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On the 100th anniversary of V.V. Perekalin

Synthesis and Structure of β-Aryl-α-nitroacrylates

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Abstract—The method for preparation of ethyl α-nitrocinnamates by nitroacetic acid ester alkenylation with aromatic aldehydes in the presence of acetic acid and β-alanine has been modified. Structures of the prepared compounds have been proved by electronic, IR, ^{1}H, and ^{13}C–{^{1}H} NMR spectroscopy (including heteronuclear correlation experiments ^{1}H–^{13}C HMQC and ^{1}H–^{13}C HMBC). In solution these compounds exist in the form of *Z*-isomer; the $Z \rightleftarrows E$ isomerization is observed in the case of the compound containing strong electron-donor group [$N(CH_3)_2$] at benzene ring.

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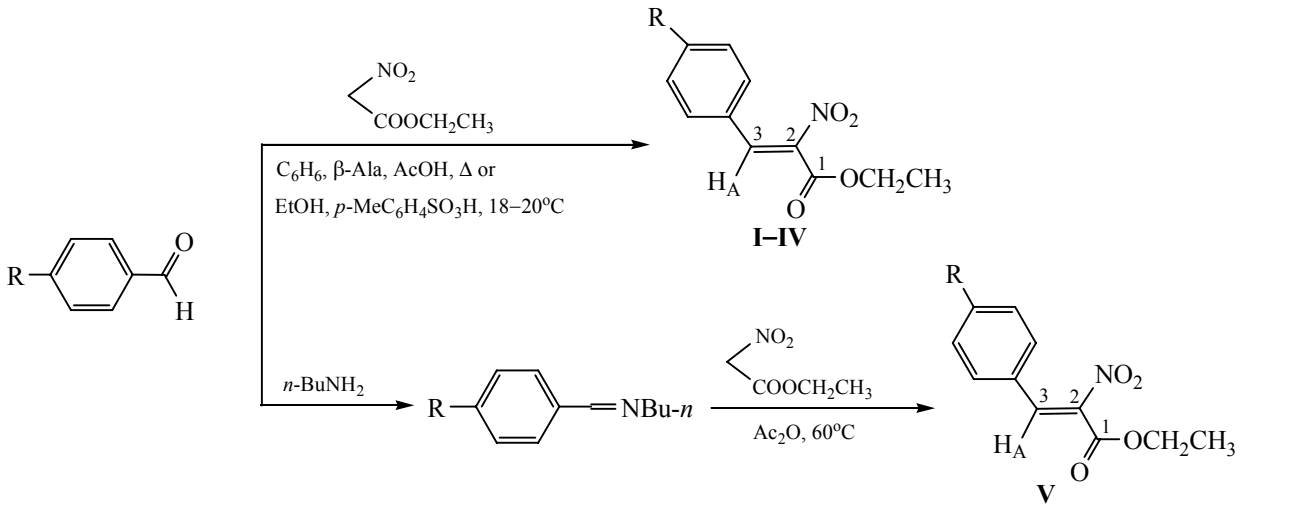
Conjugated nitroethenes are highly reactive synthons suitable for preparation of organic compounds of different classes [1, 2].

Introduction of ester group at the *gem*-position with respect to the nitro group expands the range of nitroethenes synthetic applications due to the increased electrophilicity of the double bond and possibilities of additional transformations involving the electron-withdrawing ester group. The α-nitroalkenecarboxylates can be converted into α-amino acids through hydrogenation and subsequent hydrolysis. These polyfunctional electron-deficient compounds are of particular interest due to the possibility of their conversion into carbocyclic and heterocyclic systems, the promising intermediates in drug design.

