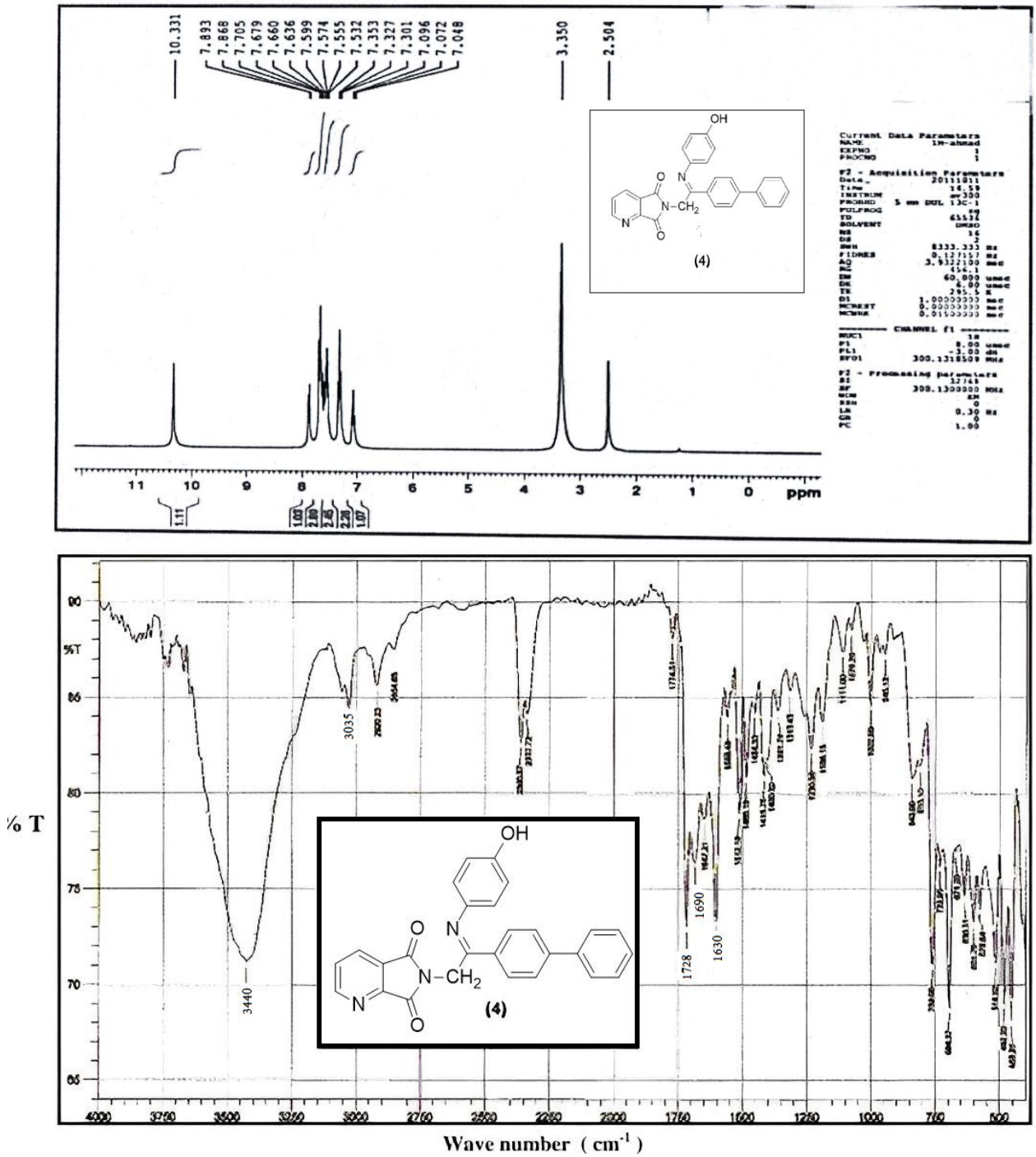

Figure S2. NMR and IR. Charts for compound 4.

