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Some Chemical Transformations of Alkyl (4-Aminophenyl)carbamates

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Abstract—Azo coupling of diazonium salts derived from alkyl (4-aminophenyl)carbamates with ethyl α-methylacetoacetate gave ethyl 5-alkoxycarbonylamino-1*H*-indole-2-carboxylates. The condensation of aminophenylcarbamates with aromatic aldehydes in ethanol afforded the corresponding Schiff bases. Cyclohexyl {4-[(4-methoxyphenyl)methylidene]aminophenyl}carbamate reacted with chloroacetyl chloride in dioxane in the presence of triethylamine to produce cyclohexyl {4-[3-chloro-2-(4-methoxyphenyl)-4-oxoazetidin-1-yl]phenylcarbamate, and the reaction of benzyl {4-[(4-nitrophenyl)methylidene]aminophenyl}carbamate with sulfanylacetic acid in DMF led to the formation of benzyl {4-[2-(4-nitrophenyl)-4-oxo-1,3-thiazolidin-3-yl]-phenyl}carbamate.

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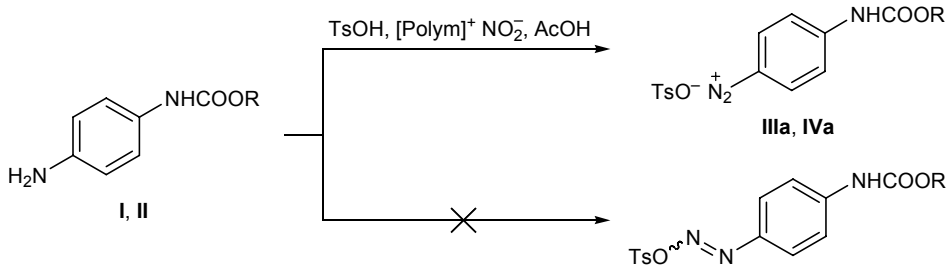
Aromatic and heteroaromatic amines attract much interest as important intermediate products in the synthesis of various aromatic and heterocyclic compounds [1–4]. Amino derivatives of alkyl phenylcarbamates were synthesized previously by reduction of the corresponding alkyl (4-nitrosophenyl)carbamates with sodium dithionite [5]. It was also shown [5] that the Japp–Klingemann–Fischer reaction with benzyl and cyclohexyl (4-aminophenyl)carbamates **I** and **II** yields carbamate derivatives of indoles having no substituents in positions *1* and *3*. The azo coupling with ethyl α-methylacetoacetate was carried out with arenediazonium chlorides and *p*-toluenesulfonates obtained by diazotization of aminophenylcarbamates **I** and **II** with nitrous acid and polymeric diazotizing agent $[Polym]^+ NO_2^-$. In the latter case, the diazotization was performed by adding polymeric diazotizing agent (prepared by saturation of AV-17-8 anion exchanger with nitrite ions [6]) and alkyl (4-aminophenyl)carbamate **I** or **II** to a solution of *p*-toluenesulfonic acid in glacial acetic acid.

