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Effect of Microwave Irradiation on the Direction and Stereochemistry of Rodionov and Michael Reactions

N. N. Romanova, A. G. Gravis, P. V. Kudan, I. F. Leshcheva, and N. V. Zyk

Faculty of Chemistry, Moscow State University, Vorob'evy gory, Moscow, 119899 Russia
e-mail: rom@org.chem.msu.su

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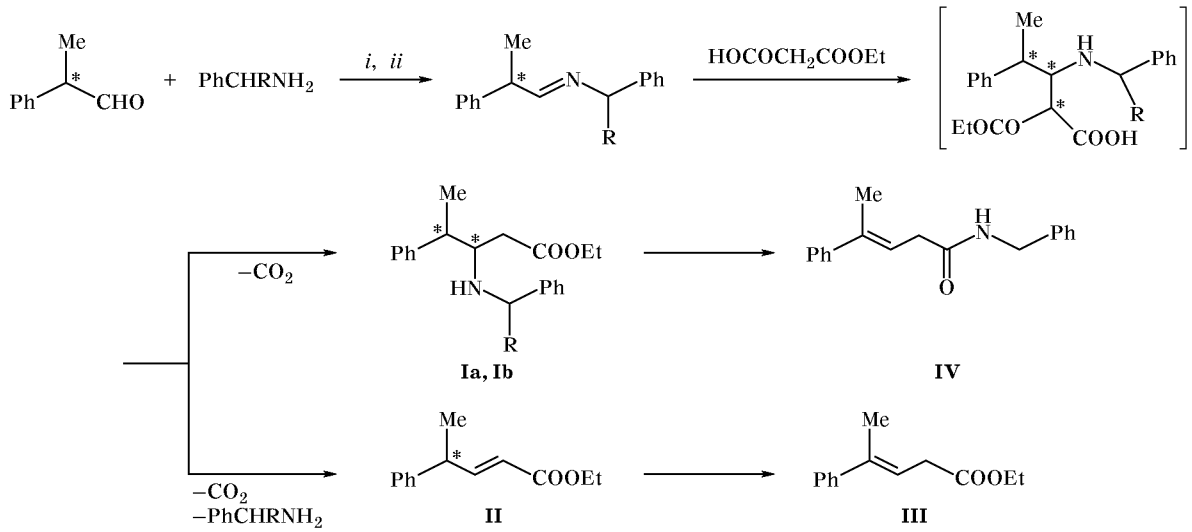
Abstract—The reactions of 2-phenylpropanal with ethyl hydrogen malonate and benzylamine (or benzylammonium acetate) and of ethyl 4-phenyl-2-pentenoate with benzylamine take different pathways, depending on the conditions.

Microwave irradiation (MW) is now widely used in organic synthesis as a method of activation of various reactions. In particular, this technique makes it possible to shorten the reaction time to a few minutes and in most cases considerably increases the yield of final products, as compared to traditional synthetic procedures [1, 2]. Up to now, there is no general theory of the effect of MW irradiation on chemical processes, but the presence or absence of a specific MW effect in organic reactions is extensively discussed in the literature [2].

We previously showed that β-aryl-β-amino acid ethyl esters can be synthesized within a short time by three-component condensation of the corresponding aromatic aldehyde, amine (or ammonium acetate), and ethyl hydrogen malonate (so-called Rodionov reaction) under MW irradiation in the absence of a solvent [3]. We have found that this technique can also be applied to the synthesis of N-substituted γ-phenyl-β-amino acid esters from 2-phenylpropanal.

Comparison of the chemical and stereochemical outputs of the same reaction carried out under conditions of microwave and thermal activation would reveal the presence or absence of a specific MW effect. For this purpose, as model reactions we studied the Rodionov condensation of benzylamine (or

Scheme 1.



I, R = H (**a**), Me (**b**); *i*: reaction with benzylammonium acetate; *ii* reaction with benzylamine.