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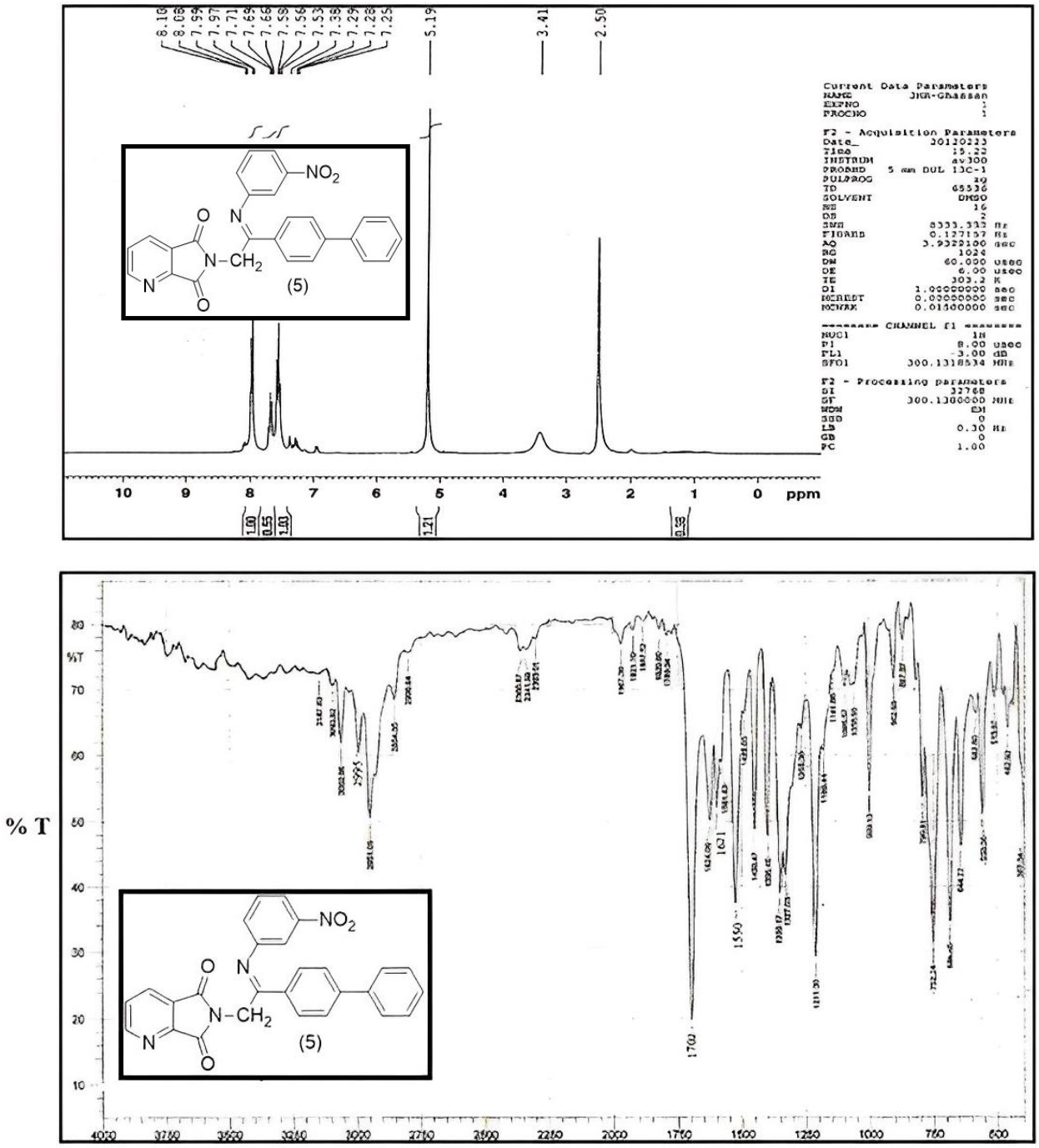

Figure S3. NMR and IR. Charts for compound 5.

