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Alkenylation of Arylamines and *N*-Arylacetamides with Acetylene Compounds in Superacids

A. O. Shchukin[a], A. V. Vasil'ev[a], S. A. Aristov[a], G. K. Fukin[b], and A. P. Rudenko[a]

[a]*St. Petersburg State Academy of Forestry Engineering, St. Petersburg, 194021 Russia*
e-mail: aleksvasil@mail.ru
[b]*Razuvaev Institute of Organometallic Chemistry, Russian Academy of Sciences,Russia*

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Abstract—Vinyl type cations generated in superacid HSO_3F by the protonation of the triple bond of acetylene compounds efficiently react with arylammonium ions and *N*-arylacetamides yielding alkenylation products of the aromatic rings in the given amino derivatives. The regio- and stereoselectivity of electrophilic aromatic substitution was investigated involving vinyl type cations and arylammonium ions or *N*-arylacetamides in HSO_3F.

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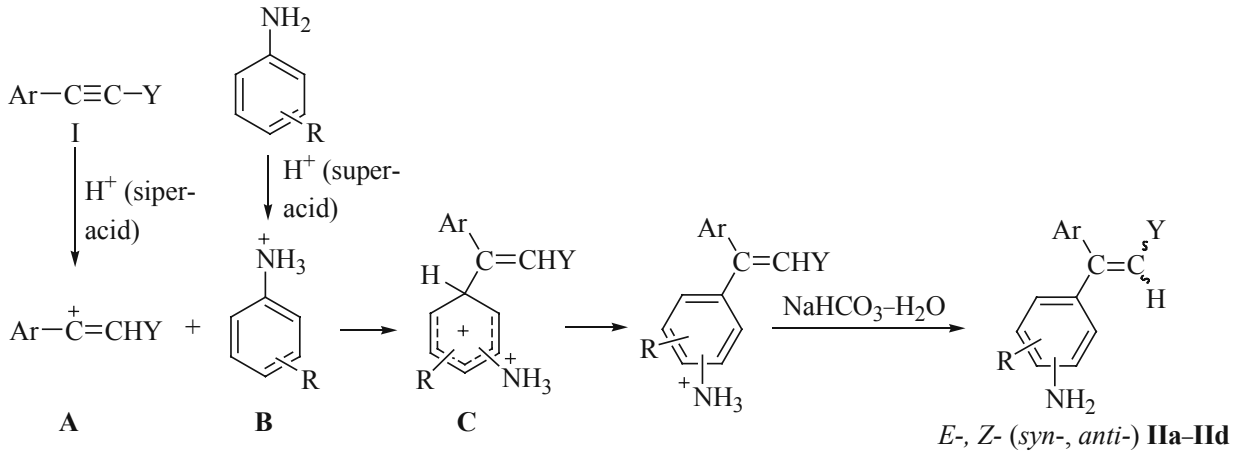
Arylamines (aniline derivatives) are widely employed in the synthesis of organic compounds [1], they are practically important for production of dyes, pharmaceuticals, and other valuable products [2]. The development of procedures for aniline derivatives preparation is an urgent target.

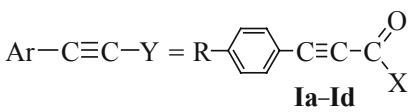
In a series of studies [3–8] we demonstrated a new method for aromatic compounds alkenylation with vinyl type cations generated by the protonation of an acetylene bond of alkynes in superacids.

Here we report on reactions of acetylene compounds **Ia–Id** (Scheme 1) with arylamines and N-arylacetamides in superacid HSO_3F at low temperature –75...–30°C. Problems are considered of regio- and stereoselectivity of electrophilic aromatic substitution involving vinyl type cations and arylammonium ions or *N*-arylacetamides.

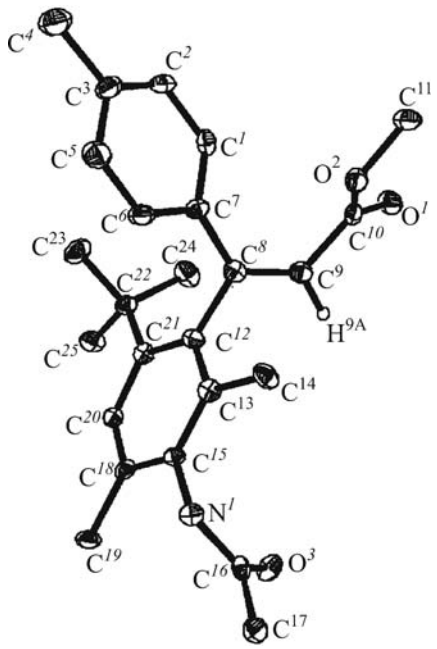
Aniline derivatives exist in superacids as arylammonium ions **B** [9] (Scheme 1). Reactions of cations **A** formed of compounds **I** with ions **B** occur through a unique for electrophilic aromatic substitution

Scheme 1.