Unlike simple β-nitrostyrenes, most of α-nitrocinnamic acid esters cannot be obtained via condensation of aromatic aldehydes with nitroacetic acid ester under basic catalysis conditions. In the presence of basic agents, the condensation results in the substituted 2,4-dinitroglutarates salts or products of their heterocyclization – five-membered isoxazoline *N*-oxides and isoxazoles [3–8]. Such reaction pathway can be probably due to the interaction of nitroacetic acid ester with the initially formed highly reactive *gem*-substituted nitroethenes. Sensationally been recently shown that reaction of aromatic aldehydes with nitroacetic acid ester in alcohol in the presence of diethylamine results in six-membered substituted 5-hydroxy-1,2-oxazin-6-one-3-carboxylates, the structure of their isolated salts having been confirmed by X-ray diffraction analysis [9]. The *gem*-alkoxycarbonylnitroethenes can be prepared from nitroacetic acid ester by using the Schiff's bases or diacetals instead of aldehydes [3, 10–12]. The direct alkenylation of ethyl nitroacetate with heterocyclic aromatic aldehydes has been achieved for the first time in a mixture of anhydrous THF and CCl_4 in the presence of an excess of titanium tetrachloride and *N*-methylmorpholine [13]. However, this method is quite expensive, and time-taking.

We succeeded in preparation of α-nitrocinnamic esters **I**–**IV** via condensation of ethyl nitroacetate with aromatic aldehydes under acid catalysis conditions: refluxing the reagents in benzene in the presence of acetic acid and β-alanine in a flask equipped with a Dean–Stark trap. Similar techniques were applied previously for the synthesis of *gem*-benzoylnitroethenes [14, 15] and α-nitro-β-furyl(thienyl)acrylates [16]. However, synthesis of the title compounds under such conditions had some limitations. In particular, the preparation of the α-nitrocynnamic acid ester derivative **V**, bearing a nitro group in the *para*-position of the benzene ring, was a success only when performed according to the reported method [3] (reaction of nitroacetic acid ester with the Schiff's base synthesized from 4-nitrobenzaldehyde and *n*-butylamine).



R = H (**I**), OMe (**II**), NMe_2 (**III**), Cl (**IV**), NO_2 (**V**).

α-Nitrocinnamic acid ester **III** containing *p*-dimethylamino substituent at the benzene ring could be obtained as well from 4-dimethylaminobenzaldehyde and nitroacetic ester in ethanol in the presence of *p*-toluenesulfonic acid at room temperature [17]. However, we failed to synthesize compounds **I**, **II**, and **IV** under those conditions. For example, reaction of 4-methoxybenzaldehyde with nitroacetic acid ester in the presence of *p*-toluenesulfonic acid gave 2-(4-methoxyphenyl)-1-nitroethene (the product could be isolated after the prolonged exposure). Probably, in those cases the desired process was accompanied by hydrolysis of the ester group followed by decarboxylation.

The melting points and spectral properties of the prepared β-aryl-α-nitroacrylates were identical to those reported in [3, 13, 18–23].

The structures of **I**–**V** were elucidated via comprehensive analysis of their spectral features (^{1}H and ^{13}C–{^{1}H} NMR, UV, and IR) and their comparison with the respective data for model nitroethenes [24–27]. The main parameter to judge about the functionalized nitroethenes geometry was the chemical shift of olefinic proton H_A in ^{1}H NMR spectra. In contrast to the *E*-isomers of model nitroethenes (H_A signal at 7.87–8.03 ppm, $^3J(H_AH_B)$ 13.5–14 Hz), the *gem*-alkoxycarbonylnitroethenes existed in the form of *Z*-isomer in $CDCl_3$ solutions; this was confirmed by upfield position of the H_A signal (7.39–7.60 ppm) (Table 1).