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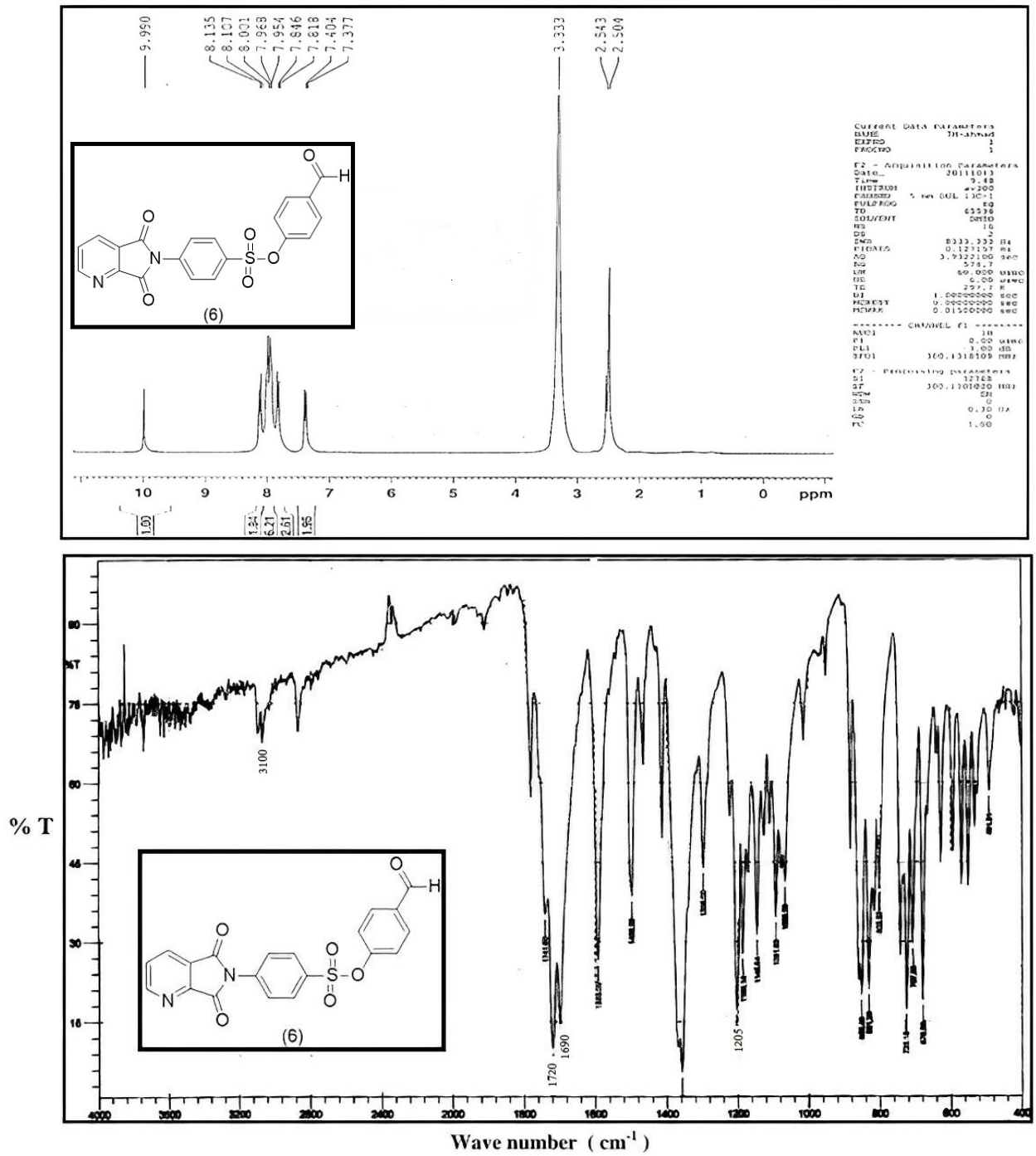

Figure S4. NMR and IR. Charts for compound 6.

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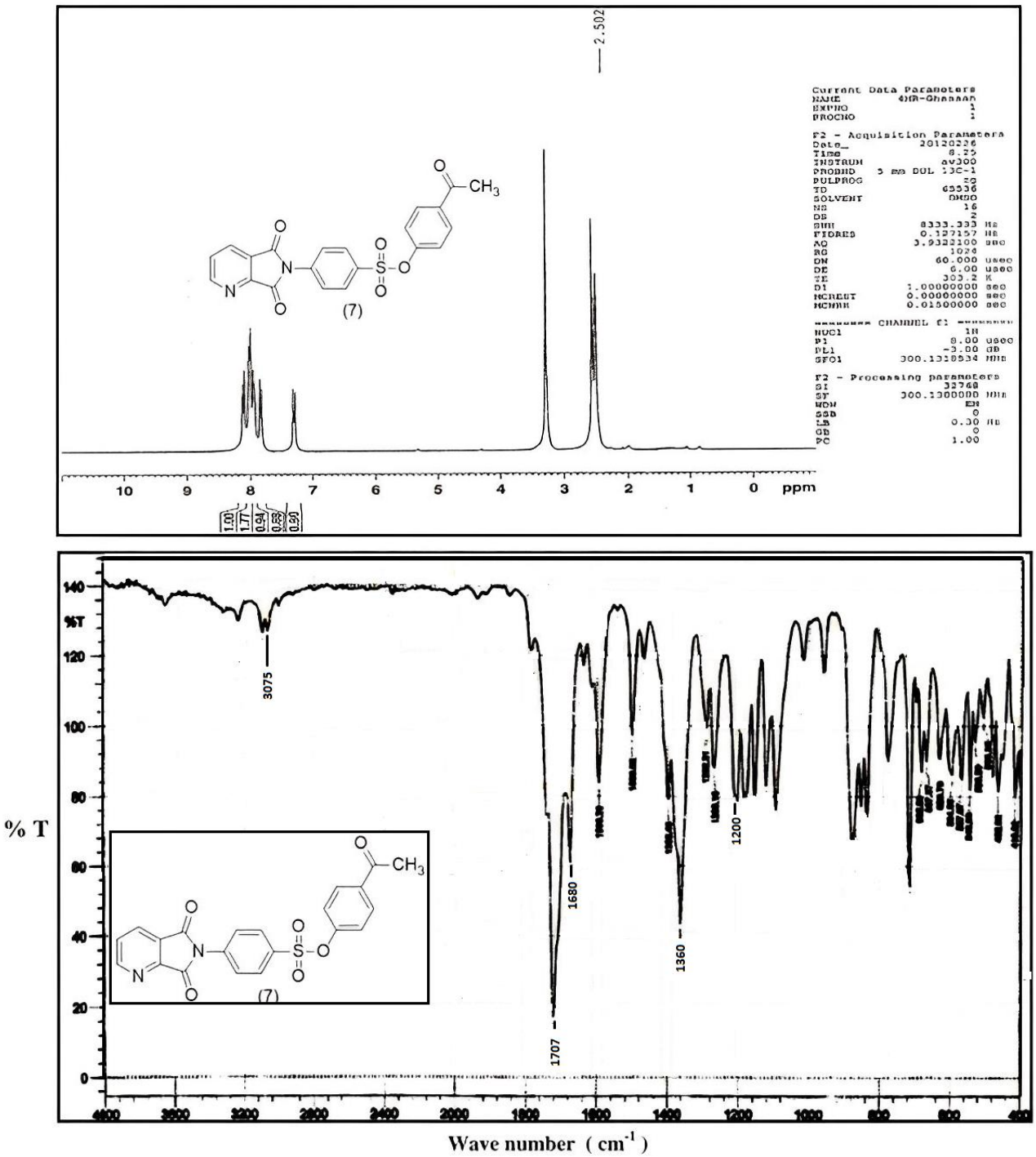