X = OMe, R = H (**a**), Me (**b**), MeO (**c**); X = R = Me (**d**).



Structure of (*E*)—methyl 3-(3-acetylamino-6-*tetr*-butyl-2,4-dimethylphenyl)-3-(4-methylphenyl)propenoate *E*-**IIIj** according to XRD data.

intermediate formation of a double-charged arenonium ion **C**. The deprotonation of ion **C** and the subsequent workup of the reaction mixture (treating with concn. HCl at –70°C, then neutralization with a saturated aqueous $NaHCO_3$ solution) resulted in the final products **II** of alkenylation of the aromatic rings in the initial arylamines (Scheme 1).

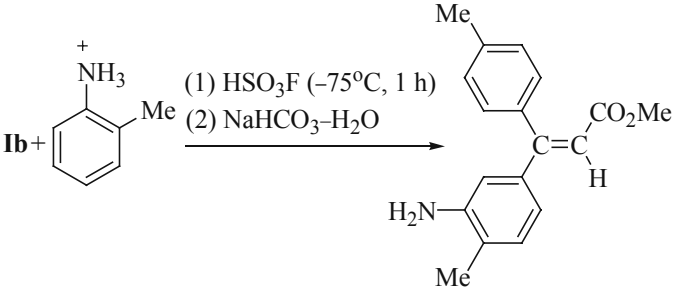
We formerly established [7, 8, 10, 11] that the proton and the aryl moiety (or the superacid anion) added to the triple bond of acetylene substrate in superacids at low temperature (–75...–50°C) by a *syn*-type. At higher temperature (–30...20°C) in the superacids the *syn*-products transformed into *anti*-isomers.

The precise *E*-/*Z*-configuration of alkenylation products of arylamines and their derivatives *E*-/*Z*-(**IIa–IIz** and **IIIa–IIIm**) presented on Schemes 2–14 was established from the data of XRD analysis of *E*-**IIIj** substance (see the figure) and ^1H NMR spectra. Like in [7, 8] the stereochemical reference for establishing the *E*-/*Z*-configuration of compounds **IIa–IIz** and **IIIa–IIIm** was the signal of the vinyl proton at the double bond =C=CH–. This proton signal in the majority of *E*-isomers is observed at δ 5.78–6.42 ppm, and in *Z*-isomers, in the region δ 6.45–6.88 ppm.

Electron-acceptor group NH_3^+ deactivates the π-system of the aromatic ring in ion **B** (Scheme 1) with respect to electrophilic substitution. For instance, the protonated forms of aniline and *N,N*-dimethylaniline in HSO_3F at –30°C do not react with vinyl type cations formed from compounds **Ia** and **Id** and do not give products of **II** structure.

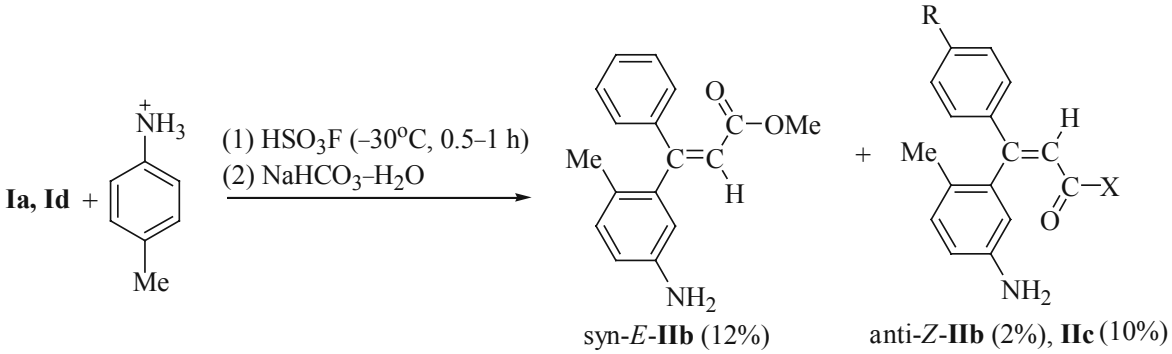
The presence of one methyl group in the aromatic ring of 2-methylaniline made it possible to involve the corresponding arylammonium ion into the substitution reaction with the vinyl cataion generated from compound **Ib** (Scheme 2). As a result of a concerted orientation of