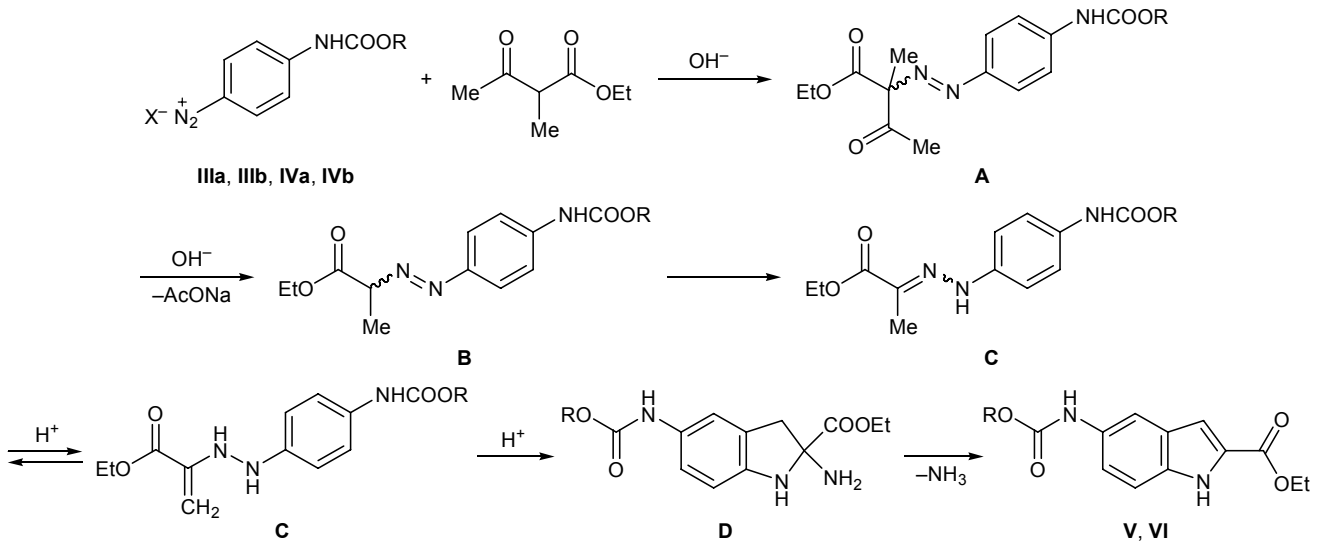
Unlike diazotization with nitrous acid, the reaction with $[Polym]^+ NO_2^-$ was complete in 30 min at room temperature, and the diazotization products were isolated as individual substances by precipitation with diethyl ether after separation of the anion exchanger by filtration. As shown in [7], diazotization with $[Polym]^+ NO_2^-$ of aromatic amines having electron-withdrawing substituents in the benzene ring gives the corresponding arenediazonium *p*-toluenesulfonates, whereas aromatic amines with electron-donating groups are converted into (*Z*)-1-aryl-2-tosyldiazenes

Scheme 1.



I, **IIIa**, R = $PhCH_2$; **II**, **IVa**, R = *cyclo*-C_6H_{11}.

Scheme 2.



X = TsO (**a**), Cl (**b**).

which can also be involved in azo coupling. We have found that, despite electron-donor properties of alkoxycarbonylamino group, the diazotization of **I** and **II** with $[Polym]^+ NO_2^-$ yields arenediazonium *p*-toluenesulfonates **IIIa** and **IVa** (Scheme 1).

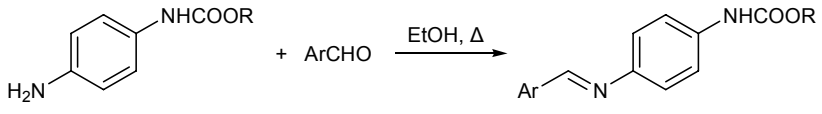
The formation of diazonium salts **IIIa** and **IVa** follows from their IR, ^{13}C NMR, and mass spectra. Apart from other bands, the IR spectra of **IIIa** and **IVa** contained a medium-intensity absorption band at 2276–2280 cm^{-1} due to stretching vibrations of the $N{\equiv}N^+$ group. In the ^{13}C NMR spectra of **IIIa** and **IVa** the signal of the carbon atom bearing the diazonium group was observed at δ_C 112.42 and 111.12 ppm, respectively, due to magnetic anisotropy of the N≡N group. If these compounds contained an N=N bond, the signal from the neighboring aromatic carbon atom would appear in a weaker field (δ_C 120–131 ppm) [7]. The mass spectra of **IIIa** and **IVa** revealed no molecular ion peaks because of low volatility of arenediazonium *p*-toluenesulfonates, but ion peaks corresponding to thermal decomposition products formed as a result of loss of nitrogen molecule were present [8, 9].

The observed pattern in the diazotization of aromatic amines having a carbamate group is likely to be determined by the ability of the latter to undergo acid-catalyzed tautomeric transformation into imidoyl structure which acts as a weak electron acceptor.

