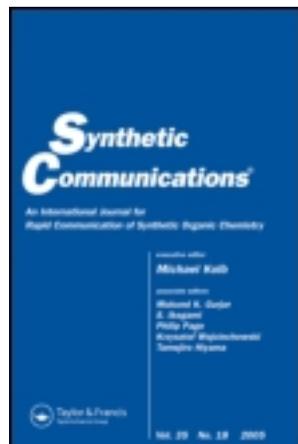


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Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/lcyc20>

Synthesis of Novel Cinnamoyl Amides Using a Solvent-Free Microwave-Assisted Method

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Accepted author version posted online: 10 May 2012. Version of record first published: 08 Nov 2012.

To cite this article: Dr. Rolando F. Pellón & Maite L. Docampo (2013): Synthesis of Novel Cinnamoyl Amides Using a Solvent-Free Microwave-Assisted Method, *Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry*, 43:4, 537-552

To link to this article: <http://dx.doi.org/10.1080/00397911.2011.604148>

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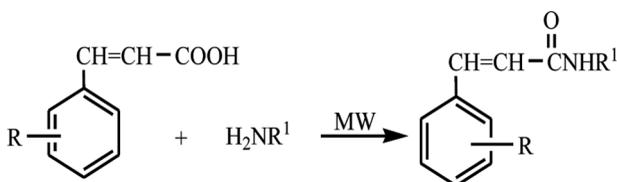
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SYNTHESIS OF NOVEL CINNAMOYL AMIDES USING A SOLVENT-FREE MICROWAVE-ASSISTED METHOD

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GRAPHICAL ABSTRACT



Abstract A novel series of potential biologically active new cinnamoyl amides were synthesized in good yield and short reaction times. We have studied the one-pot, solvent-free reaction of cinnamic acid derivatives with aromatic amines using 1,3-dicyclohexylcarbodiimide under microwave irradiation in the presence of small quantities of dimethylformamide to improve energy transfer.

Keywords Cinnamoyl amides; condensation; microwave heating; solvent-free reaction; synthetic methods

INTRODUCTION

Cinnamic acid and its derivatives are important reagents in organic synthesis as intermediates and final products. Various cinnamic acid derivatives have been studied as cancer-preventive agents^[1] and antimalaric agents.^[2] Also, inhibitory effects on melanin biosynthesis in skin were demonstrated.^[3] Cinnamoyl amides are potential hypoglycemic agents^[4] and were evaluated as anti-inflammatory and analgesic activities.^[5] Several of these compounds were obtained and showed an improvement in anti-Chagas activity in comparison to the earlier derivatives.^[6] The best activity was obtained with an ethyl, ethenyl, and thiomethyl or a hydrogen bond acceptor as a nitro group in the *para*-position of the terminal phenyl residue.^[7] The growing interest during recent years in natural and synthetic amides of cinnamic acid

Received June 16, 2011.

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is due to the metabolic stability of the amide group in comparison to that of the ester group.^[8]

A conventional synthesis of amide compounds has been performed by a condensation between carboxylic acid and amine with heating.^[9] Cinnamoyl amides can be obtained via acyl chlorides, which were prepared by the action of thionyl chloride on cinnamic acids and reacted in situ with the corresponding amine in chloroform as solvent.^[10] Yesilada and Zurlo^[5] reported the synthesis of cinnamoyl tertiary amides using 1,3-dicyclohexylcarbodiimide/4-hydroxybenzotriazol in dimethylformamide (DMF) as solvent at room temperature. However, these synthetic routes often involve long reaction times, toxic reagents or solvents, and labor-intensive operations.

On the other hand, Nomura *et al.*^[11] tried to prepare the amide compounds of cinnamic acids with alkylamines by microwave (MW) irradiation. However, the styrenes (descarboxylation reaction) were obtained predominantly from a cinnamoyl amide-forming reaction.

Previous work^[12] of our group has demonstrated that MW or ultrasound irradiation gives significant rate enhancements and improved yields in organic reactions. Many reactions that typically need many hours to reach completion with conventional heating can be brought to full conversion in only seconds or minutes by utilizing these powerful tools.^[13]

To overcome these disadvantages in the amide-forming reaction, in this work we describe a facile one-pot condensation reaction of cinnamic acid and aromatic amines under solvent-free conditions using MW irradiation to obtain new cinnamoyl amide derivatives.

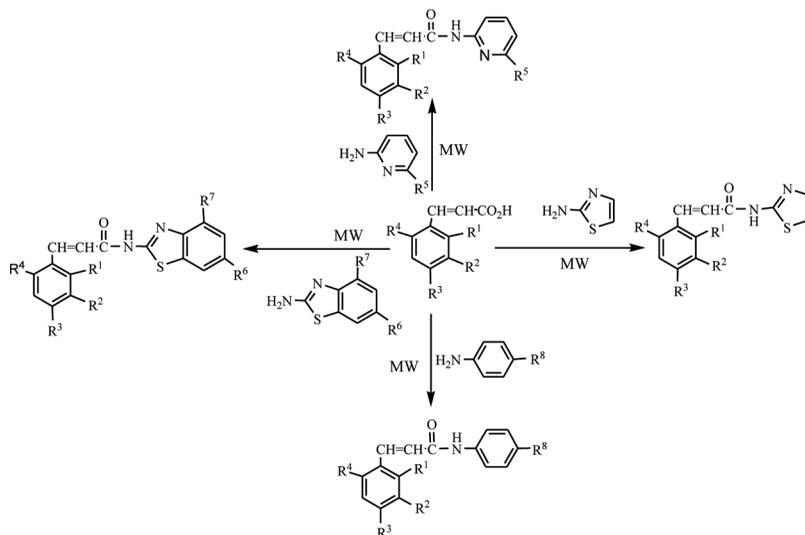
RESULTS AND DISCUSSION

As a first part of this work we studied the condensation of cinnamic acid derivatives with aromatic amines under MW irradiation in absence of solvent (Scheme 1). In our initial screening experiments, we chose the condensation of cinnamic acid (**1a**) with 2-aminothiazole (**2a**) as the model for exploring suitable reaction conditions. The synthetic route is shown in Scheme 1.

Before performing any new reaction under MW irradiation, we needed to test the thermal behavior of every reagent as well as reaction mixtures when they are submitted to the electromagnetic field. This preliminary step allowed determination of adequate conditions of incident power and irradiation time. No degradation of reactants and products was obtained under MW irradiation at 180 W and 10 min, which are, respectively, the greatest irradiation power and longest time used in the experiments.

We examined the condensation of equimolecular amounts of cinnamic acid and 2-aminothiazole with microwave heating at 180 W for 10 min (Table 1 entry 1), but the condensation did not take place satisfactorily, obtaining only 12% yield of the corresponding cinnamoyl amide.

To ensure the reaction to go to completion we decided to use a coupling dehydrating agent such as 1,3-dicyclohexylcarbodiimide (DDC), which is converted in the process to dicyclohexylurea. Using a relationship of 1:1:2 (cinnamic

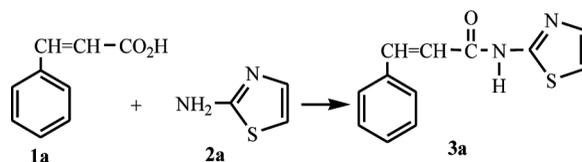


Scheme 1. Condensation of cinnamic acid derivatives with aromatic amines under microwave irradiation in the absence of solvent.

acid-amine-DCC) and the MW activation conditions described previously, we obtained 43% yield of cinnamoyl amide (Table 1, entry 2).