Figure S5. NMR and IR. Charts for compound 7.

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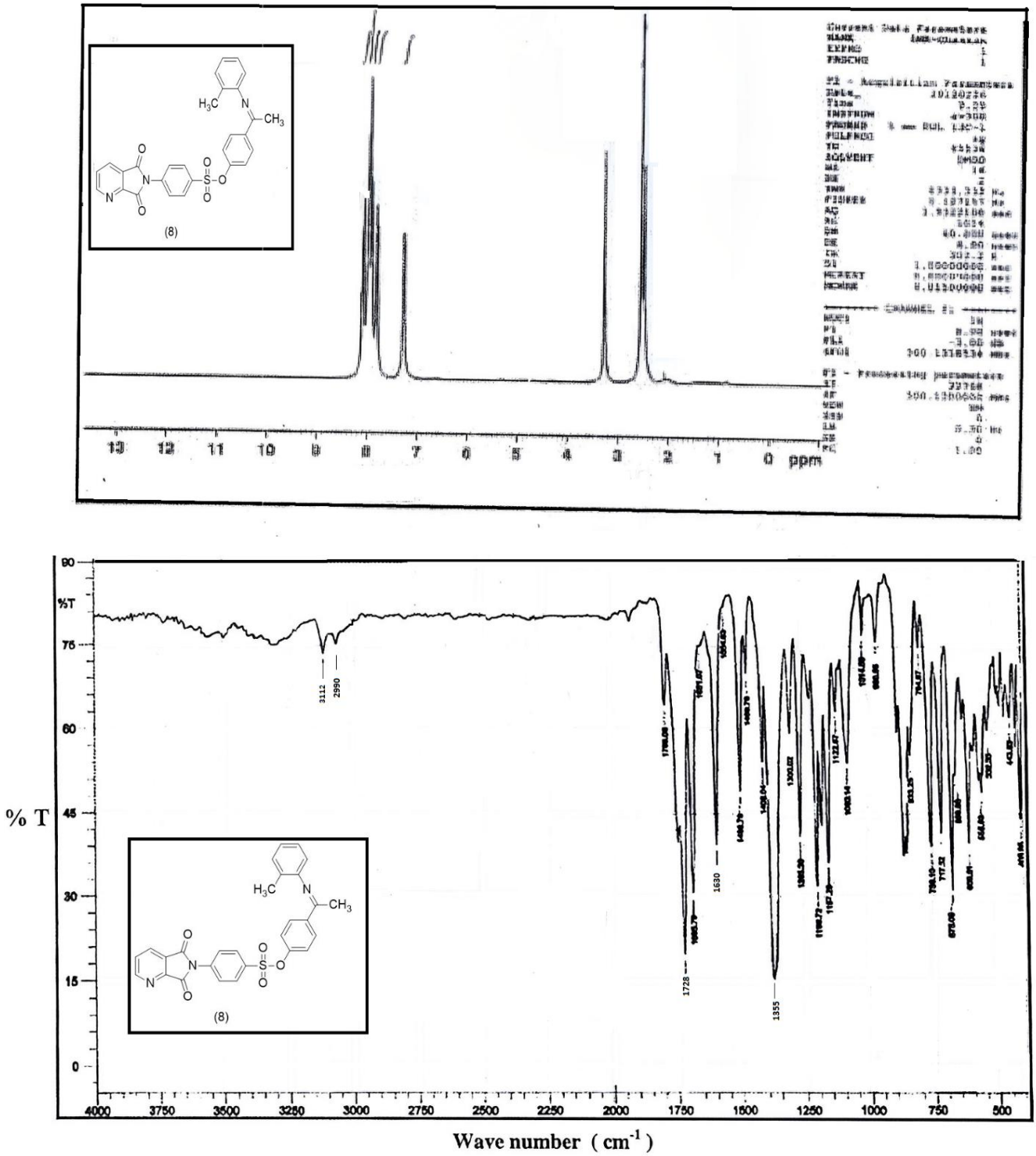