Arenediazonium *p*-toluenesulfonates **IIIa** and **IVa** and the corresponding chlorides **IIIb** and **IVb** were brought into azo coupling with ethyl α-methylacetoacetate in alkaline medium (Scheme 2). Initially formed azo coupling product **A** underwent acid decomposition by the action of concentrated alkali to give azo compound **B**. Unlike common acid decomposition of β-keto esters, no hydrolysis of the ester group occurred; moreover, the carbamate moiety also remained unchanged. Passing of dry hydrogen chloride through a solution of azo compound **B** in anhydrous ethanol induced isomerization to enamine **C** which underwent acid-catalyzed cyclization to indole **V** or **VI** via elimination of ammonia molecule from intermediate **D**. The structure of **V** and **VI** was confirmed by their IR and ^1H NMR spectra.

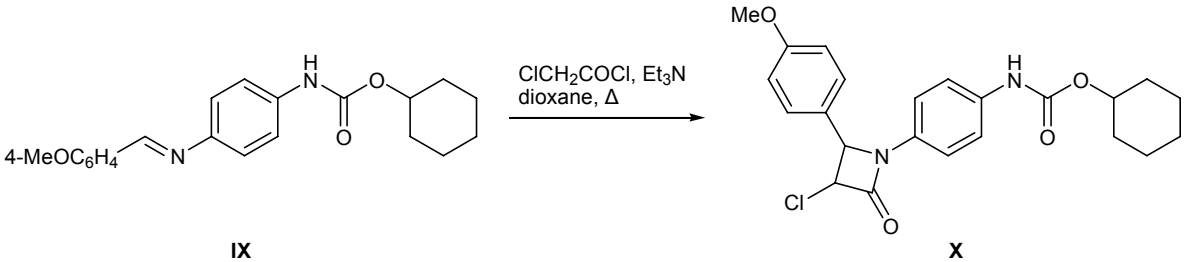
Primary aromatic amines are widely used to prepare Schiff bases exhibiting a great pharmacological potential. Compounds possessing anticonvulsant [10], cardiotonic [11], antiproliferative [12], antifungal [13], antitumor [14], and antimicrobial activity [15] were

Scheme 3.



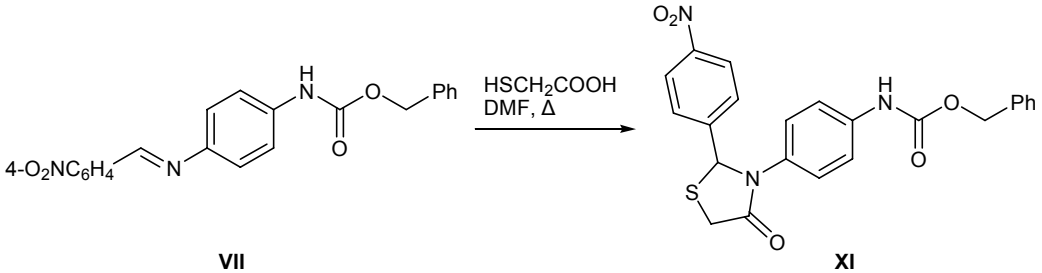
VII, **VIII**, R = Ph CH_2; **IX**, R = *cyclo*-C_6H_{11}; **VII**, Ar = 4-$O_2NC_6H_4$; **VIII**, Ar = 2,4-$(HO)_2C_6H_3$; **IX**, Ar = 4-$MeOC_6H_4$.

Scheme 4.



IX **X**

Scheme 5.



VII **XI**

found among Schiff bases. They are also used as intermediate products in the synthesis of various biologically active compounds, in particular thiazolidinone, azetidinone, formazan, arylacetamide, and many other derivatives [16–18].

With a view to obtain potentially biologically active Schiff bases having a carbamate functionality, alkyl (4-aminophenyl)carbamates **I** and **II** were brought into condensation with aromatic aldehydes in ethanol in the presence of a catalytic amount of glacial acetic acid (Scheme 3). As expected, the products were the corresponding Schiff bases **VII**–**IX**. Unlike the initial compounds, the IR spectra of **VII**–**IX** contained an absorption band at 1645–1650 cm^{-1} due to stretching vibrations of the C=N bond. Apart from other signals, Schiff base **VII** displayed in the ^1H NMR spectrum a one-proton singlet at δ 8.82 ppm from the azomethine proton.