With the aim of enhancing yield, we examined this reaction in wet conditions using a small amount of DMF, and cinnamoyl amide (**3a**) was obtained in 90% yield (Table 1, entry 3). This polar molecule (i.e., highly sensitive to MW irradiation) was

Table 1. MW-assisted solvent-free condensation reaction of cinnamic acid (**1a**) and 2-aminothiazole (**2a**): optimization conditions^a



Entry	Molar ratio ^b	DMF	Yield (%) ^c	Standard deviation (S)
1	1:1:0	None	12	±1.5
2	1:1:2	None	43	±2.0
3	1:1:2	Drops	90	±1.7
4	1:1:1.5	Drops	91	±2.2
5	1:1:1.2	Drops	89	±1.3
6	1:1:1.1	Drops	90	±1.6
7	1:1:1.0	Drops	92	±1.4
8	1:1:0.5	Drops	79	±1.7

^aMW conditions: P = 180 W, reaction time: 10 min.

^bCinnamic acid-amine-DCC.

^cIsolated yields.

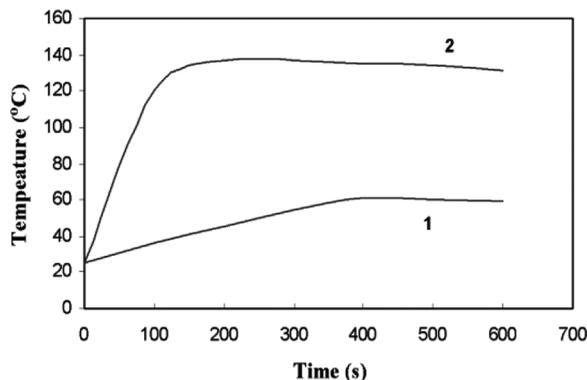


Figure 1. Profile of rise in temperature for the MW-assisted solvent-free synthesis of **3a**. Without DMF (1); adding drops of DMF (2) (20 mmol of **1a**–20 mmol of **2a**–40 mmol of DCC); performed at 180 W full time.

added to improve the energy transfer and to allow higher temperatures.^[14] To attribute these effects, the profiles of temperature increases were carefully studied.

The employment in this investigation of a modern MW reactor (Synthewave S402) for organic synthesis permitted controlled reaction conditions such as temperature and pressure. We used this reactor to monitor the temperature profiles during the condensation of cinnamic acid and 2-aminothiazole in presence of DCC under MW irradiation at 180 W from 0 to 10 min reaction time (with and without DMF), and the results are shown in Fig. 1.

The maximum temperature is in the range of 59–61 °C when DMF was not present, obtaining a poor yield (43%) of compound **3a**. Further, upon the addition of DMF, the reaction mixture was rapidly heated, reaching a range of 131–137 °C after 3 min of exposure to MW, obtaining 90% yield.

Next, we investigated the effect of relative amounts of DCC in the presence of DMF. The main results are summarized in Table 1 (entries 3–8). The reaction under MW conditions was obtained successfully using a ratio of cinnamic acid–amine–DCC 1:1:1 in the presence of small amounts of DMF (Table 1, entry 7).

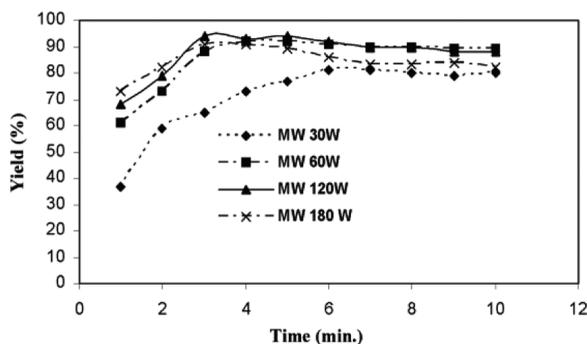


Figure 2. Effect of the irradiation power in the condensation reaction of cinnamic acid **1a** (20 mmol) with 2-aminothiazole **2a** (20 mmol) in presence of DCC (20 mmol) and drops of DMF.

Table 2. Comparison between microwave (MW) irradiation and conventional heating for the synthesis of new cinnamoyl amides^a

Entry	Substituents	Method	T (°C) ^{b,c}	t (min)	Yield (%) ^d
3a	R ¹ = R ² = R ³ = R ⁴ = H	MW	135	3	94
		Δ	135	360	8
3b	R ¹ = R ⁴ = H; R ² = R ³ = OCH ₃	MW	135	2	96
		Δ	135	300	12
3c	R ¹ = R ⁴ = R ⁵ = H; R ² = R ³ = OCH ₃	MW	140	1	97
		Δ	140	300	14
3d	R ¹ = R ⁴ = H; R ² = R ³ = OCH ₃ ; R ⁵ = CH ₃	MW	140	1	95
		Δ	140	300	18
3e	R ¹ = R ² = R ³ = R ⁴ = R ⁶ = R ⁷ = H	MW	135	3	90
		Δ	135	360	6
3f	R ¹ = R ⁴ = R ⁶ = R ⁷ = H; R ² = R ³ = OCH ₃	MW	135	2	93
		Δ	135	300	7
3g	R ¹ = R ³ = R ⁶ = R ⁷ = H; R ² = R ⁴ = OCH ₃	MW	135	2	92
		Δ	135	300	8
3h	R ¹ = R ² = R ⁴ = R ⁶ = R ⁷ = H; R ³ = OCH ₃	MW	135	2	90
		Δ	135	300	6
3i	R ¹ = R ² = R ⁴ = R ⁶ = R ⁷ = H; R ³ = OC ₂ H ₅	MW	135	2	92
		Δ	135	300	5
3j	R ¹ = R ² = R ⁴ = R ⁶ = R ⁷ = H; R ³ = OC ₃ H ₇	MW	140	2	91
		Δ	140	300	5
3k	R ¹ = R ² = R ⁴ = R ⁶ = R ⁷ = H; R ³ = OC ₄ H ₉	MW	140	2	92
		Δ	140	300	7
3l	R ¹ = R ² = R ⁴ = R ⁶ = R ⁷ = H; R ³ = CH ₃	MW	130	3	89
		Δ	130	360	4
3m	R ¹ = R ² = R ⁴ = R ⁶ = R ⁷ = H; R ³ = C ₂ H ₅	MW	130	3	90
		Δ	130	360	5
3n	R ² = R ³ = R ⁴ = R ⁶ = R ⁷ = H; R ¹ = Cl	MW	135	2	91
		Δ	135	300	9

(Continued)

Table 2. Continued

Entry	Substituents	Method	T (°C) ^{b,c}	t (min)	Yield (%) ^d
3o	R ¹ = R ² = R ⁴ = R ⁶ = R ⁷ = H; R ³ = Cl	MW	135	2	90
		Δ	135	300	8
3p	R ¹ = R ⁴ = R ⁷ = H; R ² = R ³ = R ⁶ = OCH ₃	MW	140	2	93
		Δ	140	300	10
3q	R ¹ = R ⁴ = R ⁷ = H; R ² = R ³ = OCH ₃ ; R ⁶ = OC ₂ H ₅	MW	140	2	91
		Δ	140	300	9
3r	R ¹ = R ⁴ = R ⁷ = H; R ² = R ³ = OCH ₃ ; R ⁶ = CH ₃	MW	135	2	89
		Δ	135	300	9
3s	R ¹ = R ⁴ = R ⁷ = H; R ² = R ³ = OCH ₃ ; R ⁶ = SO ₂ CH ₃	MW	140	2	90
		Δ	140	300	5
3t	R ¹ = R ⁴ = R ⁷ = H; R ² = R ³ = OCH ₃ ; R ⁶ = NO ₂	MW	140	2	93
		Δ	140	300	11
3u	R ¹ = R ⁴ = R ⁶ = H; R ² = R ³ = OCH ₃ ; R ⁷ = Cl	MW	135	3	88
		Δ	135	360	3
3v	R ¹ = R ⁴ = R ⁶ = H; R ² = R ³ = OCH ₃ ; R ⁷ = CH ₃	MW	135	3	89
		Δ	135	360	4

3w	R ¹ = R ² = R ³ = R ⁴ = R ⁸ = H	MW	145	1	98
		Δ	145	300	12
3x	R ¹ = R ² = R ³ = R ⁴ = H; R ⁸ = COOH	MW	140	1.5	97
		Δ	140	300	10

^aReaction conditions: cinnamic acid (20 mmol), amine (20 mmol), DCC (20 mmol), DMF (3 drops), 120 W.