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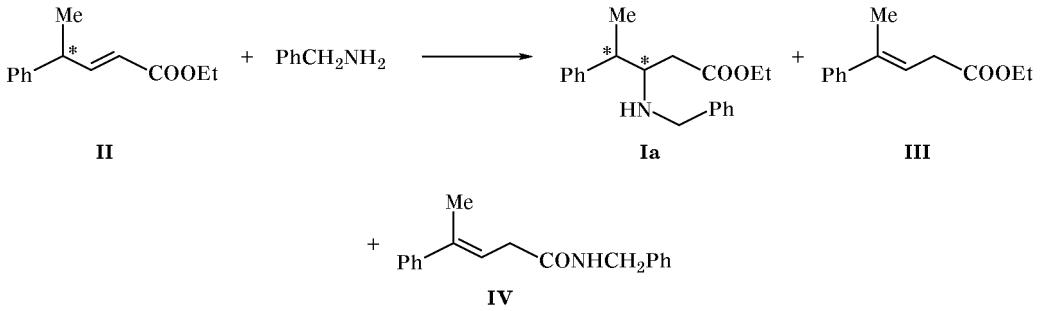
Scheme 2.



II **Ia** **III** **IV**

benzylammonium acetate), ethyl hydrogen malonate, and 2-phenylpropanal, which should yield ethyl 3-benzylamino-4-phenylpentanoate and ethyl 4-phenyl-2-pentenoate, and conjugate Michael addition of benzylamine to ethyl 4-phenyl-2-pentenoate, which should lead to formation of ethyl 3-benzylamino-4-phenylpentanoate.

The three-component condensation follows a mechanism which was proposed for the first time by Rodionov [4] and was confirmed by our studies [3]. Initially, 2-phenylpropanal reacts with benzylamine to give the corresponding intermediate Schiff base which then reacts with ethyl hydrogen malonate at the carbon atom of the CH=N moiety. The resulting adduct undergoes either decarboxylation to yield β-amino ester **I** or simultaneous decarboxylation and elimination of the amine molecule; in the latter case, the product is ethyl 4-phenyl-2-pentenoate (**II**) (Scheme 1). In addition, some side processes are possible. For example, the thermal reaction is accompanied by migration of the double C=C bond in ester **II** to the β,γ-position, as well as by ammonolysis of the 4-phenyl-3-pentenoic acid ester **III** thus formed to afford *N*-benzyl-4-phenyl-3-pentenamide (**IV**) (Scheme 1, see table).

The proposed mechanism of the Rodionov reaction is supported by the following. When the corresponding Schiff base is used instead of aldehyde and amine, almost the same chemical and stereochemical results are obtained [3]. Our experiments also showed that no condensation of ethyl hydrogen malonate with 2-phenylpropanal or aromatic aldehydes occurs under analogous conditions (the reaction is possible when the initial compounds are applied to silica gel [5] or bentonite [6]). Schiff bases derived from lower aliphatic aldehydes are unstable, and they readily undergo polymerization even under usual conditions. Therefore, ethyl *N*-(1-phenylethyl)-3-aminobutyrate is not formed on MW irradiation of a mixture of acetaldehyde with (*S*)-α-phenylethylamine and ethyl hydrogen malonate. β-Amino esters are stable to both temperature and MW irradiation, and no elimination of amine from amino ester occurs. Moreover, under similar conditions, the Michael reaction leads to different chemical and stereochemical results (Scheme 2; see table, run nos. 1, 4, 6).

Products of the Rodionov (Scheme 1) and Michael reactions (Scheme 2)

Run no.	Reaction	R	Conditions	Yield, %			
				I	**II**	**III**	**IV**
1	Rodionov (*i*)	H	MW	38, de 35%	60, *E*/*Z* > 99:1	0	0
2	Rodionov (*i*)	H	Δ	0	20, *E*/*Z* = 0:1	54, *E*/*Z* = 12:1	0
3[a]	Rodionov (*i*)	Me	MW	37[b]	58, *E*/*Z* > 99:1	0	0
4	Rodionov (*ii*)	H	MW	16, de 15%	61, *E*/*Z* = 23:1	3[c]	0
5	Rodionov (*ii*)	H	Δ	4, de 20%	6[c]	58, *E*/*Z* = 6:1	25, *E*/*Z* = 14:1
6	Michael	–	MW	<4, de 9%	7[c]	56, *E*/*Z* = 16:1	0
7	Michael	–	Δ	0	5, *E*/*Z* = 10:1	19, *E*/*Z* = 2:1	39, *E*/*Z* = 4:1

[a] *N*-Benzylacetamide was formed in 22% yield.

[b] Diastereoisomer ratio 8:5:4:3.

[c] The *trans*/*cis* ratio was not determined.