Figure S6. NMR and IR. Charts for compound 8.

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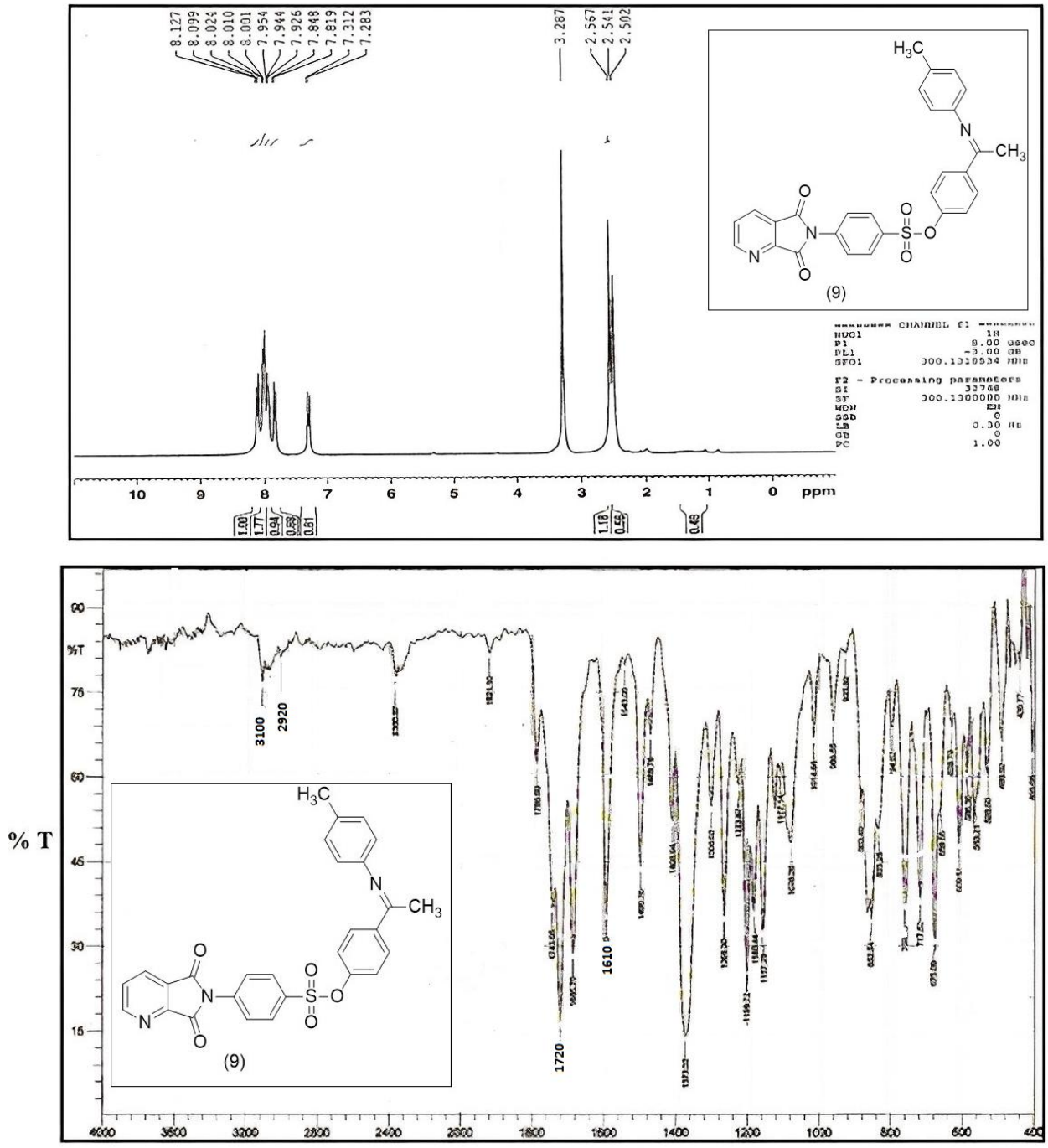

Figure S7. NMR and IR. Charts for compound 9.