The reaction of Schiff base **IX** with chloroacetyl chloride in the presence of triethylamine in boiling dioxane (12 h) afforded cyclohexyl {4-[3-chloro-2-(4-methoxyphenyl)-4-oxoazetidin-1-yl]phenyl}carbamate (**X**) (Scheme 4) whose structure was determined by IR and ^1H NMR spectroscopy. In the ^1H NMR spectrum of **X** we observed singlets at δ 5.70 ppm from 2-H and at δ 4.89 ppm from 3-H in the azetidine ring, which is consistent with published data for structurally related azetidine derivatives [1, 2].

Compound **VII** reacted with sulfanylacetic acid on heating in boiling anhydrous dimethylformamide over a period of 12 h to produce thiazolidinone derivative **XI** (Scheme 5). Unlike initial Schiff base **VII**, the IR spectrum of **XI** lacked absorption band at 1645 cm^{-1} assignable to C=N stretching vibrations, but a band at 687 cm^{-1} appeared due to stretching vibrations of the C–S–C fragment; in addition, a carbonyl absorption band was present at 1780 cm^{-1} [ν(C=O) in the thiazolidine ring] together with the carbamate carbonyl band at 1710 cm^{-1}. Singlets at δ 4.15 (2H) and 5.32 ppm (1H) in the ^1H NMR spectrum of **XI** were assigned to the C^5H_2 methylene group and 2-H proton in the thiazolidine ring.

Thus the described transformations of alkyl aminophenylcarbamates demonstrate a wide potential for their functionalization with formation of new compounds that are promising from the viewpoint of their biological activity.

EXPERIMENTAL

The ^1H NMR spectra were recorded on a Bruker DRX-500 spectrometer at 500.13 MHz using tetramethylsilane as internal reference. The ^{13}C NMR spectra were measured with complete decoupling from protons on a Bruker WM-400 instrument at 100 MHz using DMSO-d_6 as solvent. The IR spectra were recorded in the range from 4000 to 400 cm^{-1} on an InfraLUM FT-02 spectrometer with Fourier transform from samples prepared as KBr pellets. The mass spectra (electron impact, 70 eV) were obtained on

a Finnigan MAT INCOS 50 instrument. The purity of the isolated compounds was checked by TLC on Silufol UV-254 plates; spots were visualized by treatment with iodine vapor.

Polymeric diazotizing agent $[Polym]^+ NO_2^-$. Sodium nitrite, 2.07 g (30 mmol), was dissolved in 30 ml of distilled water, 3.5 g of AV-17-8 anion exchanger (an analog of Amberlyst A26) was added, the mixture was stirred for 10 min, and the exchanger was filtered off and washed with distilled water until neutral washings. The resulting polymeric diazotizing agent contained 3.5 mmol of nitrite ions per gram, which is consistent with the data of [6, 7].

Arenediazonium *p*-toluenesulfonates IIIa and IVa (*general procedure*). To a solution of *p*-toluenesulfonic acid in acetic acid we added 1 g of $[Polym]^+ NO_2^-$ and 1.2 mmol of aromatic amine **I** or **II**, and the mixture was stirred for 10 min. The precipitate was filtered off, and the product was isolated from the filtrate by precipitation with diethyl ether and dried in air.

4-Benzyloxycarbonylaminobenzenediazonium *p*-toluenesulfonate (IIIa). Yield 83%, colorless crystals, mp 169–170°C (decomp.). IR spectrum, ν, cm^{-1}: 3310 (NH), 2276 (N≡N), 1720 (C=O), 1610, 1575, 1560 (C=C_{arom}). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.29 s (3H, CH_3), 5.23 s (2H, C**H**$_2$Ph), 7.13 d (2H, H_{arom}, J = 8.5 Hz), 7.15–4.48 m (7H, H_{arom}), 7.92 d (2H, H_{arom}, J = 8.0 Hz), 8.55 d (2H, H_{arom}, J = 8.5 Hz), 11.15 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 21.21 (CH_3), 68.12 (**C**H_2Ph), 110.60 (2C, C_{arom}), 112.42 (C^i); 125.95, 127.14, 127.74, 127.84, 128.58, 135.90, 136.15, 138.36, 139.95, 143.10 (15C, C_{arom}); 153.93 (C=O). Mass spectrum, m/z (I_{rel}, %): 397 (1) $[M - N_2]^+$, 333 (1) $[M - N_2 - SO_2]^+$, 317 (1), 242 (3), 227 (2), 198 (3), 183 (1), 172 (3), 155 (3), 119 (1), 107 (14), 91 (100), 77 (7), 65 (17), 51 (7), 44 (20). Calculated: M 425.10.