^bMethod MW: The temperature was controlled throughout the reaction and evaluated by infrared detector, which indicated the surface temperature after calibration with an optical fibre.

^cMethod Δ: The temperature was controlled using a digital thermometer inside the reaction mixture.

^dYields (%) refer to those of pure isolated products properly characterized by spectroscopic data (¹H, ¹³C NMR, MS).

Finally, we performed several experiments at various powers to study the effect of irradiation time on yields of cinnamoyl amide **3a** under the conditions studied previously. The best results (94% yield) were obtained within 3 min at a power of 120 W (Fig. 2).

With the optimized procedure in hand, the scope of this condensation was further explored with different cinnamic acid and amines (thiazolamines, benzothiazolamines, aminopyridines, aminobenzoic acids, and anilines), and the results are summarized in Table 2. In general, no significant difference of the reactivity was observed for the examined reactants (cinnamic acid and amines) with varied electronic properties, electron-rich and electron-poor; the rates and yields of the reactions were comparable (Table 2).

To show the advantages of the MW heating mode, Table 2 shows results for the synthesis of new cinnamoyl amides compared to classic heating. To check the

possibility of intervention of specific nonpurely thermal effects of microwaves, they are also compared to solvent-free reaction by classical heating (thermostated oil bath) in the same conditions as under microwave (time, temperature, profiles of rise in temperature, vessels). The lower yields obtained with conventional heating mode, even after 6 h of reaction, indicate once more that the effect of MW irradiation is not purely thermal (Table 2).

We suggest that the improvement achieved with this method, due to a strong specific MW effect, could be connected to the reaction mechanism and evolution of polarity during the course of the reaction.

CONCLUSIONS

A novel series of potentially biologically active cinnamoyl amides was synthesized using the MW-assisted procedure described here. This procedure leads to good yields, in the absence of solvent, within very short reaction times and with simplified and safe workup.

EXPERIMENTAL

Reactions were monitored by thin-layer chromatography (TLC) on Merck 60 F₂₅₄ (0.25-mm) plates, which were visualized using a CAMAG UV-Cabinet II at 254 nm using chloroform–ethyl acetate (4:1 v/v). Melting points were determined in open capillaries with a Gallenkamp melting-point apparatus and are uncorrected. ¹H and ¹³C NMR spectra were recorded on a Bruker Avance 300 instrument at 298 K, using CDCl₃ as solvent unless otherwise stated. The ¹H and ¹³C chemical shifts are reported in parts per million (ppm) relative to residual deuterated solvent signal as an internal reference. The coupling constants (*J*) are given in hertz (Hz). Mass spectra were recorded by using Spectrometer Q-TOFII-Micromass, Manchester Ing. Elemental analyses were carried out on a Fisons EA 1108 CHNS-O apparatus at the Microanalytical Unit. All reagents purchased from commercial sources were used without further purification. All experiments were replicated to ensure the reproducibility, and the yields reported are averages of at least three independent runs.

Microwave Irradiation Experiments

All MW irradiation experiments were carried out in a MW reactor monomode system (Synthewave 402: prototype designed by Prolabo). The temperature is controlled by an infrared (IR) detector, which indicates the surface temperature. (IR measurement was calibrated using a thermocouple introduced in the reaction mixture.) Mechanical stirring provides good homogeneity of the materials. Automatic control of the irradiation (power and temperature) and data treatment were followed by a computer system. All the reactions were performed in a cylindrical Pyrex vessel.

For the sake of comparison, reactions were conducted under classical heating in a thermostated oil bath. Measurements of the temperature evolution were made in the reaction medium with a digital thermometer.

Condensation of Cinnamic Acids (1a–1k) with Amines (2a–2m) Under Microwave Irradiation

Cinnamic acid derivative (20 mmol), amine (20 mmol), DCC (20 mmol), and DMF (three drops) were placed in a MW monomode reactor in a cylindrical Pyrex vessel. The mixture was irradiated at 120 W with controlled temperatures for reaction times as indicated in Table 2. Continuous mechanical stirring provided a good homogeneity of materials. When the irradiation was stopped, the reaction mixture was treated with dichloromethane (CH_2Cl_2) and the precipitated DHU was filtered off. The organic layer was concentrated in vacuum, and the crude products were crystallized from methanol to give the pure products **3a–3x** in 88–98% yields.

The structures of the products obtained were confirmed by melting point, elemental analyses, ^1H NMR, ^{13}C NMR, and mass spectra.

(2E)-3-Phenyl-N-1,3-thiazol-2-ylacrylamide (Table 2, 3a)

The mixture was irradiated for 3 min. Yield: 94%. Mp 202–203 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 6.68 (d, 1H, H_b , $^3J = 15.8$ Hz), 7.21 (d, 1H, Ar, H_4), 7.27–7.30 (m, 1H, Ar, H_4), 7.43 (dd, 2H, Ar, H_3 , H_5), 7.51 (d, 1H, Ar, H_5), 7.59 (dd, 2H, Ar, H_2 , H_6), 7.77 (d, 1H, H_a , $^3J = 15.8$ Hz), 12.10 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C-4': 111.8; C-b: 117.3; C-2, C-6: 128.2; C-3, C-5: 129.2; C-4: 130.2; C-1: 134.9; C-5': 138.5; C-a: 141.9; C-2': 153.9; C(C=O): 164.3. ESI-MS (m/z): 231.2759 $[\text{M} + \text{H}]^+$. Anal. calcd. for $\text{C}_{12}\text{H}_{10}\text{N}_2\text{OS}$ (230.29): C, 62.59; H, 4.38. Found: C, 62.57; H, 4.39.

(2E)-3-(3,4-Dimethoxyphenyl)-N-1,3-thiazol-2-ylacrylamide (Table 2, 3b)

The mixture was irradiated for 2 min. Yield: 96%. Mp 208–209 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 3.79, 3.82 (s, 3H, OCH_3), 6.71 (d, 1H, H_b , $^3J = 15.7$ Hz), 6.90 (d, 1H, Ar, H_5), 7.11 (dd, 1H, Ar, H_6), 7.22 (d, 1H, Ar, H_4), 7.25 (d, 1H, Ar, H_2), 7.53 (d, 1H, Ar, H_5), 7.70 (d, 1H, H_a , $^3J = 15.7$ Hz), 12.20 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH_3): 55.9; 55.8; C-2: 110.8; C-4': 112.9; C-5: 113.6; C-b: 118.2; C-6: 123.1; C-1: 127.1; C-5': 137.8; C-a: 142.3; C-3: 148.9; C-4: 150.8; C-2': 154.1; C(C=O): 163.4. ESI-MS (m/z): 291.3405 $[\text{M} + \text{H}]^+$. Anal. calcd. for $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_3\text{S}$ (290.34): C, 57.92; H, 4.86. Found: C, 57.91; H, 4.88.