Microwave irradiation of a mixture of equimolar amounts of benzylammonium acetate, ethyl hydrogen malonate, and 2-phenylpropanal over a period of 10 min (Scheme 1) resulted in formation of β-amino ester **Ia** in 38% yield; analogous reaction with (*S*)-α-phenylethylamine gave 37% of β-amino ester **Ib** (see table, run nos. 1, 3). Ethyl 4-phenyl-2-pentenoate (**II**) was formed exclusively as *trans* isomer in 60 and 58% yield, respectively.

It should be emphasized that the chemical yield of β-amino esters in the Rodionov reaction increases in going from aromatic aldehydes to 2-phenylpropanal (from 17–23% [3] to 37–38%); correspondingly, the yield of the unsaturated ester falls down from 80–65% [3] to 58–60%. This may be explained by the fact that the double carbon–carbon bond in the resulting cinnamates is conjugated not only with the carbonyl group but also with the phenyl ring. The double C=C bond in ethyl 4-phenyl-2-pentenoate (**II**) is conjugated only with the carbonyl group, which favors formation of β-amino esters.

By contrast, thermal activation of the reaction of 2-phenylpropanal with benzylammonium acetate and ethyl hydrogen malonate does not lead to formation of β-amino ester **Ia** (see table, run no. 2). In this case, the products are ethyl 4-phenyl-2-pentenoate (**II**, yield 20%, *trans*/*cis*-isomer ratio 10:1) and isomeric ethyl 4-phenyl-3-pentenoate (**III**, yield 54%, *trans*/*cis*-isomer ratio 11:1). In addition, partial transformation of benzylammonium acetate into *N*-benzylacetamide (yield 22%) occurs under these conditions.

When the Rodionov reaction is carried out with free benzylamine, the chemical yield of β-amino ester **Ia** sharply decreases (to 16%), while the yield of unsaturated ester **II** remains almost unchanged (61%). The ratio of the *trans* and *cis* isomers of ester **II** is 23:1. Also, a small amount (3%) of ethyl 4-phenyl-3-pentenoate (**III**) is formed (see table, run no. 4). Just compound **III** is the major product (yield 58%, *trans*/*cis*-isomer ratio 6:1) obtained from the same reaction mixture under conditions of thermal activation (see table, run no. 5). In this case, the yield of β-amino ester **Ia** falls down to 4%, and that of ester **II**, to 6%. Moreover, the condensation is accompanied by ammonolysis of ester **III**, and a considerable part of **III** is converted into amide **IV** with a chemical yield of 25% (*trans*/*cis*-isomer ratio 14:1).

Microwave irradiation of ethyl *trans*-4-phenyl-2-pentenoate (**II**) with 1.5 equiv of benzylamine gives rise to a small amount of the conjugate addition product, β-amino ester **Ia** (<4%, Scheme 2). Here, the main process is migration of the double bond, and ethyl 4-phenyl-3-pentenoate (**III**) becomes the major product (yield 56%, *trans*/*cis*-isomer ratio 16:1; see table, run no. 6). On the other hand, the thermally activated Michael reaction (run no. 7) involves only double bond migration to the β,γ-position and ammonolysis of a larger part of ester **III** thus formed. As a result, ethyl 4-phenyl-3-pentenoate is obtained in a chemical yield of 19% (*trans*/*cis*-isomer ratio 2:1), and the yield of *N*-benzyl-4-phenyl-3-pentenamide (**IV**) is 39% (*trans*/*cis*-isomer ratio 4:1).

Thus the main factors determining the course of the Rodionov reaction are the activation mode and the acidity of the medium. The use of ammonium acetate instead of free amine (Johnson's modification) [7], which was previously proposed as a method for fixing ammonia in the reaction mixture, in our case leads to increased acidity of the medium and hence increased yield of β-amino esters. Presumably, in the reaction with ester **II**, specifically under conditions of thermal activation, free benzylamine acts as a base rather than nucleophile, and (instead of the expected Michael addition) the main process is migration of the double C=C bond in the initial ester from α,β to β,γ position with respect to the carbonyl group according to the mechanism proposed for the first time by Corey [8] (see table, run nos. 4–7). In addition, ammonolysis of ester **III** becomes possible in the thermal reaction (run nos. 5, 7).