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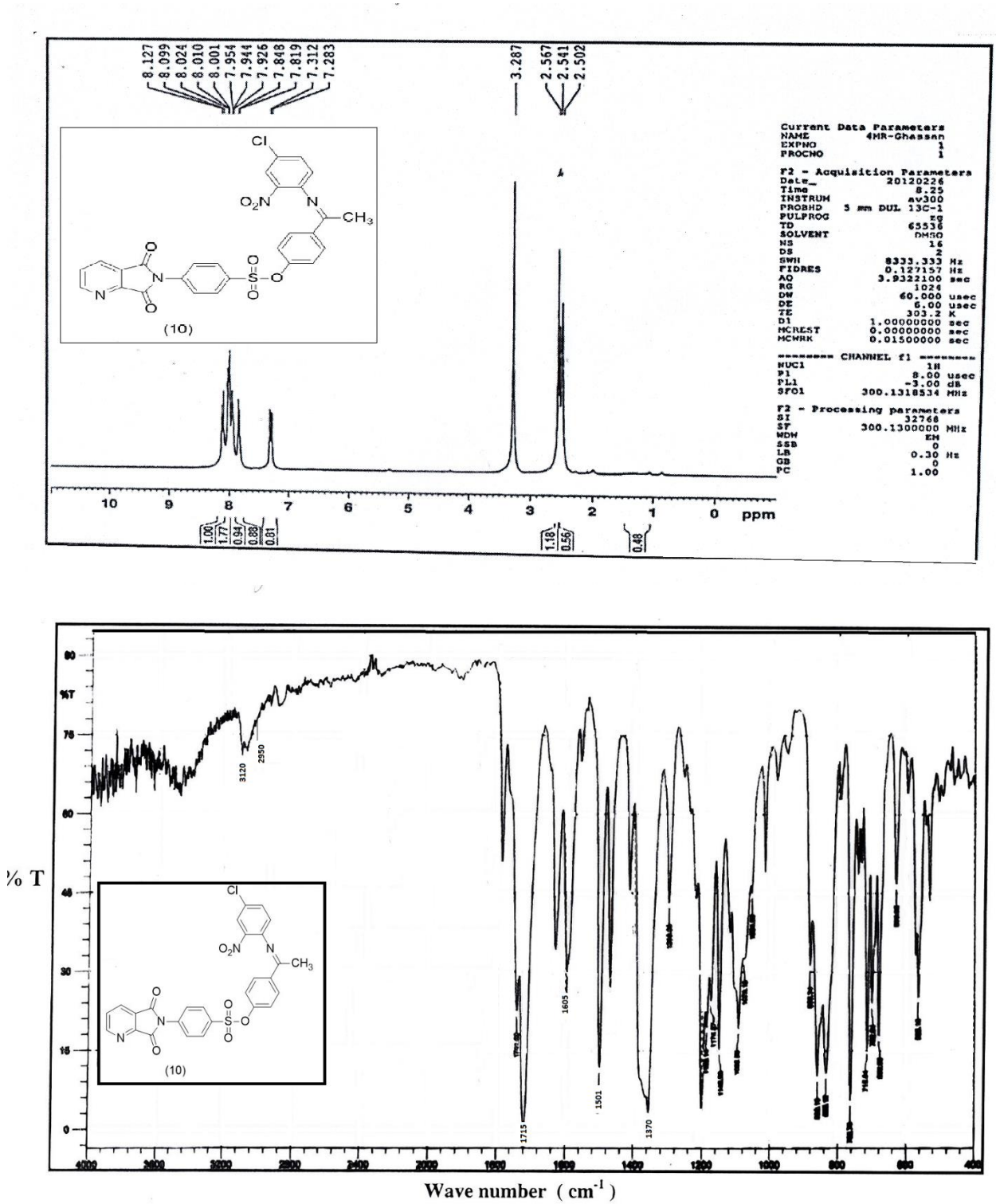

Figure S8. NMR and IR. Charts for compound 10.