(2E)-3-(3,4-Dimethoxyphenyl)-N-pyridin-2-ylacrylamide (Table 2, 3c)

The mixture was irradiated for 1 min. Yield: 97%. Mp 153–155 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 3.80, 3.84 (s, 3H, OCH_3), 6.69 (d, 1H, H_b , $^3J = 15.9$ Hz), 6.92 (d, 1H, Ar, H_5), 7.15 (dd, 1H, Ar, H_6), 7.21 (d, 1H, Ar, H_2), 7.30 (ddd, 1H, Ar, H_5), 7.65 (d, 1H, H_a , $^3J = 15.9$ Hz), 7.75 (dd, 1H, Ar, H_4), 8.19 (dd, 1H, Ar, H_3), 8.54 (dd, 1H, Ar, H_6), 12.10 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH_3): 55.9; 56.0; C-2: 110.2; C-5: 112.0; C-3': 114.4; C-b: 118.3; C-5': 122.9; C-6: 124.2; C-1: 127.6; C-4': 139.2; C-a: 140.9; C-3: 149.1; C-6': 149.8; C-4: 151.3; C-2': 152.5; C(C=O): 168.7. ESI-MS

(*m/z*): 285.3109 [M + H]⁺. Anal. calcd. for C₁₆H₁₆N₂O₃ (284.31): C, 67.59; H, 5.67. Found: C, 67.62; H, 5.66.

**(2E)-3-(3,4-Dimethoxyphenyl)-N-(6-methylpyridin-2-yl)acrylamide
(Table 2, 3d)**

The mixture was irradiated for 1 min. Yield: 95%. Mp 158–160 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 2.46 (s, 3H, CH₃), 3.81, 3.83 (s, 3H, OCH₃), 6.67 (d, 1H, H_b, ³J = 16.1 Hz), 6.90 (dd, 1H, Ar, H₅), 7.07 (d, 1H, Ar, H₅), 7.14 (dd, 1H, Ar, H₆), 7.23 (d, 1H, Ar, H₂), 7.63 (d, 1H, H_a, ³J = 16.1 Hz), 7.67 (dd, 1H, Ar, H₄), 8.16 (dd, 1H, Ar, H₃), 11.90 (s, 1H, NH). ¹³C NMR (75.03 MHz, DCCl₃, δ ppm, 25 °C): C(CH₃): 23.1; C(OCH₃): 56.2, 56.4; C-2: 109.8; C-5: 111.4; C-3': 117.9; C-b: 118.8; C-5': 123.4; C-6: 124.2; C-1: 127.9; C-4': 137.2; C-a: 139.9; C-3: 149.2; C-4: 151.9; C-6': 155.2; C-2': 156.5; C(C=O): 168.1. ESI-MS (*m/z*): 299.3411 [M + H]⁺. Anal. calcd. for C₁₇H₁₈N₂O₃ (298.34): C, 68.44; H, 6.08. Found: C, 68.40; H, 6.11.

(2E)-N-1,3-Benzothiazol-2-yl-3-phenylacrylamide (Table 2, 3e)

The mixture was irradiated for 3 min. Yield: 90%. Mp 246–247 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 6.81 (d, 1H, H_b, ³J = 16.0 Hz), 7.25–7.30 (m, 1H, Ar, H₄), 7.35 (ddd, 1H, Ar, H₆), 7.40 (ddd, 1H, Ar, H₇), 7.45 (dd, 2H, Ar, H₃, H₅), 7.63 (dd, 2H, Ar, H₂, H₆), 7.78 (d, 1H, H_a, ³J = 16.0 Hz), 7.80–7.88 (m, 2H, Ar, H₅, H₈), 12.00 (s, 1H, NH). ¹³C NMR (75.03 MHz, DCCl₃, δ ppm, 25 °C): C-b: 118.4; C-8': 119.1; C-5': 120.5; C-6': 121.0; C-7': 123.7; C-3, C-5: 128.1; C-2, C-6: 129.1; C-4: 130.0; C-4': 131.6; C-1: 134.1; C-a: 143.1; C-2': 148.6; C-9': 158.0; C(C=O): 164.0. ESI-MS (*m/z*): 281.3443 [M + H]⁺. Anal. calcd. for C₁₆H₁₂N₂OS (280.34): C, 68.55; H, 4.31. Found: C, 68.51; H, 4.39.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(3,4-dimethoxyphenyl)acrylamide
(Table 2, 3f)**

The mixture was irradiated 2 min. Yield: 93%. Mp 215–217 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 3.81, 3.82 (s, 3H, OCH₃), 6.79 (d, 1H, H_b, ³J = 16.2 Hz), 6.89 (d, 1H, Ar, H₅), 7.10 (dd, 1H, Ar, H₆), 7.28 (d, 1H, Ar, H₂), 7.35 (ddd, 1H, Ar, H₆), 7.41 (ddd, 1H, Ar, H₇), 7.82 (d, 1H, H_a, ³J = 16.2 Hz), 7.88 (dd, 1H, Ar, H₈), 8.00 (dd, 1H, Ar, H₅), 12.10 (s, 1H, NH). ¹³C NMR (75.03 MHz, DCCl₃, δ ppm, 25 °C): C(OCH₃): 55.5, 55.4; C-2: 110.9; C-5: 112.2; C-b: 117.9; C-8': 118.8; C-5': 120.9; C-6': 121.3; C-7': 124.1; C-6: 124.7; C-1: 129.0; C-4': 131.5; C-a: 142.8; C-2': 148.5; C-3: 148.9; C-4: 151.0; C-9': 157.9; C(C=O): 164.2. ESI-MS (*m/z*): 341.4021 [M + H]⁺. Anal. calcd. for C₁₈H₁₆N₂O₃S (340.40): C, 63.51; H, 4.74. Found: C, 63.55; H, 4.70.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(2,5-dimethoxyphenyl)acrylamide
(Table 2, 3g)**

The mixture was irradiated for 2 min. Yield: 92%. Mp 173–174 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 3.77, 3.86 (s, 3H, OCH₃), 6.92 (d, 1H, H_b,

$^3J = 15.7$ Hz), 7.07 (dd, 1H, Ar, H₄), 7.12 (d, 1H, Ar, H₃), 7.18 (d, 1H, Ar, H₆), 7.31 (dt, 1H, Ar, H₆'), 7.44 (dt, 1H, Ar, H₇'), 7.76 (d, 1H, H_a, $^3J = 15.7$ Hz), 7.91 (dd, 1H, Ar, H₈'), 7.99 (dd, 1H, Ar, H₅'), 12.40 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH₃): 55.4, 56.0; C-3: 113.3; C-b: 117.1; C-6: 117.9; C-8': 118.8; C-4: 120.2; C-5': 121.0; C-6': 121.4; C-7': 124.5; C-1: 126.2; C-4': 131.5; C-a: 139.9; C-2': 148.5; C-2: 153.0; C-5: 152.4; C-9': 157.9; C(C=O): 164.3. ESI-MS (m/z): 341.4003 $[\text{M} + \text{H}]^+$. Anal. calcd. for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_3\text{S}$ (340.40): C, 63.51; H, 4.74. Found: C, 63.52; H, 4.71.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(4-methoxyphenyl)acrylamide
(Table 2, 3h)**