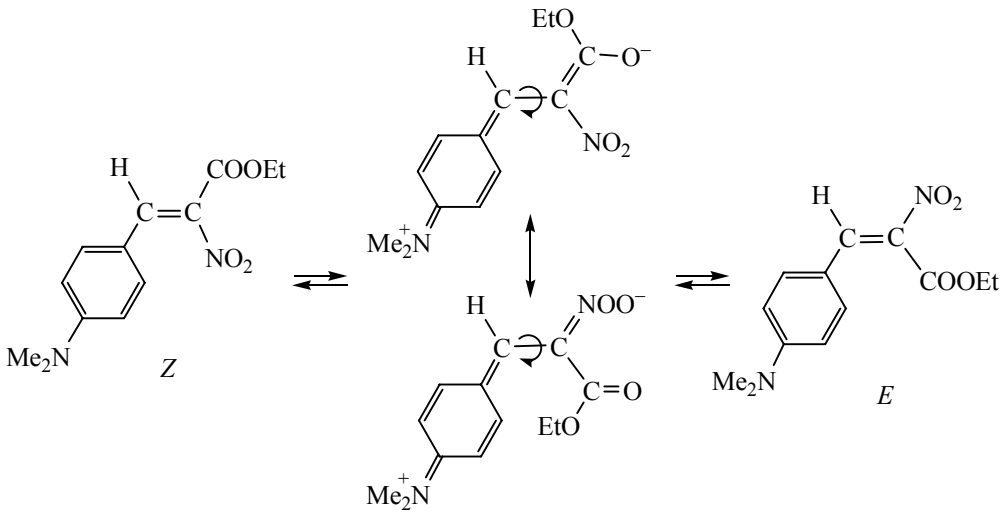
In the case of nitrocinnamic ester **III** (bearing the dimethylamino group, an electron-donating substituent with strong +*M* effect), the barrier of internal rotation around the carbon–carbon bond was reduced due to a significant contribution of bipolar structures to the ground state; thus, acceleration of the *Z*–*E* transition could be expected as compared to the unsubstituted analog **I**.

Indeed, in the ^{1}H NMR spectrum of **III** in $CDCl_3$ solution, after 2–3 days exposure the proton signals were split into pairs thus indicating the presence of two isomers in the solution (Table 1, Fig. 1b). The spectrum contained the signals of olefinic protons at 7.38 ppm (*Z*-) and 8.01 ppm (*E*-); the ratio of *Z*- and *E*-isomers was of ~1:1.1. In more polar solvents (methanol-d_4 and DMSO-d_6), compound **III** formed a mixture of *Z*- and *E*-isomers (in the ratio of ~1:1) immediately after dissolution.

The similar readily occurring isomers transformations were observed in the cases of indolylnitroacrylates and nitroenamines [28–30].

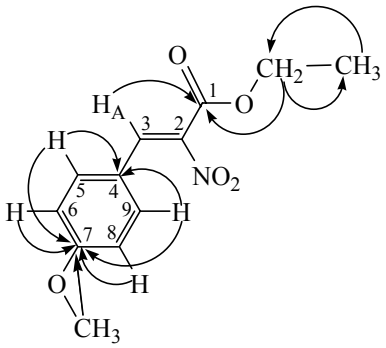
α-Nitroacrylate **V** containing strong electron withdrawing nitro group in benzene ring existed in the form of *Z*-isomer in methanol-d_4 solution. Upon exposure at 18–20°C, it reacted with methanol-d_4, and within 7 min the Ad_N product was detected in the spectra.

In the ^{13}C–{^{1}H} NMR spectra of α-nitroacrylates **I**–**V**, all respective carbon signals were identified (Table 2). The signals of ester moiety methyl and methylene carbon atoms appeared at 13.97–14.28 ppm and 62.77–63.81 ppm, respectively. The signals of C^2 with two electron-withdrawing substituents (nitro and ester groups) attached were registered in a downfield region (135.53–142.69 ppm) as compared with those of C^3 (119.61–138.11 ppm). The signals at 158.48–162.96 ppm were assigned to the carbonyl carbon atom.



The ^1H and $^{13}C–\{^1H\}$ NMR spectra signals assignment was confirmed by heteronuclear correlation $^1H–^{13}C$ HMQC and $^1H–^{13}C$ HMBC experiments. In particular, the $^1H–^{13}C$ HMQC spectrum ($CDCl_3$) of **II** contained cross-peaks of ester protons (1.32 and 4.33 ppm) with carbon atoms (14.16 and 62.89 ppm) signals; the cross-peak of methoxy group protons (3.81 ppm) with carbon atom (55.59 ppm) was observed as well. The signal of H_A olefinic proton (7.43 ppm) correlated with the signal of C^3 (132.68 ppm). Finally, the cross-peaks characteristic of the *p*-substituted benzene ring were observed: protons signals at 6.88 ppm (C^5H, C^9H) and 7.36 ppm (C^6H, C^8H) correlated with the signals of aromatic carbon atoms at 114.99 ppm ($C^{5,9}$) and 132.27 ppm ($C^{6,8}$), respectively.