4-Cyclohexyloxycarbonylaminobenzenediazonium *p*-toluenesulfonate (IVa). Yield 81%, colorless crystals, mp 193–195°C (decomp.). IR spectrum, ν, cm^{-1}: 3310 (NH), 2280 (N≡N), 1720 (C=O), 1610, 1570 (C=C_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 20.88 (CH_3); 23.38, 24.18, 31.54 (5C, CH_2); 69.28 (OCH), 111.12 (C^i); 112.56, 125.76, 128.75, 138.95, 139.04, 144.20, 145.78 (11C, C_{arom}); 154.02 (C=O). Mass spectrum, m/z (I_{rel}, %): 389 (3) $[M - N_2]^+$, 325 (1) $[M - N_2 - SO_2]^+$, 319 (1), 244 (3), 219 (2), 190 (3), 175 (1), 155 (3), 119 (2), 99 (100), 83 (15), 77 (6), 65 (17), 51 (6), 44 (19). Calculated: M 417.14.

Ethyl 5-benzyloxycarbonylamino-1*H*-indole-2-carboxylate (V). *a*. A solution of 1.54 g (0.01 mol) of ethyl α-methylacetoacetate in 1 ml of dioxane was cooled to 0°C, 3.5 ml of cold 50% aqueous potassium hydroxide was added, the mixture was diluted with water (20 ml), 4.3 g (0.01 mol) of diazonium salt **IIIa** was quickly added under stirring, and the mixture was stirred for 5 min. The aqueous phase was extracted with diethyl ether (2 × 15 ml), the combined extracts were dried over sodium sulfate, the solvent was distilled off under reduced pressure, the residue was dissolved in anhydrous ethanol, dry hydrogen chloride was passed through the solution until ammonium chloride began to separate therefrom, and the mixture was left overnight. The mixture was then poured into ice water, and the crystalline product was filtered off, dried in air, and recrystallized from ethanol. Yield 2.10 g (63%), light brown crystals, mp 168–169°C. IR spectrum, ν, cm^{-1}: 3330–3310 (NH), 1710, 1680 (C=O), 1620, 1590, 1555 (C=C_{arom}). ^1H NMR spectrum (acetone-d_6), δ, ppm: 1.32 t (3H, Me, J = 6.8 Hz), 4.20–4.33 m (4H, OCH_2), 5.81 s (1H, 3-H), 7.30–7.65 m (5H, H_{arom}), 7.81 d (1H, H_{arom}, J = 10 Hz), 7.93 d (1H, H_{arom}, J = 10 Hz), 8.10 s (1H, 4-H), 8.37 br.s (1H, NH), 9.50 br.s (1H, NH). Found, %: C 67.33; H 5.40; N 7.95. $C_{19}H_{18}N_2O_4$. Calculated, %: C 67.46; H 5.33; N 8.28.

b. Carbamate **I**, 2.42 g (0.01 mol), was dissolved in a mixture of 12.6 ml of hot water and 2.5 ml of 20% aqueous HCl, the mixture was cooled to 0–5°C, and an equal volume of hydrochloric acid was added. The resulting solution was subjected to diazotization by adding dropwise under stirring and efficient cooling a solution of 0.76 g (0.01 mol) of sodium nitrite in 20 ml of water. When the addition was complete, the mixture was kept for 1 h in the cold. A solution of 1.54 g (0.01 mol) of ethyl α-methylacetoacetate was cooled to 0°C, 3.5 ml of cold 50% aqueous potassium hydroxide was added, the mixture was diluted with ice water (20 ml), the solution of diazonium salt **IIIb** was quickly added, and the mixture was stirred for 5 min. The azo compound was isolated and subjected to heterocyclization to indole **V** as described above in *a*. Yield 1.76 g (52%), light brown crystals, mp 168–169°C. Found, %: C 67.39; H 5.35; N 7.99. $C_{19}H_{18}N_2O_4$. Calculated, %: C 67.46; H 5.33; N 8.28.