The mixture was irradiated 2 min. Yield: 90%. Mp 221–222 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 3.82 (s, 3H, OCH₃), 6.82 (d, 1H, H_b, $^3J = 16.1$ Hz), 7.04 (d, 2H, Ar, H₃, H₅), 7.31 (dt, 1H, Ar, H₆'), 7.44 (dt, 1H, Ar, H₇'), 7.62 (d, 2H, Ar, H₂, H₆), 7.75 (d, 1H, H_a, $^3J = 16.1$ Hz), 7.87 (dd, 1H, Ar, H₈'), 7.98 (dd, 1H, Ar, H₅'), 12.45 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH₃): 55.2; C-3, C-5: 114.4; C-b: 117.6; C-8': 119.6; C-5': 120.2; C-6': 121.1; C-7': 124.2; C-1: 126.7; C-2, C-6: 129.7; C-4': 131.5; C-a: 142.7; C-2': 148.5; C-9': 158.0; C-4: 161.1; C(C=O): 164.1. ESI-MS (m/z): 311.3725 $[\text{M} + \text{H}]^+$. Anal. calcd. for $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_2\text{S}$ (310.37): C, 65.79; H, 4.55. Found: C, 65.83; H, 4.49.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(4-ethoxyphenyl)acrylamide
(Table 2, 3i)**

The mixture was irradiated 2 min. Yield: 92%. Mp 229–230 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 1.35 (t, 3H, OCH₂CH₃), 4.10 (q, 2H, OCH₂CH₃), 6.81 (d, 1H, H_b, $^3J = 15.8$ Hz), 7.06 (d, 2H, Ar, H₃, H₅), 7.30 (dt, 1H, Ar, H₆'), 7.42 (ddd, 1H, Ar, H₇'), 7.60 (d, 2H, Ar, H₂, H₆), 7.74 (d, 1H, H_a, $^3J = 15.8$ Hz), 7.86 (dd, 1H, Ar, H₈'), 7.98 (dd, 1H, Ar, H₅'), 12.39 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH₂CH₃): 14.3; C(OCH₂CH₃): 63.2; C-5: 114.9; C-b: 117.2; C-8': 119.8; C-5': 120.7; C-6': 121.4; C-7': 123.8; C-1: 126.5; C-2, C-6: 129.9; C-4': 131.3; C-a: 142.7; C-2': 148.6; C-9': 159.0; C-4: 160.3; C(C=O): 164.5. ESI-MS (m/z): 325.3999 $[\text{M} + \text{H}]^+$. Anal. calcd. for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_2\text{S}$ (324.40): C, 66.64; H, 4.97. Found: C, 66.79; H, 4.86.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(4-propoxyphenyl)acrylamide
(Table 2, 3j)**

The mixture was irradiated for 2 min. Yield: 91%. Mp 218–220 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 0.99 (t, 3H, OCH₂CH₂CH₃), 1.75 (m, 2H, OCH₂CH₂CH₃), 4.00 (t, 2H, OCH₂CH₂CH₃), 6.81 (d, 1H, H_b, $^3J = 16.1$ Hz), 7.02 (d, 2H, Ar, H₃, H₅), 7.32 (dt, 1H, Ar, H₆'), 7.43 (dt, 1H, Ar, H₇'), 7.60 (d, 2H, Ar, H₂, H₆), 7.74 (d, 1H, H_a, $^3J = 16.1$ Hz), 7.85 (dd, 1H, Ar, H₈'), 7.97 (dd, 1H, Ar, H₅'), 12.35 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH₂CH₂CH₃): 10.1; C(OCH₂CH₂CH₃): 21.8; C(OCH₂CH₂CH₃): 69.0; C-5: 114.9; C-b: 117.5; C-8': 119.1; C-5': 120.4; C-6': 121.2; C-7': 124.1; C-1: 126.5; C-2,

C-6: 129.7; C-4': 131.5; C-a: 142.8; C-2': 148.8; C-9': 158.2; C-4: 160.5; C(C=O): 164.9. ESI-MS (m/z): 339.4231 $[M + H]^+$. Anal. calcd. for $C_{19}H_{18}N_2O_2S$ (338.42): C, 67.43; H, 5.36. Found: C, 67.52; H, 5.44.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(4-butoxyphenyl)acrylamide
(Table 2, 3k)**

The mixture was irradiated for 2 min. Yield: 92%. Mp 196–197 °C. 1H NMR (300.13 MHz, $DCCl_3$, δ ppm, 25 °C): 0.94 (m, 3H, $OCH_2CH_2CH_2CH_3$), 1.44 (m, 2H, $OCH_2CH_2CH_2CH_3$), 1.72 (m, 2H, $OCH_2CH_2CH_2CH_3$), 4.03 (t, 2H, $OCH_2CH_2CH_2CH_3$), 6.80 (d, 1H, H_b , $^3J = 16.0$ Hz), 7.05 (d, 2H, Ar, H_3 , H_5), 7.33 (dt, 1H, Ar, H_6), 7.45 (ddd, 1H, Ar, H_7'), 7.61 (d, 2H, Ar, H_2 , H_6), 7.75 (d, 1H, H_a , $^3J = 16.0$ Hz), 7.84 (dd, 1H, Ar, H_8), 7.99 (dd, 1H, Ar, H_5), 12.40 (s, 1H, NH). ^{13}C NMR (75.03 MHz, $DCCl_3$, δ ppm, 25 °C): $C(OCH_2CH_2CH_2CH_3)$: 13.8; $C(OCH_2CH_2CH_2CH_3)$: 18.8; $C(OCH_2CH_2CH_2CH_3)$: 30.8; $C(OCH_2CH_2CH_2CH_3)$: 67.6; C-5: 115.2; C-b: 117.8; C-8': 119.4; C-5': 120.6; C-6': 121.5; C-7': 123.9; C-1: 126.1; C-2, C-6: 130.0; C-4': 131.8; C-a: 143.1; C-2': 148.9; C-9': 158.3; C-4: 160.9; C(C=O): 164.5. ESI-MS (m/z): 353.4532 $[M + H]^+$. Anal. calcd. for $C_{20}H_{20}N_2O_2S$ (352.45): C, 68.16; H, 5.72. Found: C, 67.98; H, 5.88.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(4-methylphenyl)acrylamide
(Table 2, 3l)**

The mixture was irradiated 3 min. Yield: 89%. Mp 241–242 °C. 1H NMR (300.13 MHz, $DCCl_3$, δ ppm, 25 °C): 2.35 (CH_3), 6.87 (d, 1H, H_b , $^3J = 16.2$ Hz), 7.29 (d, 2H, Ar, H_3 , H_5), 7.34 (dt, 1H, Ar, H_6), 7.42 (ddd, 1H, Ar, H_7'), 7.53 (d, 2H, Ar, H_2 , H_6), 7.79 (d, 1H, H_a , $^3J = 16.2$ Hz), 7.89 (dd, 1H, Ar, H_8), 7.95 (dd, 1H, Ar, H_5), 12.50 (s, 1H, NH). ^{13}C NMR (75.03 MHz, $DCCl_3$, δ ppm, 25 °C): $C(CH_3)$: 20.8; C-b: 118.1; C-8': 119.3; C-5': 120.5; C-6': 121.3; C-7': 123.8; C-2, C-6: 128.1; C-3, C-5: 128.9; C-4': 131.9; C-1: 138.1; C-a: 142.3; C-4: 147.8; C-2': 148.8; C-9': 157.5; C(C=O): 163.9. ESI-MS (m/z): 295.3704 $[M + H]^+$. Anal. calcd. for $C_{17}H_{14}N_2OS$ (294.37): C, 69.36; H, 4.79. Found: C, 69.40; H, 4.72.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(4-ethylphenyl)acrylamide
(Table 2, 3m)**