Comparison of the NMR spectra of ethyl 4-phenyl-2-pentenoate and ethyl 4-phenyl-3-pentenoate with published data for the same compounds [9] showed that the Rodionov and Michael reactions give esters **II** and **III** predominantly as the corresponding *trans* isomers. In the microwave-activated Rodionov condensation with the use of ammonium acetates, the formation of ethyl *cis*-4-phenyl-2-pentenoate (**II**) is completely suppressed (see table, run nos. 1, 3); the reaction with free benzylamine under MW irradiation gives rise to small amounts of the *cis* isomers of **II** and **III** (run nos. 4, 6); thermal activation of these processes sharply increases the fraction of *cis* isomers among the products (run nos. 2, 5, 7). A plausible explanation is that the intermediate species tends to adopt the most energetically favorable conformation, but the possibility for conformational transition (and hence formation of the *cis* isomer) depends on the reaction conditions.

The formation of β-amino esters **Ia** and **Ib** during the Rodionov condensation involves appearance of a new asymmetric center at the carbon atom in the β-position with respect to the carbonyl group. Therefore, the presence of a chiral center in the molecule

of initial 2-phenylpropanal gives rise to β-amino esters having two (in the reaction with benzylamine) or three (in the reaction with α-phenylethylamine) asymmetric centers. By the reaction with benzylammonium acetate we isolated β-amino ester (**Ia**) as a mixture of two pairs of diastereoisomers, one of these prevailing (de 35%; see table, run no. 1). The observed value of diastereoisomeric excess is comparable with our previous stereochemical results obtained in the Rodionov reaction with aromatic aldehydes (de 27–51%) [3]. These data can also be regarded as one more indirect proof for intermediate formation of Schiff base in this reaction. The Michael synthesis of β-amino esters under the same conditions (MW irradiation of the reactants in the absence of a solvent) is characterized by almost complete absence of stereoselectivity (de 0–7%) [10]. The reaction of ethyl 4-phenyl-2-pentenoate with benzylamine gave β-amino ester **Ia** with a de value of 9% (run no. 6). A relatively high stereoselectivity of the Rodionov reaction may be interpreted as follows. While reacting with each other, intermediate Schiff base and ethyl hydrogen malonate tend to occupy the least sterically hindered positions. The presence of free benzylamine in the reaction mixture makes the above interaction less selective, and the stereoselectivity of the Rodionov reaction decreases more than twofold (from 35 to 15%; see table, run nos. 1, 4). It should be pointed out that the thermally activated Rodionov condensation gives a racemic pair of β-amino ester **Ia** diastereoisomers (de 20%) in which the configuration of asymmetric centers differs from that observed for the usually predominating enantiomeric pair (run no. 5). Due to the presence of an additional chiral center in (*S*)-α-phenylethylamine, β-amino ester **Ib** is formed as four enantiomeric pairs of diastereoisomers at a ratio of 8:5:4:3 (see table, run no. 3).

Up to now, numerous examples have been reported [2, 11] on comparative analysis of reactions whose regio- and stereoselectivity is controlled by MW irradiation and some other method. According to Perreux and Loupy [2], the available data suggest the existence of a specific MW effect which implies mainly reduction of the activation energy; the effect is the stronger, the more polar is the transition state. Our results show (see table, run nos. 1–5) that the thermal Rodionov reaction takes a different pathway than the same reaction activated by MW irradiation; the chemical yields of the products are also different. On the other hand, the mode of activation of the Michael reaction (run nos. 6, 7) is insignificant provided that the ammonolysis process is not taken into account. Depending on the activation mode, different ratios of the *cis* and *trans* isomers are obtained. In addition, the fraction of the *cis* isomers of esters **II** and **III** increases several times under conditions of thermal activation.

We can conclude that the rate-determining stage in the Rodionov reaction is likely to be attack by ethyl hydrogen malonate on the intermediate Schiff base. The energy of activation for Schiff base formation is small (it is formed even under normal conditions), and the energy of activation for the decarboxylation process is also obviously lower than the energy of activation for condensation of ethyl hydrogen malonate with the Schiff base (no products containing a carboxy group were detected in the mixture). Most probably, the reaction of ethyl hydrogen malonate with the Schiff base involves a dipolar or ionic transition state; therefore, a strong MW effect is observed in the Rodionov condensation, and its results differ from those obtained in the thermal reaction. The transition state in the reaction of benzylamine with ethyl 4-phenyl-2-pentenoate (Scheme 2; see table, run nos. 6, 7) is not characterized by such a strong charge separation (the charge is delocalized over the whole ester molecule [8]), and almost no MW effect is observed. However, we cannot assure that MW effect is responsible for suppression of the ammonolysis process.