Scheme 2.



syn-*E*-**IIa** (17%)

Scheme 3.



syn-*E*-**IIb** (12%) anti-*Z*-**IIb** (2%), **IIc** (10%)

X = OMe, R = H (**b**); X = R = Me (**c**).

electron-donor CH_3 group and electron-acceptor NH_3^+ group the substitution occurred in the para-position to the methyl group with the stereoselective formation of a product of *syn*-addition *E*-**IIa**.

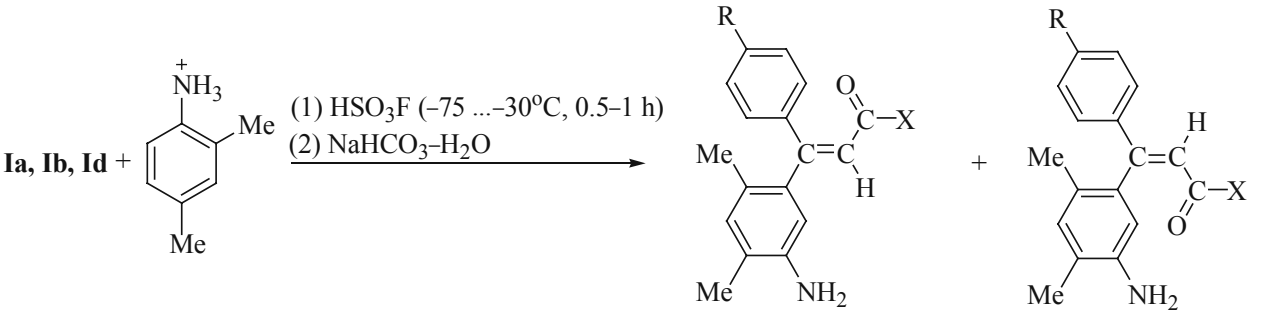
In reaction of acetylene derivatives **Ia** and **Id** with 4-methylphenylammonium ion in HSO_3F at –30°C formed a mixture of substances *E*-/*Z*-**IIb** or compound *Z*-**IIc** respectively that were products of substitution in the *ortho*-position to the methyl group of the initial 4-methylaniline (Scheme 3). A similar regioselectivity (Schemes 2 and 3) was previously observed in reaction of trifluoromethylation and bromination of 2- and 4-methylanilines in superacids [12, 13].

The alkenylation of 2,4-dimethylphenylammonium ion with acetylene derivatives **Ia, Ib**, and **Id** occurred regioselectively exclusively in the position *5* of the ring of the arylammonium ion giving products of *syn*- and *anti*-addition *E*-/*Z*-(**IId–IIf**) (Scheme 4). The ^{1}H NMR spectra of compounds *E*-/*Z*-(**IId–IIf**) contain two narrow singlets in the region δ 6.38–6.93 ppm corresponding to aromatic protons in the positions *2* and *5* of the benzene ring containing an amino group (see EXPERIMENTAL). This character of spectra excluded the formation of alternative substitution products in positions *3* or *6* of the aromatic ring of the initial amine, for in this case the aromatic protons would appear in the ^{1}H NMR spectra of the alkenylation product as doublets.

Similarly the analysis of ^{1}H NMR spectra of the individually isolated products *E*-(**IIg** and **IIh**) of alkenylation of 3,4-dimethylphenylammonium ion (Scheme 5) unambiguously indicated the formation of two isomers originating from the substitution at the positions *6* and *5* of the ring of arylammonium ion respectively. In the spectrum of compound *E*-**IIg** two singlets are observed at δ 6.43 and 6.83 ppm belonging to aromatic protons in the positions *3* and *6* of the ring substituted with amino group. In the spectrum of isomer *E*-**IIh** two characteristic doublets appeared at δ 6.39 and 6.51 ppm with a coupling constant of 2.3 Hz corresponding to two aromatic protons in the *meta*-position to each other (see Experimental).