Ethyl 5-cyclohexyloxycarbonylamino-1*H*-indole-2-carboxylate (VI). *a*. Compound **VI** was synthesized as described above for **V** (method *a*) from 0.42 g (0.001 mol) of diazonium salt **IVa**. Yield 0.2 g (61%), light brown crystals, mp 163–165°C (from CH_2Cl_2–

petroleum ether, 1:2 by volume). IR spectrum, ν, cm^{-1}: 3340–3315 (NH), 1710, 1690 (C=O), 1610, 1590, 1535 (C=C_{arom}). ^1H NMR spectrum (acetone-d_6), δ, ppm: 1.16–1.47 m (9H, $CH_2C\mathbf{H}_3$, cyclohexyl), 2.20–2.27 m and 2.38–2.41 m (2H each, cyclohexyl), 4.40 q (2H, OCH_2, J = 6.7 Hz), 4.90–4.93 m (1H, OCH), 7.04 s (1H, H_{arom}), 7.28 s (1H, 3-H), 7.54 d (1H, H_{arom}, J = 7.7 Hz), 8.90 d (1H, H_{arom}, J = 7.7 Hz), 9.53 br.s (1H, 5-NH), 11.78 br.s (1H, 1-H). Found, %: C 65.43; H 6.58; N 8.36. $C_{18}H_{22}N_2O_4$. Calculated, %: C 65.46; H 6.67; N 8.49.

b. The procedure was analogous to that described above for compound **V** (method *b*). Light brown crystals, mp 163–165°C (from CH_2Cl_2–petroleum ether, 1:2 by volume). Found, %: C 65.48; H 6.56; N 8.34. $C_{18}H_{22}N_2O_4$. Calculated, %: C 65.46; H 6.67; N 8.49.

Benzyl {4-[(4-nitrophenyl)methylidene]aminophenyl}carbamate (VII). A mixture of 2.42 g (0.01 mol) of compound **I** and 1.51 g (0.01 mol) of 4-nitrobenzaldehyde in 25 ml of ethanol containing one drop of glacial acetic acid was heated for 5 h under reflux. The mixture was cooled, and the precipitate was filtered off, dried in air, and recrystallized from ethanol–dioxane (5:1). Yield 2.85 g (76%), yellow crystals, mp 159–160°C. IR spectrum, ν, cm^{-1}: 3310 (NH), 1710 (C=O), 1645 (C=N), 1610, 1595, 1575 (C=C_{arom}), 1517 ($\nu_{as}NO_2$), 1345 (ν_sNO_2). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 5.16 s (2H, $C\mathbf{H}_2Ph$), 7.32–7.51 m (9H, H_{arom}), 8.18 d (2H, H_{arom}, J = 8.0 Hz), 8.35 d (2H, H_{arom}, J = 8.0 Hz), 8.82 s (1H, N=CH), 9.92 br.s (1H, NH). Mass spectrum, m/z (I_{rel}, %): 376 (7) $[M + 1]^+$, 375 (16) $[M]^+$, 331 (6), 267 (5), 240 (55), 194 (26), 91 (97), 77 (17). Found, %: C 67.01; H 4.55; N 11.06. $C_{21}H_{17}N_3O_4$. Calculated, %: C 67.20; H 4.53; N 11.20. M 375.40.

Compounds **VIII** and **IX** were synthesized in a similar way.

Benzyl {4-[(2,4-dihydroxyphenyl)methylidene]aminophenyl}carbamate (VIII) was synthesized from 2.42 g (0.01 mol) of compound **I** and 1.38 g (0.01 mol) of 2,4-dihydroxybenzaldehyde. Yield 3.08 g (85%), yellow crystals, mp 152–153°C (from EtOH–dioxane, 5:1 by volume). IR spectrum, ν, cm^{-1}: 3310 (NH), 1710 (C=O), 1650 (C=N), 1610, 1595, 1575 (C=C_{arom}). Found, %: C 69.71; H 4.84; N 7.58. $C_{21}H_{18}N_2O_4$. Calculated, %: C 69.61; H 4.97; N 7.74.