The mixture was irradiated for 3 min. Yield: 90%. Mp 166–167 °C. 1H NMR (300.13 MHz, $DCCl_3$, δ ppm, 25 °C): 1.20 (t, 2H, CH_2CH_3), 3.25 (q, 2H, CH_2CH_3), 6.92 (d, 1H, H_b , $^3J = 16.3$ Hz), 7.26 (d, 2H, Ar, H_3 , H_5), 7.35 (dt, 1H, Ar, H_6), 7.44 (ddd, 1H, Ar, H_7'), 7.58 (d, 2H, Ar, H_2 , H_6), 7.78 (d, 1H, H_a , $^3J = 16.3$ Hz), 7.85 (dd, 1H, Ar, H_8), 7.99 (dd, 1H, Ar, H_5), 12.48 (s, 1H, NH). ^{13}C NMR (75.03 MHz, $DCCl_3$, δ ppm, 25 °C): $C(CH_2CH_3)$: 15.0; $C(CH_2CH_3)$: 27.9; C-b: 118.3; C-8': 119.7; C-5': 120.8; C-6': 121.0; C-7': 124.2; C-2, C-6: 128.0; C-3, C-5: 128.3; C-4': 131.6; C-1: 138.8; C-a: 142.9; C-4: 146.1; C-2': 148.3; C-9': 157.9; C(C=O): 163.4. ESI-MS (m/z): 309.4015 $[M + H]^+$. Anal. calcd. for $C_{18}H_{16}N_2OS$ (308.40): C, 70.10; H, 5.23. Found: C, 70.27; H, 5.11.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(2-chlorophenyl)acrylamide
(Table 2, 3n)**

The mixture was irradiated for 2 min. Yield: 91%. Mp 235–237 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 6.74 (d, 1H, H_b, ³J = 15.9 Hz), 7.17 (ddd, 1H, Ar, H₄), 7.29 (ddd, 1H, Ar, H₆), 7.34 (ddd, 1H, Ar, H₅), 7.41 (ddd, 1H, Ar, H₇'), 7.51 (dd, 1H, Ar, H₆), 7.70 (d, 1H, H_a, ³J = 15.9 Hz), 7.75 (dd, 1H, Ar, H₃), 7.80 (dd, 1H, Ar, H₈'), 7.91 (dd, 1H, Ar, H₅'), 12.47 (s, 1H, NH). ¹³C NMR (75.03 MHz, DCCl₃, δ ppm, 25 °C): C-b: 117.9; C-8': 119.0; C-5': 120.3; C-6': 121.1; C-7': 124.3; C-5: 127.1; C-6: 128.7; C-4: 130.0; C-3: 130.8; C-4': 132.1; C-2: 134.4; C-1: 135.1; C-a: 142.1; C-2': 147.9; C-9': 157.1; C(C=O): 163.4. ESI-MS (*m/z*): 315.7919 [M + H]⁺. Anal. calcd. for C₁₆H₁₁ClN₂OS (314.79): C, 61.05; H, 3.52. Found: C, 61.10; H, 3.48.

**(2E)-N-1,3-Benzothiazol-2-yl-3-(4-chlorophenyl)acrylamide
(Table 2, 3o)**

The mixture was irradiated for 2 min. Yield: 90%. Mp 242–243 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 6.77 (d, 1H, H_b, ³J = 15.8 Hz), 7.27 (ddd, 1H, Ar, H₆), 7.37 (ddd, 1H, Ar, H₇'), 7.42–7.45 (m 2H, Ar, H₃, H₅), 7.50–7.52 (m, 2H, Ar, H₂, H₆), 7.71 (d, 1H, H_a, ³J = 15.8 Hz), 7.81 (dd, 1H, Ar, H₈'), 7.96 (dd, 1H, Ar, H₅'), 12.40 (s, 1H, NH). ¹³C NMR (75.03 MHz, DCCl₃, δ ppm, 25 °C): C-b: 119.1; C-8': 119.5; C-5': 120.8; C-6': 121.0; C-7': 124.5; C-3, C-5: 128.8; C-2, C-6: 130.2; C-4': 131.1; C-1: 134.9; C-4: 135.6; C-a: 142.8; C-2': 147.4; C-9': 157.5; C(C=O): 164.2. ESI-MS (*m/z*): 315.7918 [M + H]⁺. Anal. calcd. for C₁₆H₁₁ClN₂OS (314.79): C, 61.05; H, 3.52. Found: C, 61.08; H, 3.50.

(2E)-3-(3,4-Dimethoxyphenyl)-N-(6-methoxy-1,3-benzothiazol-2-yl)acrylamide (Table 2, 3p)

The mixture was irradiated for 2 min. Yield: 93%. Mp 228–229 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 3.81, 3.82, 3.84 (s, 3H, OCH₃), 6.68 (dd, 1H, Ar, H₇), 6.82 (d, 1H, H_b, ³J = 15.8 Hz), 7.05 (d, 1H, Ar, H₅), 7.16 (dd, 1H, Ar, H₆), 7.24 (d, 1H, Ar, H₂), 7.57 (d, 1H, Ar, H₅'), 7.65 (d, 1H, Ar, H₈'), 7.73 (d, 1H, H_a, ³J = 15.8 Hz), 12.33 (s, 1H, NH). ¹³C NMR (75.03 MHz, DCCl₃, δ ppm, 25 °C): C(OCH₃): 55.3, 55.4, 55.5, C-5': 104.6; C-2: 110.5; C-5: 111.8; C-7', 114.7; C-b: 117.9; C-8', 120.9; C-6: 123.1; C-1: 126.9; C-4', 132.9; C-a: 142.7; C-2': 145.8; C-3: 148.9; C-9': 150.3; C-4: 151.0; C-6': 156.0; C(C=O): 163.9. ESI-MS (*m/z*): 371.4139 [M + H]⁺. Anal. calcd. for C₁₉H₁₈N₂O₄S (370.42): C, 61.61; H, 4.90. Found: C, 61.78; H, 4.83.

(2E)-3-(3,4-Dimethoxyphenyl)-N-(6-ethoxy-1,3-benzothiazol-2-yl)acrylamide (Table 2, 3q)

The mixture was irradiated for 2 min. Yield: 91%. Mp 224–225 °C. ¹H NMR (300.13 MHz, DCCl₃, δ ppm, 25 °C): 1.35 (t, 3H, OCH₂CH₃), 3.82, 3.84 (s, 3H, OCH₃), 4.85 (q, 2H, OCH₂CH₃), 6.69 (dd, 1H, Ar, H₇), 6.80 (d, 1H, H_b,

$^3J = 15.2$ Hz), 7.03 (d, 1H, Ar, H₅), 7.18 (dd, 1H, Ar, H₆), 7.28 (d, 1H, Ar, H₂), 7.59 (d, 1H, Ar, H₅), 7.64 (d, 1H, Ar, H₈), 7.75 (d, 1H, H_a, $^3J = 15.2$ Hz), 12.31 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH₂CH₃): 14.3; C(OCH₃): 55.4, 55.5; C(OCH₂CH₃): 63.5; C-5': 105.7; C-2: 110.8; C-5: 111.9; C-7': 114.9; C-b: 117.5; C-8': 120.7; C-6: 122.9; C-1: 127.0; C-4': 132.6; C-a: 142.4; C-2': 145.3; C-3: 148.8; C-9': 150.2; C-4: 150.8; C-6': 152.9; C(C=O): 163.8. ESI-MS (m/z): 385.4508 [M + H]⁺. Anal. calcd. for C₂₀H₂₀N₂O₄S (384.45): C, 62.48; H, 5.24. Found: C, 62.35; H, 5.38.