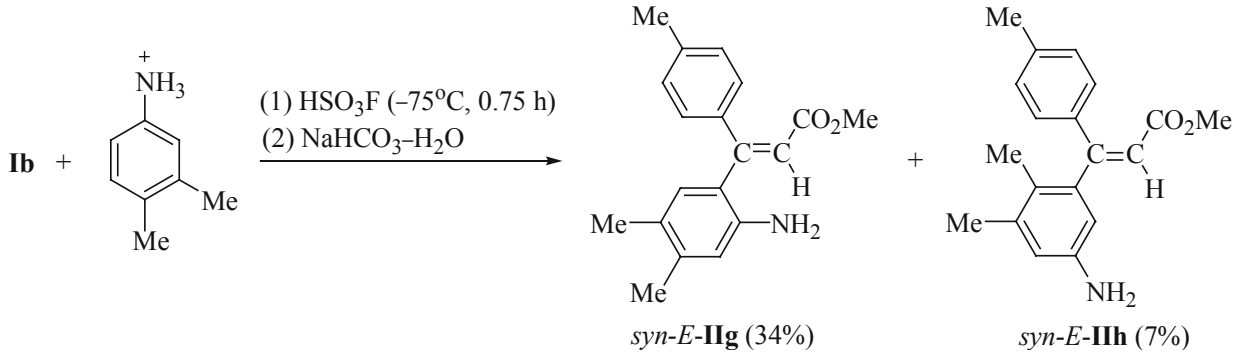
Vinyl type cations generated from acetylene substrates **Ia–Id** in HSO_3F at –75...–30°C are sufficiently reactive for the electrophilic attack even on sterically hindered arylammonium ions with methyl and bulky *tert*-butyl

Scheme 4.



X = OMe, R = H (**d**), Me (**e**); X = R = Me (**f**).

Scheme 5.



substituents (Schemes 6–8). Therewith the preparative yields of the alkenylation products *E*-/*Z*-(**IIi**–**IIw**) were 11–44% (see Experimental).

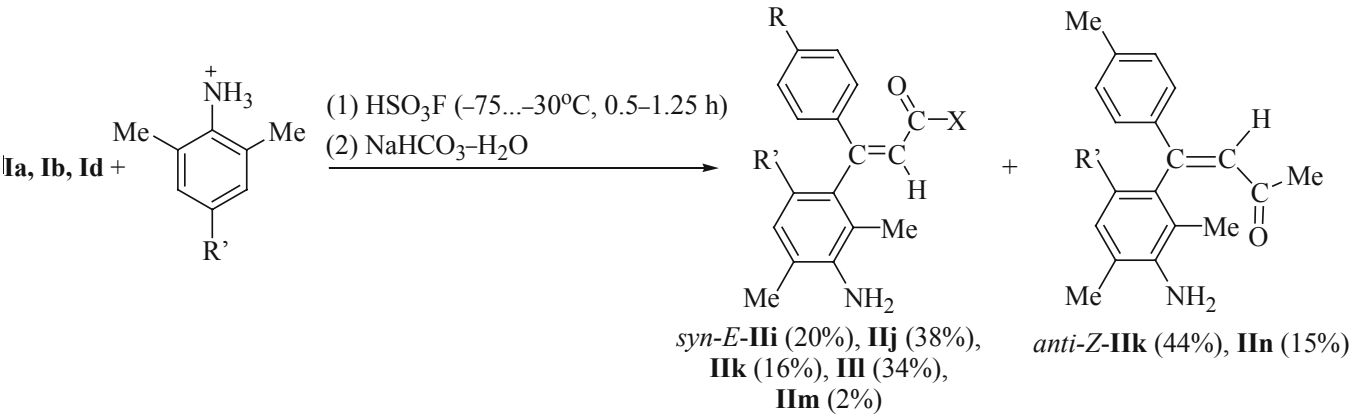
Special attention required the formation of substances *E*-(**IIs** and **IIt**) (Scheme 7) and *E*-(**IIu** and **IIv**), *Z*-**IIw** (Scheme 8), products of substitution in *ortho*- and *para*-position with respect to NH_3^+ group despite its electron-acceptor character. The chromatographic behavior of compounds *E*-(**IIs** and **IIt**) also should be noted: Their R_f values (TLC on silica gel) exceed the R_f value of the initial 2,4,5-trimethylphenylamine. It is apparently due to the spatial shielding of the *ortho*-amino group in structures *E*-(**IIs** and **IIt**) and consequently to the decrease in the sorption ability of compounds *E*-(**IIs** and **IIt**).