Thus we have shown that, depending on the mode of activation, the Rodionov condensation can take different pathways leading to formation of different products. The MW-activated process can be used for the synthesis of sterically hindered N-substituted β-amino esters and α,β-unsaturated esters which are difficult or impossible to obtain by other methods. The thermally activated reaction gives β,γ-unsaturated esters and the corresponding amides.

EXPERIMENTAL

The IR spectra were recorded on an IKS-22 spectrometer from samples prepared as thin films. The NMR spectra were obtained on a Varian VXR-400 instrument in chloroform-*d* using TMS as internal reference. The mass spectra were run on an HP-5990 GC–MS system with an HP-5972 mass-selective detector.

General procedure for MW-activated reactions. A glass flask was charged with equimolar amounts of initial reactants (usually, 1 mmol; in the Michael reaction, 1.5 equiv of the amine was taken), and the mixture was irradiated in a Funai MO785VT domestic

microwave oven for 10 min at a power of 175 W (until carbon dioxide no longer evolved). The resulting mixture was subjected to chromatographic separation on a column charged with silica gel L 40/100 μm; a 2:1 benzene–ethyl acetate mixture was used as eluent. β-Amino esters **Ia** and **Ib** are light yellow viscous oily substances. Ethyl 4-phenyl-2-pentenoate (**II**) and ethyl 4-phenyl-3-pentenoate (**III**) are colorless liquids.

General procedure for thermally activated reactions. A mixture of equimolar amounts of the reactants (in the Michael reaction, 1.5 equiv of the amine was taken) was heated for 3 h at 120–140°C, and the products were separated by column chromatography as described above. *N*-benzyl-4-phenyl-3-pentenamide (**IV**) is a colorless crystalline substance.

Ethyl 3-benzylamino-4-phenylpentanoate (Ia). R_f 0.65. IR spectrum, ν, cm^{-1}: 1745 (C=O), 3350 (N–H). ^1H NMR spectrum, δ, ppm (*J*, Hz): major pair of diastereoisomers: 1.25 t (3H, C**H**$_3$CH$_2$, ^3J = 7.15), 1.32 d (3H, C**H**$_3$CH, ^3J = 6.91), 1.55 br.s (1H, NH), 2.42 d (2H, CH$_2$CO, ^3J = 6.91), 3.01 q (1H, C**H**CH$_3$, ^3J = 6.91), 3.22 q (1H, C**H**CH$_2$, ^3J = 6.91), 3.72 d (1H, C**H**$_2$Ph, ^2J = 13.00), 3.79 d (1H, C**H**$_2$Ph, ^2J = 13.00), 4.12 q (2H, C**H**$_2$CH$_3$, ^3J = 7.15), 7.10–7.36 m (10H, Ph). Mass spectrum, *m*/*z* (I_{rel}, %): 311 (<1%) M^+, 224 (24%), 206 (98%), 105 (10%), 91 (100%), 77 (7%); minor pair of diastereoisomers: 1.24 t (3H, C**H**$_3$CH$_2$, ^3J = 7.13), 1.38 d (3H, C**H**$_3$CH, ^3J = 7.12), 2.23 d.d (1H, CH$_2$CO, ^3J = 6.60), ^2J = 15.11), 3.21 q (2H, C**H**CH$_3$, ^3J = 6.60), 4.04 q (2H, C**H**$_2$CH$_3$, ^3J = 7.15) (the other signals are overlapped by those of the major diastereoisomers). Mass spectrum, *m*/*z* (I_{rel}, %): 311 (<1%) M^+, 224 (4%), 206 (36%), 105 (8%), 91 (100%), 77 (7%).

Ethyl 3-[(*S*)-1-phenylethylamino]-4-phenylpentanoate (Ib). R_f 0.5. IR spectrum, ν, cm^{-1}: 1735 (C=O); 3330 (N–H). ^{13}C NMR spectrum, δ_C, ppm (the signals are given for diastereoisomers in the order of decreasing fraction): isomer 1: 14.18, 16.97, 24.59, 36.04, 41.04, 55.25, 57.01, 60.14, 144.57, 145.90, 172.73; isomer 2: 14.16, 17.22, 24.85, 36.15, 43.16, 55.14, 57.36, 60.60, 144.22, 145.93, 172.47; isomer 3: 14.06, 17.37, 24.45, 37.43, 40.26, 55.02, 57.54, 60.21, 143.56, 145.30, 172.54; isomer 4: 14.06, 17.37, 24.31, 36.42, 43.42, 55.27, 57.54, 60.21, 142.99, 145.08, 172.48; for all isomers, signals from the aromatic carbon atoms appear in the region δ_C 126.0–128.5 ppm.