(2E)-3-(3,4-Dimethoxyphenyl)-N-(6-methyl-1,3-benzothiazol-2-yl)acrylamide (Table 2, 3r)

The mixture was irradiated for 2 min. Yield: 89%. Mp 227–228 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 2.45 (s, 3H, CH₃), 3.80, 3.85 (s, 3H, OCH₃), 6.83 (d, 1H, H_b, $^3J = 15.6$ Hz), 7.03 (d, 1H, Ar, H₅), 7.17 (dd, 1H, Ar, H₆), 7.21 (dd, 1H, Ar, H₇), 7.26 (d, 1H, Ar, H₂), 7.60 (d, 1H, Ar, H₈), 7.71 (d, 1H, H_a, $^3J = 15.6$ Hz), 7.78 (d, 1H, Ar, H₅), 12.45 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(CH₃): 20.6; C(OCH₃): 55.4, 55.3; C-2: 110.9; C-5: 111.8; C-b: 117.3; C-8': 119.7; C-5': 120.8; C-6: 123.9; C-7': 126.9; C-1: 127.2; C-6': 131.6; C-4': 132.6; C-a: 142.6; C-2': 146.4; C-3: 148.9; C-4: 150.9; C-9': 152.0; C(C=O): 163.9. ESI-MS (m/z): 355.4249 [M + H]⁺. Anal. calcd. for C₁₉H₁₈N₂O₃S (354.42): C, 64.39; H, 5.12. Found: C, 64.41; H, 5.19.

(2E)-3-(3,4-Dimethoxyphenyl)-N-[6-(methylsulfonyl)-1,3-benzothiazol-2-yl]acrylamide (Table 2, 3s)

The mixture was irradiated for 2 min. Yield: 90%. Mp 196–197 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 3.12 (s, 3H, CH₃), 3.83, 3.85 (s, 3H, OCH₃), 6.77 (d, 1H, H_b, $^3J = 15.9$ Hz), 7.02 (d, 1H, Ar, H₅), 7.14 (dd, 1H, Ar, H₆), 7.24 (d, 1H, Ar, H₂), 7.55 (dd, 1H, Ar, H₇), 7.77 (d, 1H, H_a, $^3J = 15.9$ Hz), 7.89 (d, 1H, Ar, H₈), 8.23 (d, 1H, Ar, H₅), 12.46 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(CH₃): 40.9; C(OCH₃): 55.6, 55.7; C-2: 110.6; C-5: 111.7; C-b: 116.8; C-5': 121.5; C-6: 122.7; C-8': 123.1; C-7': 125.6; C-1: 127.1; C-4': 133.5; C-6': 134.9; C-a: 143.9; C-2': 147.7; C-3: 149.1; C-4: 151.3; C-9': 159.5; C(C=O): 164.7. ESI-MS (m/z): 419.4901 [M + H]⁺. Anal. calcd. for C₁₉H₁₈N₂O₅S₂ (418.49): C, 54.53; H, 4.34. Found: C, 54.55; H, 4.29.

(2E)-3-(3,4-Dimethoxyphenyl)-N-(6-nitro-1,3-benzothiazol-2-yl)acrylamide (Table 2, 3t)

The mixture was irradiated for 2 min. Yield: 93%. Mp 230–232 °C. ^1H NMR (300.13 MHz, DCCl_3 , δ ppm, 25 °C): 3.81, 3.83 (s, 3H, OCH₃), 6.84 (d, 1H, H_b, $^3J = 16.1$ Hz), 7.04 (d, 1H, Ar, H₅), 7.19 (dd, 1H, Ar, H₆), 7.25 (d, 1H, Ar, H₂), 7.77 (d, 1H, H_a, $^3J = 16.1$ Hz), 7.88 (d, 1H, Ar, H₆), 8.08 (dd, 1H, Ar, H₇), 8.67 (d, 1H, Ar, H₅), 12.35 (s, 1H, NH). ^{13}C NMR (75.03 MHz, DCCl_3 , δ ppm, 25 °C): C(OCH₃): 55.8, 55.6; C-2: 110.8; C-5: 112.0; C-b: 117.0; C-5': 119.1; C-7': 120.6; C-8': 121.9; C-6: 123.2; C-1: 127.0; C-4': 131.8; C-6': 140.4; C-a: 140.9; C-2':

147.1; C-3: 148.6; C-4: 150.7; C-9': 158.8; C(C=O): 161.9. ESI-MS (m/z): 386.3925 $[M + H]^+$. Anal. calcd. for $C_{18}H_{15}N_3O_5S$ (385.39): C, 56.10; H, 3.92. Found: C, 55.97; H, 4.05.

(2E)-N-(4-Chloro-1,3-benzothiazol-2-yl)-3-(3,4-dimethoxyphenyl)acrylamide (Table 2, 3u)

The mixture was irradiated for 3 min. Yield: 88%. Mp 158–159 °C. 1H NMR (300.13 MHz, $DCCl_3$, δ ppm, 25 °C): 3.78, 3.80 (s, 3H, OCH_3), 6.77 (d, 1H, H_b , $^3J = 15.9$ Hz), 6.87 (d, 1H, Ar, H_5), 7.00 (dd, 1H, Ar, $H_{7'}$), 7.20 (dd, 1H, Ar, H_6), 7.27 (d, 1H, Ar, H_2), 7.36 (t, 1H, Ar, $H_{6'}$), 7.75 (d, 1H, H_a , $^3J = 15.9$ Hz), 7.79 (dd, 1H, Ar, $H_{5'}$), 12.52 (s, 1H, NH). ^{13}C NMR (75.03 MHz, $DCCl_3$, δ ppm, 25 °C): C(OCH_3): 55.8, 55.9; C-2: 110.7; C-5: 111.6; C-b: 116.8; C-5': 120.0; C-8': 121.0; C-6': 121.9; C-6: 122.7; C-7': 124.5; C-1: 127.1; C-4': 132.4; C-a: 142.9; C-2': 147.8; C-3: 149.2; C-4: 151.4; C-9': 157.5; C(C=O): 164.7. ESI-MS (m/z): 375.8411 $[M + H]^+$. Anal. calcd. for $C_{18}H_{15}ClN_2O_3S$ (374.84): C, 57.68; H, 4.03. Found: C, 57.55; H, 4.18.

(2E)-3-(3,4-Dimethoxyphenyl)-N-(4-methyl-1,3-benzothiazol-2-yl)acrylamide (Table 2, 3v)

The mixture was irradiated 3 min. Yield: 89%. Mp 204–205 °C. 1H NMR (300.13 MHz, $DCCl_3$, δ ppm, 25 °C): 2.51 (s, 3H, CH_3), 3.82, 3.84 (s, 3H, OCH_3), 6.69 (dd, 1H, Ar, $H_{7'}$), 6.78 (d, 1H, H_b , $^3J = 15.8$ Hz), 6.99 (d, 1H, Ar, H_5), 7.05 (t, 1H, Ar, $H_{6'}$), 7.12 (dd, 1H, Ar, H_6), 7.29 (d, 1H, Ar, H_2), 7.68 (dd, 1H, Ar, $H_{5'}$), 7.74 (d, 1H, H_a , $^3J = 15.8$ Hz), 12.51 (s, 1H, NH). ^{13}C NMR (75.03 MHz, $DCCl_3$, δ ppm, 25 °C): C(CH_3): 17.9; C(OCH_3): 55.6, 55.4; C-2: 110.9; C-5: 111.7; C-b: 116.9; C-6': 119.0; C-6: 122.0; C-5': 123.4; C-7': 126.6; C-1: 128.1; C-8': 129.6; C-4': 131.8; C-a: 142.1; C-2': 145.1; C-3: 149.8; C-4: 150.9; C-9': 155.2; C(C=O): 164.1. ESI-MS (m/z): 355.4199 $[M + H]^+$. Anal. calcd. for $C_{19}H_{18}N_2O_3S$ (354.42): C, 64.39; H, 5.12. Found: C, 64.47; H, 5.19.