In the $^1H–^{13}C$ HMBC spectrum of **II** ($CDCl_3$), methyl protons of the ester group (1.32 ppm) correlated with the carbon atoms of methylene moiety (62.89 ppm), and methylene protons of the ester group (4.33 ppm) correlated with the carbon atoms of the methyl (14.16 ppm) and carbonyl (159.68 ppm) groups. The latter signal correlated with the signal of olefinic proton H_A (7.43 ppm) as well. The proton signal at 6.88 ppm (C^5H, C^9H) revealed cross-peaks with the signals of carbon atoms at 121.36 ppm (C^4) and 162.96 ppm (C^7); the latter signal being correlated with the protons signals at 7.36 (C^6H, C^8H) and 3.81 ppm (OCH_3) as well.



Z-Configuration of the discussed *gem*-substituted nitroethenes determined from ^1H NMR spectra was additionally confirmed by electronic and IR spectra. For example, in the electronic spectrum of the model

Table 1. ^1H NMR spectral data for α-nitrocinnamic acids esters **I–V** in $CDCl_3$ (δ, ppm)

Comp. no.	H_A	CH_3	OCH_2	CH_3O [$(CH_3)_2N$]	Ar	Configuration
I	7.53 s	1.35 t	4.37 q	–	7.30–7.50 m	*Z*
II	7.43 s	1.32 t	4.33 q	3.81 s	6.88 d, 7.36 d	*Z*
III	7.39 s	1.33 t	4.32 q	[3.05 s]	6.62 d, 7.30 d	*Z*
III[a]	7.39 s	1.33 t	4.32 q	[3.05 s]	6.62 d, 7.30 d	*Z*
	8.00 s	1.38 t	4.45 q	[3.08 s]	6.65 d, 7.38 d	*E*
IV	7.47 s	1.35 t	4.36 q	–	7.33 d, 7.38 d	*Z*
V	7.60 s	1.38 t	4.41 q	–	7.58 d, 8.27 d	*Z*

[a] The spectrum was recorded in $CDCl_3$ after 2 days exposure.