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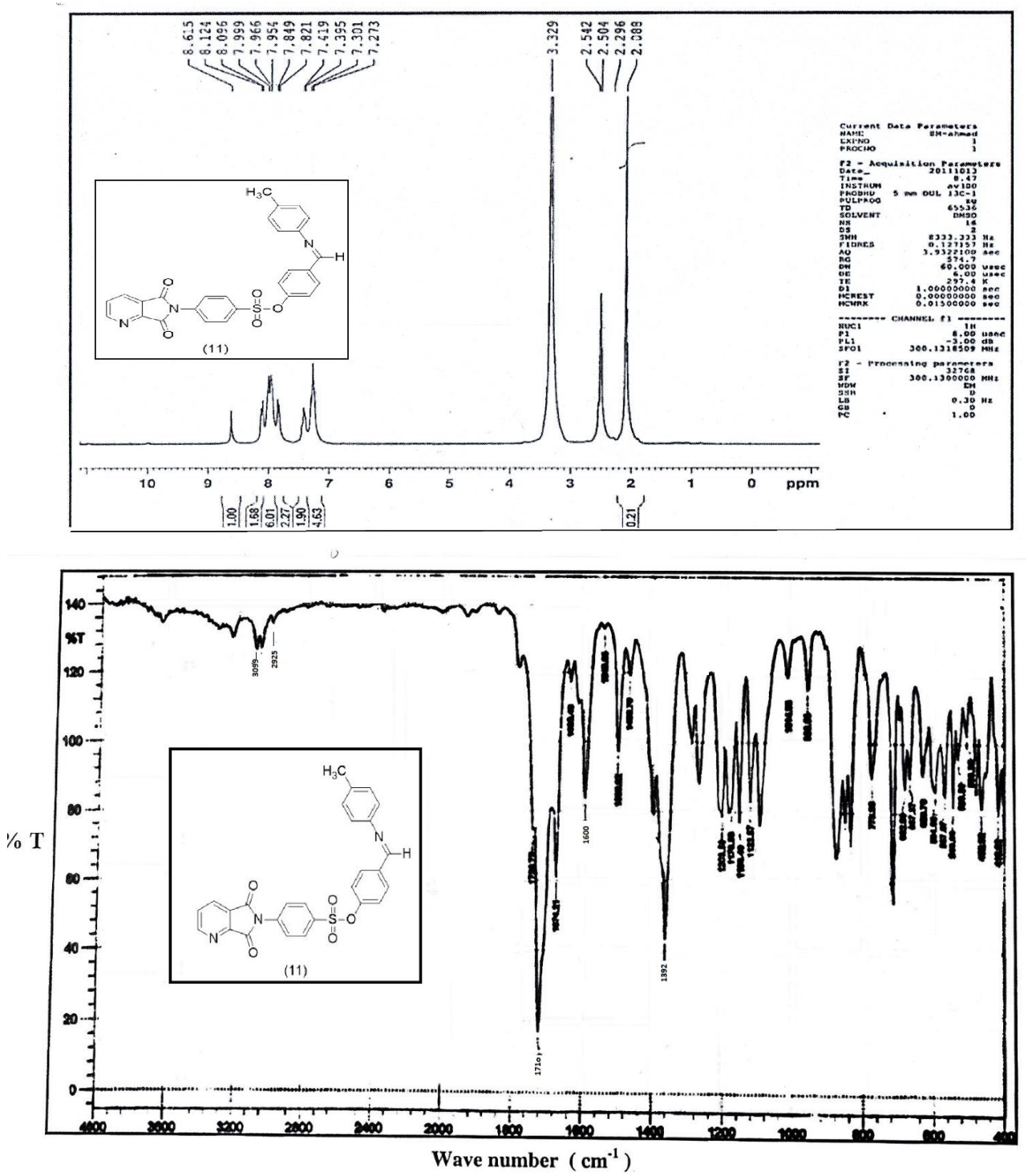

Figure S9. NMR and IR. Charts for compound 11.