(2E)-N,3-Diphenylacrylamide (Table 2, 3w)

The mixture was irradiated for 1 min. Yield: 98%. Mp 293–295 °C. 1H NMR (300.13 MHz, $DCCl_3$, δ ppm, 25 °C): 6.81 (d, 1H, H_b , $^3J = 15.6$ Hz), 7.09 (dt, 1H, Ar, H_4), 7.25 (d, 1H, Ar, H_4), 7.31 (dd, 2H, Ar, $H_{3'}$, $H_{5'}$), 7.44 (dd, 2H, Ar, H_3 , H_5), 7.71 (dd, 2H, Ar, $H_{2'}$, $H_{6'}$), 7.77 (d, 1H, H_a , $^3J = 15.6$ Hz), 12.00 (s, 1H, NH). ^{13}C NMR (75.03 MHz, $DCCl_3$, δ ppm, 25 °C): C-b: 118.3; C-4': 122.2; C-2', C-6': 123.3; C-3', C-5': 127.8; C-3, C-2, C-6: 128.3; C-5: 128.9; C-4: 129.9; C-1: 134.7; C-1': 139.2; C-a: 142.1; C(C=O): 165.9. ESI-MS (m/z): 224.2719 $[M + H]^+$. Anal. calcd. for $C_{15}H_{13}NO$ (223.27): C, 80.69; H, 5.87. Found: C, 80.67; H, 5.88.

4-{[(2E)-3-Phenylprop-2-enoyl]amino}benzoic acid (Table 2, 3x)

The mixture was irradiated for 1.5 min. Yield: 97%. Mp 152–154 °C. 1H NMR (300.13 MHz, $DCCl_3$, δ ppm, 25 °C): 6.85 (d, 1H, H_b , $^3J = 15.9$ Hz), 7.39 (dd, 1H, Ar,

H₄), 7.47 (dd, 2H, Ar, H₃, H₅), 7.57 (d, 2H, Ar, H₂, H₆), 7.63 (dd, 2H, Ar, H₂, H₆), 7.87 (d, 1H, H_a, ³J = 15.9 Hz), 7.91 (d, 2H, Ar, H₃, H₅), 12.20 (s, 1H, NH). ¹³C NMR (75.03 MHz, DCCl₃, δ ppm, 25 °C): C-b: 118.5; C-2', C-6': 121.8; C-4': 125.2; C-2, C-6: 127.8; C-3, C-5: 129.0; C-4: 130.3; C-3', C-5': 131.4; C-1: 134.5; C-a: 141.9; C-1': 143.2; C(C=O): 163.8; C(COOH): 166.9. ESI-MS (m/z): 268.2779 [M + H]⁺. Anal. calcd. for C₁₆H₁₃NO₃ (267.28): C, 71.90; H, 4.90. Found: C, 71.94; H, 4.87.

ACKNOWLEDGMENTS

The authors thank Antonio Marchal Igrain and Juan Jesús López González from University of Jaén, Spain, for their kindness for measuring NMR spectra.

REFERENCES

1. Aoyagi, Y.; Masuko, N.; Ohkubo, S.; Kitade, M.; Nagai, K.; Okazaki, S.; Wierzba, K.; Terada, T.; Sugimoto, Y.; Yamada, Y. A novel cinnamic acid derivative that inhibits Cdc25 dual-specificity phosphatase activity. *Cancer Sci.* **2005**, *96* (9), 614–619.
2. Kumar, A.; Katiyar, S. B.; Agarwal, A.; Chauhan, P. M. S. Current trends in antimalarial chemotherapy. *Drugs Fut.* **2003**, *28* (3), 243–251.
3. Hee Kong, Y.; Ock Jo, Y.; Cho, C.; Son, D.; Park, S.; Rho, J.; Yoon Choi, S. Inhibitory effects of cinnamic acid on melanin biosynthesis in skin. *Biol. Pharm. Bull.* **2008**, *31* (5), 946–948.
4. Lee, S.; Han, J. M.; Kim, H.; Kim, E.; Jeong, T. S.; Lee, W. S.; Cho, K. H. Synthesis of cinnamic acid derivatives and their inhibitory effects on LDL-oxidation, acyl-CoA: cholesterol acyltransferase-1 and -2 activity, and decrease of HDL-particle size. *Bioorg. Med. Chem. Lett.* **2004**, *14* (18), 4677–4681.
5. Yesilada, A.; Zurlo, E. 3,4-Dimethoxycinnamic acid tertiary amides: Síntesis and evaluation of anti-inflammatory and analgesic activities. *Farmaco* **1996**, *8* (9), 595–599.
6. Pardo, G. L.; Inada, N. M.; Pellón, R. F.; Docampo, M. L.; Fascio, M. L.; D' Accorso, N. B.; Vercesi, A. E. In vitro effect of a new cinnamic acid derivative against the epimastigote form of *Trypanosoma cruzi*. *Arzneimittelforschung* **2009**, *59* (4), 207–211.
7. Wiesner, J.; Mitsch, A.; Jomaa, H.; Schlitzer, M. Structure–activity relationships of novel anti-malarial agents, part 7: N-(3-Benzoyl-4-tolylacetylaminophenyl)-3-(5-aryl-2-furyl)acrylic acid amides with polar moieties. *Bioorg. Med. Chem. Lett.* **2003**, *13*, 2159–2161.
8. Spasova, M.; Philipov, S.; Nikolaeva-Glomb, L.; Galabov, A.; Milkova, T. Cinnamoyl and hydroxycinnamoyl amides of glaucine and their biological activity. *Bioorg. Med. Chem.* **2008**, *16*, 7457–7461.
9. Webb, C. N. Benzanilide. *Org. Synth.* **1941**, *1*, 82–88.
10. Kivinen, J. *The Chemistry of Acyl Halides*; Interscience: New York, 1972; p. 170–230.
11. Nomura, E.; Hosoda, A.; Mori, H.; Taniguchi, H. Rapid base-catalyzed decarboxylation and amide-forming reaction of substituted cinnamic acids via microwave heating. *Green Chem.* **2005**, *7*, 863–866.
12. Pellón, R. F.; Docampo, M.; Fascio, M. Mild method for Ullmann reaction of 2-chlorobenzoic acids and aminothiazoles or aminobenzothiazoles under ultrasonic irradiation. *Synth. Commun.* **2007**, *37* (11), 1853–1864; (b) Docampo, M.; Pellón, R. F.; Estevez-Braun, A. G.; Ravelo, A. Ultrasound-promoted reaction of 2-chlorobenzoic acids and aliphatic amines. *Eur. J. Org. Chem.* **2007**, *24*, 4111–4115; (c) Pellón, R. F.; Martín,

- A.; Mesa, M.; Docampo, M. L.; Gómez, V. Microwave-assisted synthesis of 2-phenoxybenzoic acids. *J. Chem. Res. Synop.* **2006**, 8, 527–529; (d) Martín, A.; Mesa, M.; Docampo, M. L.; Gómez, V.; Pellón, R. F. Fast synthesis of substituted N-phenylanthranilic acids using Ullmann condensation under microwave irradiation in dry media. *Synth. Commun.* **2006**, 36 (3), 271–277.
13. Chunavala, K. C.; Joshi, G.; Suresh, E.; Adimurthy, S. Thermal and microwave-assisted rapid synthesis of substituted imidazo[1,2-a]pyridines under solvent- and catalyst-free conditions. *Synthesis* **2011**, 4, 635–641.
14. Pellón, R. F.; Martín, A.; Docampo, M.; Mesa, M. Microwave-promoted Ullmann condensation of 2-aminopyridines with 2-chlorobenzoic acids. *Synth. Commun.* **2006**, 36 (6), 1715–1719.