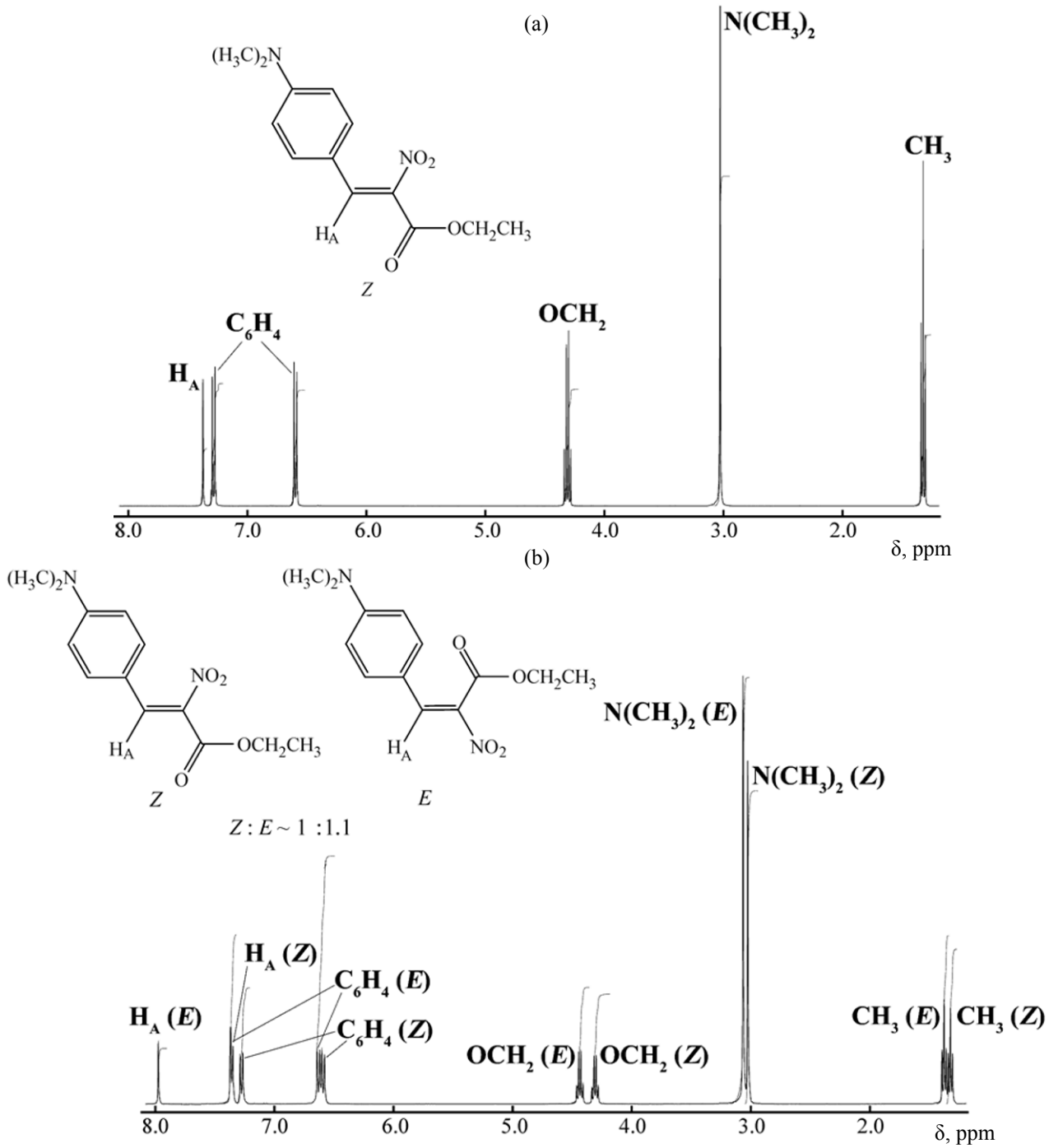


Fig. 1. ^{1}H NMR spectra of compound **III** in $CDCl_3$ immediately (a) after the dissolution and (b) after 2 days exposure.

E-2-(4-methoxyphenyl)-1-nitroethene an absorption band was observed at 355 nm (ε = 21000 l mol^{-1} cm^{-1}), characteristic of nitrostyrenes with an electron-donor substituent at the opposite end of the conjugated system with respect to the nitro group [24]. Introduction of the ester group led to the blue shift of the absorption band (λ_{max} = 319 nm, ε = 25500 l mol^{-1} cm^{-1}); that confirmed the existence of the *Z*-configuration of **II** (Table 2).

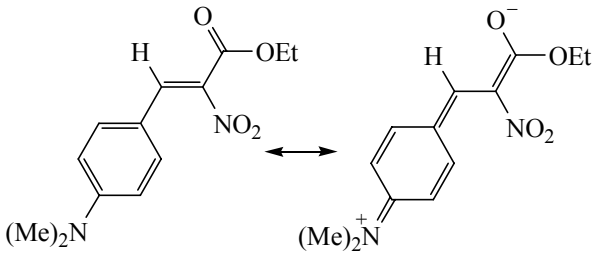
The electronic spectrum of **III** recorded in ethanol contained two absorption bands at λ_{max} = 400 nm (ε = 9400 l mol^{-1} cm^{-1}) and λ_{max} = 450 nm (ε = 10100 l mol^{-1} cm^{-1}) (Table 3). Thus, that ester of α-

nitrocinnamic acid with dimethylamino group the *para*-position underwent $Z \rightleftarrows E$ isomerization.

Further information on the studied compounds structure was obtained from their IR spectra (Table 3). The IR spectra of ethyl α-nitrocinnamate **I** and its analogs **II**–**V** contained the absorption bands of carbonyl (1715–1730 cm^{-1}) and covalently bonded nitro group (ν_{as} = 1535–1545 cm^{-1} and ν_s = 1370–1375 cm^{-1}).