Cyclohexyl {4-[(4-methoxyphenyl)methylidene]aminophenyl}carbamate (IX) was synthesized from 2.34 g (0.01 mol) of compound **II** and 1.36 g (0.01 mol) of 4-methoxybenzaldehyde. Yield 3.1 g (89%), yellow crystals, mp 135–137°C (from EtOH–dioxane, 5:1 by volume). IR spectrum, ν, cm^{-1}: 3315 (NH), 1710 (C=O), 1645 (C=N), 1610, 1595, 1575 (C=C_{arom}). Found, %: C 71.38; H 6.64; N 7.87. $C_{21}H_{24}N_2O_3$. Calculated, %: C 71.59; H 6.82; N 7.96.

Cyclohexyl {4-[3-chloro-2-(4-methoxyphenyl)-4-oxoazetidin-1-yl]phenyl}carbamate (X). Compound **IX**, 1.76 g (5 mmol), was dissolved in 25 ml of dioxane, 0.4 ml (5 mmol) of chloroacetyl chloride was added dropwise under continuous stirring at room temperature, the mixture was stirred for 10 min, 0.7 ml (5 mmol) of triethylamine was added, and the mixture was heated for 12 h under reflux, the progress of the reaction being monitored by TLC. When the reaction was complete, the mixture was poured into 100 ml of ice water, and the precipitate was filtered off, washed with water (20 ml) on a filter, dried in air, and recrystallized from ethanol. Yield 1.63 g (76%), colorless crystals, mp 158–160°C. IR spectrum, ν, cm^{-1}: 3310 (NH), 1710, 1715 (C=O), 1620, 1585, 1575 (C=C_{arom}). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.12–1.48 m (6H), 2.20–2.27 m (2H), and 2.37–2.40 m (2H) (cyclohexyl); 3.31 d (1H, CHCl, J = 1.6 Hz), 3.34 d (1H, 2-H, J = 5.5 Hz), 3.78 s (3H, OMe), 4.94–4.98 m (1H, OCH), 6.62 d (1H, H_{arom}, J = 8.7 Hz), 6.65 d (1H, H_{arom}, J = 8.7 Hz), 7.23 d (1H, H_{arom}, J = 8.7 Hz), 7.33–7.38 m (3H, H_{arom}), 7.53 d (1H, H_{arom}, J = 8.6 Hz), 7.56 d (1H, H_{arom}, J = 8.6 Hz), 9.57 br.s (1H, NH). Found, %: C 64.28; H 5.62; N 6.39. $C_{23}H_{25}ClN_2O_4$. Calculated, %: C 64.41; H 5.83; N 6.53.

Benzyl {4-[2-(4-nitrophenyl)-4-oxo-1,3-thiazolidin-3-yl]phenyl}carbamate (XI). A mixture of 1.88 g (5 mmol) of compound **VII** and 0.71 ml (0.01 mol) of sulfanylacetic acid in 12 ml of anhydrous dimethylformamide was heated for 12 h at 120°C. The mixture was cooled to room temperature and poured into 50 ml of 10% aqueous sodium hydrogen carbonate, and the precipitate was filtered off, washed with water (20 ml) on a filter, dried in air, and recrystallized from ethanol. Yield 1.46 g (65%), dark yellow crystals, mp 105–106°C. IR spectrum, ν, cm^{-1}: 3330 (NH), 1710, 1780 (C=O), 1610, 1585, 1570 (C=C_{arom}), 1517 ($\nu_{as}NO_2$), 1345 (ν_sNO_2), 687 (C–S–C). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 4.15 s (2H, CH_2), 5.21 s (2H, $C\mathbf{H}_2Ph$), 5.32 s (1H, 2-H), 7.22–7.29 m (7H, H_{arom}), 7.40 d (1H, H_{arom}, J = 9.0 Hz), 7.48 d (1H, H_{arom}, J = 9.0 Hz), 7.56 d (2H, H_{arom}, J = 8.7 Hz), 7.90 d (2H,

H_{arom}, J = 9.0 Hz), 9.62 br.s (1H, NH). Found, %: C 61.29; H 4.11; N 9.40. $C_{23}H_{19}N_3O_5S$. Calculated, %: C 61.47; H 4.23; N 9.35.

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