The primary formation of products of *syn*-addition to the acetylene bond of substrates **Ia–Id** was demonstrated by an example of compound ***E*-IIv** that later after keeping in HSO_3F at –30°C for 1 h converted into *anti*-*Z*-isomer **IIv** (Scheme 8).

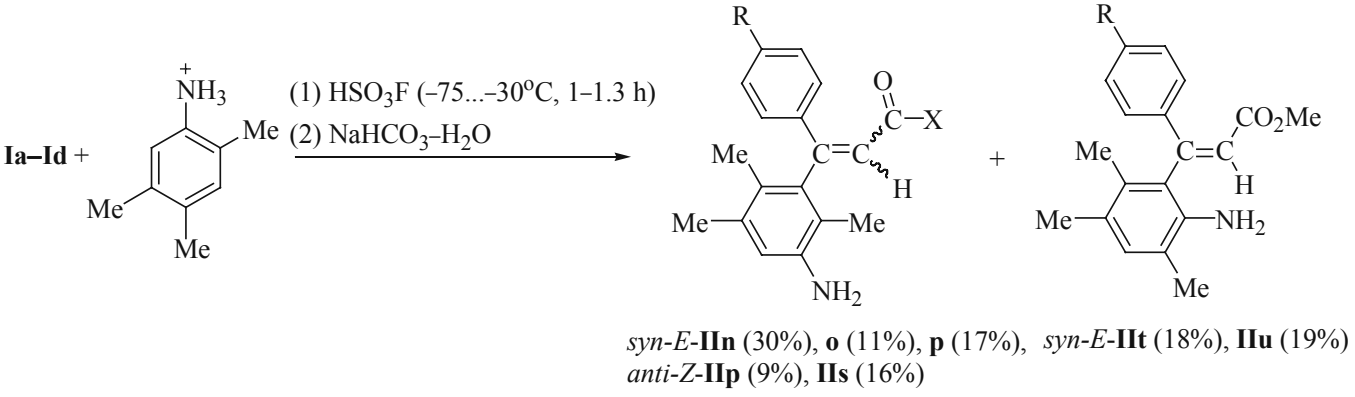
The electrophilic substitution involving 2-methoxyphenylammonium ion and vinyl cation generated from acetylene ketone **Id** occurred at the position *5* of the ring of arylammonium ion and yielded a mixture of compounds *E*-/*Z*-**IIy** (Scheme 9). In a similar reaction with acetylene ester **Ib** two compounds *E*-**IIx** and *E*-**IIz** were obtained due to substitution of the positions *5* and *3* of the aromatic ring of 2-methoxyphenylammonium ion respectively (Scheme 9). The structure of compound *E*-**IIz** was established from its ^{1}H NMR spectrum containing a complex multiplet signal in the δ 6.64–6.71 ppm characteristic of the spin system *ABC* of three contiguous aromatic protons in the ring with an amino group (see EXPERIMENTAL).

In contrast to phenylammonium ion (see above) 1-naphthylammonium ion easily undergoes alkenylation

Scheme 6.

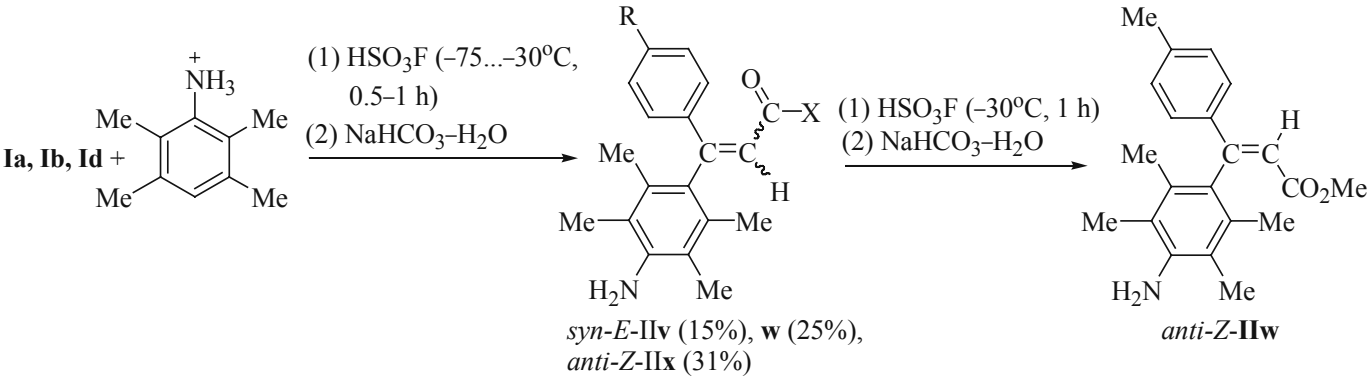


Scheme 7.