It should be noted that the IR spectra of the α-nitro cinnamic esters containing *para*-positioned electron-donor methoxy or dimethylamino group (**II** and **III**) differed significantly from those of the corresponding model *E*-2-(4-methoxyphenyl)-1-nitroethene and *E*-2-(4-*N*,*N*-dimethylaminophenyl)-1-nitroethene: in the latter spectra there were no characteristic vibrations of the covalently bonded nitro group, whereas the intense bands appeared at 1600 and 1625 cm^{-1} (C=C, $C=N^+$) as well as at 1320 and 1260–1265 cm^{-1} (NOO^-). That indicated high polarization of their molecules due to effective conjugation of nitrogen or oxygen lone-electron pair, carbon–carbon double bond, and nitro group [25]. *Z*-2-(4-Methoxyphenyl)- and *Z*-2-(4-*N*,*N*-dimethylaminophenyl)-1-nitro-1-ethoxycarbonylethenes **II** and **III** were highly polarized as well, but it was the effective conjugation with the carbonyl group that contributed significantly to the polarization of the molecules.

Similar conclusions were made in the studies of β-acetyl-β-nitrostyrenes containing electron-donor substituents and their furyl and thienyl analogs, existing predominantly in the *Z*-form [14, 26].



In [25], the existence of **I** in the *Z*-form was supported by the integral intensities of the valence vibrations absorption bands of $A_s(NO_2)$ (1.3) and A(C=C) (0.74) and comparison with the respective parameters of other *gem*-substituted β-nitrostyrenes.

The high polarization of *Z*-α-nitrocinnamic esters was confirmed by the dipole moments measurement using methyl 2-nitro-3-(4-nitrophenyl)acrylate as an example. Comparison of its experimental dipole moment (5.5 D) with that calculated by the vector-additive scheme for the *s-cis* and *s-trans* conformers indicated the predominance of *s-trans* conformer [31].

The X-ray diffraction data for methyl α-nitro-cinnamate acid and its *p*-methoxy analogue as well as for ethyl α-nitroacrylates containing *p*-nitrophenyl or ferrocenyl substituents listed in [32–36] demonstrated that both in the solid phase and in solution those compounds were of *Z*-configuration, and the nitro group was deduced out of the molecule plane. For methyl α-nitroacrylates the *s-trans* conformation was

Table 2. $^{13}C-\{^1H\}$ NMR spectral data for α-nitrocinnamic acids esters **I**–**V** in $CDCl_3$ (δ, ppm)

Comp. no.	C^3	C^2	C=O	CH_2	CH_3	OCH_3 [$N(CH_3)_2$]	Ar	Confuguration
I	133.03	140.25	159.31	63.23	14.16	–	128.98, 129.49, 129.84, 132.30	*Z*
II	132.68	138.19	159.68	62.89	14.16	55.59	114.99, 121.36, 132.27, 162.96	*Z*
III[a]	132.77	135.53	160.41	62.39	14.28	[40.05]	111.87, 115.90, 132.71, 152.95	*Z*
	138.11	136.76	162.93	62.77	13.99	[40.12]	111.97, 115.95, 132.71, 153.35	*E*
IV	131.61	140.52	159.08	63.35	14.13	–	127.43, 129.85, 131.03, 138.58	*Z*
V	130.26	142.69	158.48	63.1	14.10	–	124.52, 130.33, 135.06, 149.30	*Z*

[a] The spectrum was registered in $CDCl_3$ after 2 days exposure.

typical, whereas ethyl α-nitroacrylates were predominantly *s-cis* conformers.

To summarize, we proposed a suitable synthetic method to prepare ethyl α-nitrocinnamates via condensation of aromatic aldehydes with the esters of nitroacetic acids under reflux in benzene in the presence of acetic acid and β-alanine. The procedure significantly facilitated the synthesis of those important compounds. Their structures were confirmed by ^{1}H, ^{13}C NMR, IR, and electronic spectroscopy. It was shown that in solutions α-nitrocinnamic acids esters were *Z*-configured. α-Nitrocinnamate containing *p*-dimethylaminophenyl moiety was the most inclined to $Z \rightleftarrows E$ isomerization.

EXPERIMENTAL

^{1}H, ^{13}C–{^{1}H}NMR, ^{1}H–^{13}C HMQC, and ^{1}H–^{13}C HMBC spectra were recorded with Jeol JNM-ECX400A spectrometer operating at 399.78 (^{1}H) and 100.53 MHz (^{13}C) in chloroform-*d*, DMSO-d_6, or methanol-d_4 solution; the signals of the residual non-deuterated solvents were used as internal standard. IR spectra were recorded with Shimadzu IR-Prestige-21 Fourier spectrometer in chloroform (c = 40 mg ml^{-1}). Electronic absorption spectra were recorded with Shimadzu UV2401PC spectrophotometer in ethanol solution in quartz cuvettes (l = 1.01 mm) at 0.5–0.8 mM.