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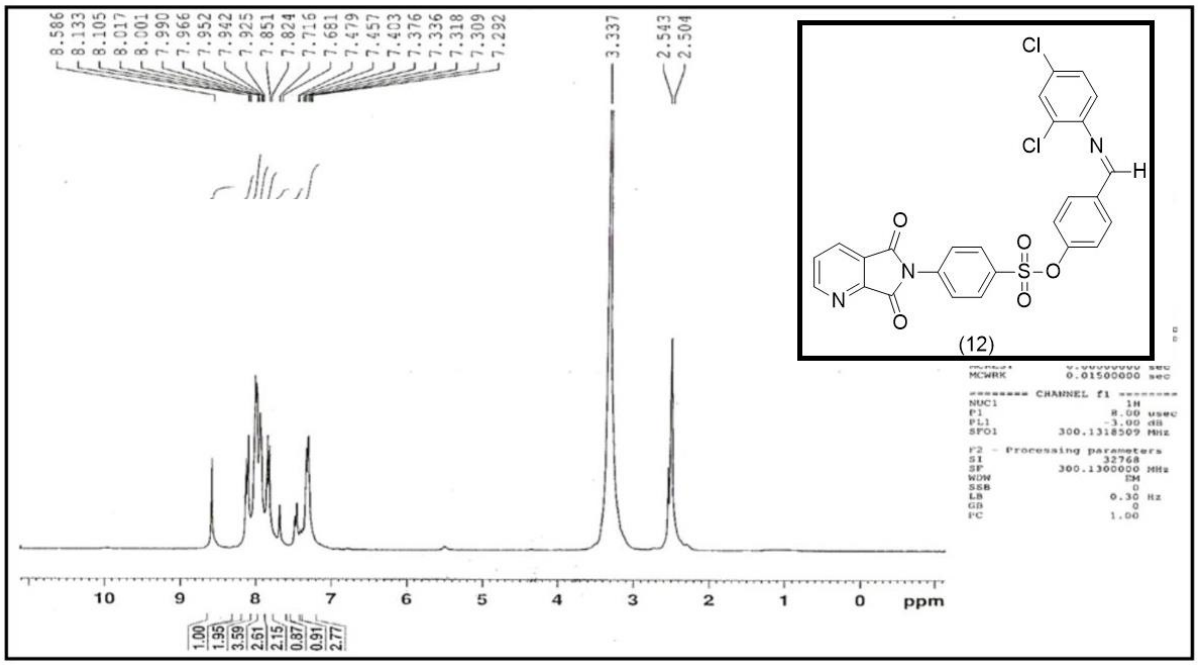

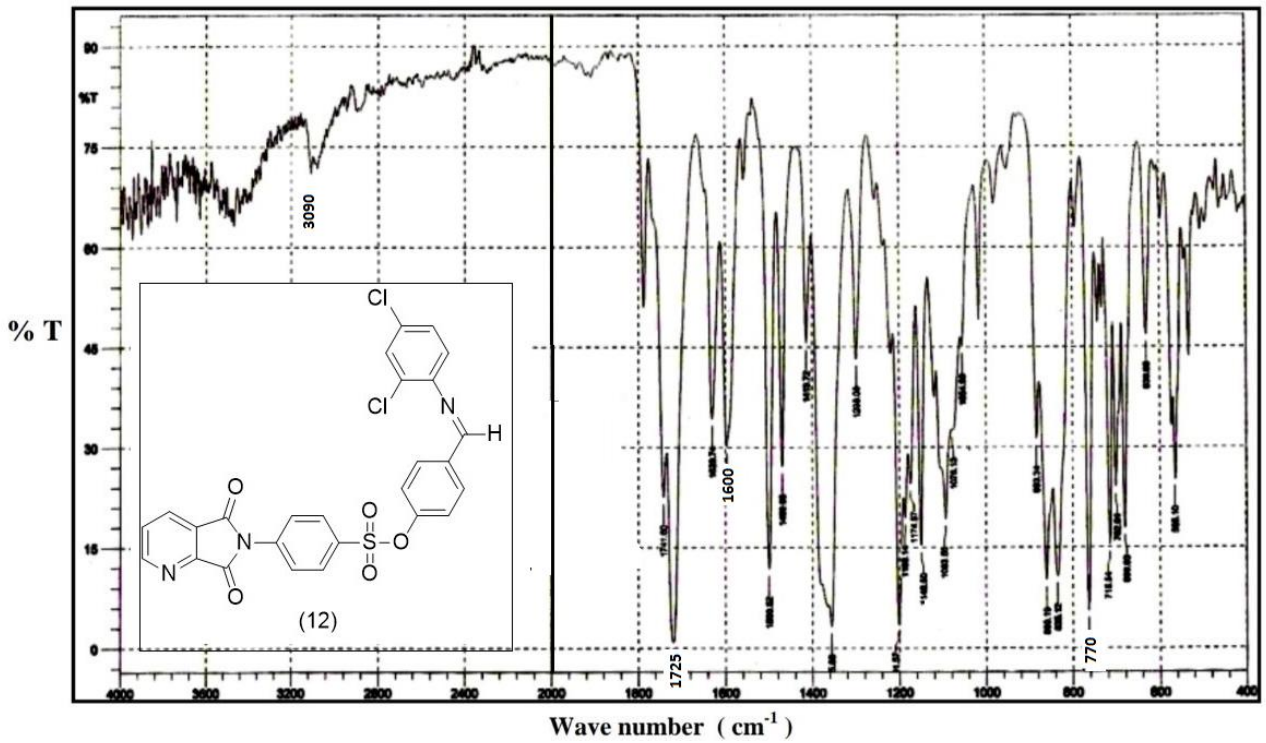

Figure S10. NMR and IR. Charts for compound 12.

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