Ethyl 4-phenyl-2-pentenoate (II). R_f 0.95. IR spectrum, ν, cm^{-1}: 1690 (C=O), 1655 (C=C). ^1H NMR spectrum, δ, ppm (*J*, Hz): *E* isomer: 1.23 t (3H, C**H**$_3$CH$_2$, ^3J = 7.13), 1.38 d (3H, C**H**$_3$CH, ^3J = 6.93), 3.57 d.q (1H, C**H**CH$_3$, ^3J = 6.93, ^4J = 1.49), 4.13 q (2H, C**H**$_2$CH$_3$, ^3J = 7.13), 5.79 d.d (1H, CH=C**H**CO, ^3J = 15.75, ^4J = 1.49), 7.10 d.d (1H, CHC**H**=CH, ^3J = 6.93, ^3J = 15.75), 7.15–7.35 m (5H, Ph). *Z* isomer: 1.26 t (3H, C**H**$_3$CH$_2$, ^3J = 7.13), 1.44 d (3H, C**H**$_3$CH, ^3J = 9.81), 3.08 d.q (1H, C**H**CH$_3$, ^3J = 9.81, ^4J = 1.09), 4.15 q (2H, C**H**$_2$CH$_3$, ^3J = 7.13), 5.71 d.d (1H, CH=C**H**CO, ^3J = 11.30, ^4J = 1.09), 6.23 d.d (1H, CHC**H**=CH, ^3J = 9.81, ^3J = 11.30), 7.15–7.35 m (5H, Ph).

Ethyl 4-phenyl-3-pentenoate (III). R_f 0.95. IR spectrum, ν, cm^{-1}: 1740 (C=O). ^1H NMR spectrum, δ, ppm (*J*, Hz): *E* isomer: 1.27 t (3H, C**H**$_3$CH$_2$, ^3J = 7.13), 2.05 d.t (3H, C**H**$_3$CPh=CH, ^4J = 1.25, ^5J = 1.07), 3.24 d.q (2H, CHC**H**$_2$CO, ^3J = 7.15, ^5J = 1.07), 4.16 q (2H, C**H**$_2$CH$_3$, ^3J = 7.13), 5.95 t.q (1H, CH$_3$CPh=C**H**CH$_2$, ^3J 7.15, ^4J = 1.25), 7.15–7.35 m (5H, Ph); *Z* isomer: 1.26 t (3H, C**H**$_3$CH$_2$, ^3J = 7.13), 2.08 d.t (3H, C**H**$_3$CPh=CH, ^4J = 1.46, ^5J = 1.38), 3.00 d.q (2H, CHC**H**$_2$CO, ^3J = 7.38, ^5J = 1.38), 4.11 q (2H, C**H**$_2$CH$_3$, ^3J = 7.13), 5.65 t.q (1H, CH$_3$CPh=C**H**CH$_2$, ^3J = 7.38, ^4J = 1.46), 7.15–7.35 m (5H, Ph).

***N*-Benzyl-4-phenyl-3-pentenamide (IV).** mp 75°C. R_f 0.6. IR spectrum, ν, cm^{-1}: 1645 (C=O), 1610 (C=C), 3290 (N–H). ^1H NMR spectrum, δ, ppm (*J*, Hz): *E* isomer: 1.96 d.t (3H, C**H**$_3$CPh=CH, ^4J = 1.12, ^5J = 0.97), 3.11 d.q (2H, CHC**H**$_2$CO, ^3J = 7.39, ^5J = 0.97), 4.33 d (2H, NHC**H**$_2$Ph, ^3J = 5.94), 5.89 t.q (1H, CH$_3$CPh=C**H**CH$_2$, ^3J = 7.39, ^4J = 1.12), 6.77 br.s (1H, NH), 7.08–7.35 m (10H, Ph); *Z* isomer: 2.01 d.t (3H, C**H**$_3$CPh=CH, ^4J = 1.51, ^5J = 1.00), 2.88 d.q (2H, CHC**H**$_2$CO, ^3J = 7.47, ^5J = 1.00), 4.29 d (2H, NHC**H**$_2$Ph, ^3J = 5.99), 5.60 t.q (1H, CH$_3$CPh=C**H**CH$_2$, ^3J = 7.47, ^4J = 1.51), 6.40 br.s (1H, NH), 7.08–7.35 m (10H, Ph).

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