Ethyl nitroacetate [37], 4-nitrobenzal-*n*-butylamine [31], and ethyl 2-nitro-3-(4-nitrophenyl)propenoate **V** [3] were prepared as described in the respective references.

Ethyl 2-nitro-3-phenylpropenoate (I). A mixture of 3.3 g (25 mmol) of ethyl nitroacetate, 4.24 g (40 mmol) of freshly distilled benzaldehyde, catalytic amount of β-alanine, and 4 ml of glacial acetic acid in 30 ml of anhydrous benzene was refluxed for 5 h in a flask equipped with a Dean–Stark trap. After cooling, the reaction mixture was washed with saturated aqueous sodium chloride and dried over calcinated $MgSO_4$. Benzene was evaporated on a rotary evaporator, and the residue was placed into refrigerator. After treating with ethanol, the crystalline substance was filtered off and dried. Yield 3.43 g (62%), pale yellow crystals, mp 63–65°C (hexane) {mp 68–69°C (methanol) [18]}.

Ethyl 3-(4-methoxyphenyl)-2-nitropropenoate (II). A mixture of 3.3 g (25 mmol) of ethyl nitroacetate, 5.39 g (40 mmol) of freshly distilled anisaldehyde, catalytic amount of β-alanine, and 4 ml of glacial acetic acid in 30 ml of anhydrous benzene was refluxed for 5 h in a flask equipped with a Dean–Stark trap. The reaction solution was then treated as described in the procedure of **I** preparation. After long-time exposure, oily substance with inclusions of crystalline solid was placed on a porous plate to obtain 2.28 g (36%) of pale yellow crystals, mp 62–64°C (hexane) {mp 66–67°C (hexane–petroleum ether) [13]}.

Table 3. Data of IR and electronic spectra of α-nitrocinnamic acids esters **I–V**

Comp. no.	Electronic spectra (EtOH)[a]		IR spectra ($CHCl_3$), ν, cm^{-1}		
	λ_{max}, nm	ε, l mol^{-1} cm^{-1}	NO_2	C=O	C=C
	280	17800	1540, 1370	1730	1645
II	319	25500	1540, 1370	1725	1640, 1600
III	400 450	9400 10100	1535, 1375	1715	1595
IV	288	17150	1540, 1370	1730	1650, 1595
V	287	15460	1545, 1370, 1530, 1350[b]	1735	1655

[a] Absorption bands at long-wave region (λ > 270 nm) are listed.
[b] Absorption bands of the stretching vibrations of nitro group in the aromatic ring.

Ethyl 3-(4-*N*,*N*-dimethylaminophenyl)-2-nitropropenoate (III). A mixture of 3.3 g (25 mmol) of ethyl nitroacetate, 5.96 g (40 mmol) of 4-*N*,*N*-dimethylaminobenzaldehyde, catalytic amount of β-alanine, and 4 ml of glacial acetic acid in 30 ml of anhydrous benzene was refluxed for 2 h in a flask equipped with a Dean–Stark trap. The reaction solution was then treated as described in the procedure of **I** preparation. Yield 6.3 g (95%), orange crystals, mp 88–90°C (ethanol) {mp 92–93°C (ethanol) [13]}.

Ethyl 3-(4-chlorophenyl)-2-nitropropenoate (IV). A mixture of 1.44 g (11 mmol) of ethyl nitroacetate, 1.9 g (14 mmol) of *p*-chlorobenzaldehyde, catalytic amount of β-alanine, and 2 ml of glacial acetic acid in 30 ml of anhydrous benzene was refluxed for 5 h in a flask equipped with a Dean–Stark trap. The reaction solution was then treated as described in the procedure of **I** preparation. Yield 1.09 g (39%), pale yellow crystals, mp 60–62°C (ethanol) {mp 69°C (benzene–petroleum ether) [3]}.

Physico-chemical studies were performed in the Center for Collective Use in Herzen State Pedagogical University of Russia.

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