X = OMe, R = H, R' = Me (**i**), *t*-Bu (**l**); X = OMe, R = Me, R' = Me (**j**), *t*-Bu (**m**); X = Me, R' = Me (**k**), *t*-Bu (**n**). X = OMe, R = H (**o**), Me (**p**), MeO (**q**); X = R = Me (**r**); R = H (**s**), Me (**t**).

Scheme 8.



X = OMe, R = H (**u**), Me (**v**); X = R = Me (**w**).

with acetylene ester **Ia** in HSO_3F at –30°C within 1 h. However this reaction possesses low regio- and stereoselectivity. In the ^1H NMR spectrum of the mixture of alkenylation products in the region characteristic of the protons at the double bond =C=CH– at δ 6.17–6.76 ppm 8 singlets were registered corresponding to various regio and stereo-(*E*-/*Z*-) isomeric products of the naphthalene skeleton alkenylation.

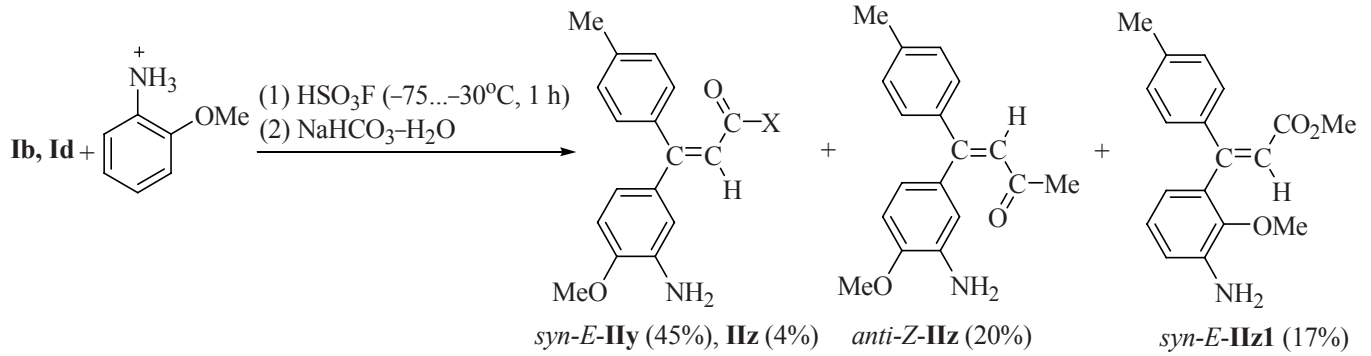
On the other hand the presence of two NH_3^+groups at the benzene ring completely deactivated the π-system for the electrophilic substitution. Thus the reaction of 1,3-diamine-2,4,6-trimethylbenzene with acetylene compound **Ib** in HSO_3F at –30°C in 0.5 h failed to provide products of alkenylation of the corresponding arylammonium ion.

N-Acetyl derivatives of arylamines (*N*-arylacetamides) readily enter into the alkenylation by acetylene compounds in superacids. According to the published data [14, 15] the protonation of amide group in superacids occurred predominantly at the carbonyl group oxygen. Still the NHAc group behaves as *ortho*-, *para*-director in the processes of electrophilic aromatic substitution even in strong superacid systems like $HF–SbF_5$, for instance, in reactions of trifluoromethylation and hydroxylation of N-arylacetamides [12, 16].

N-Phenylacetamide unlike aniline proper (see above) reacted with vinyl type cations generated from acetylene derivatives **Ia, Ib**, and **Id** in HSO_3F at –75...–30°C within 0.5–1 h giving substances *E*-/*Z*-(**IIIa–IIIc**), products of substitution in the *para*-position of the phenyl ring of the initial *N*-phenylacetamide (Scheme 10, see analogous regioselectivity in [12]).

Mono- and dimethyl-substituted in the aromatic ring *N*-arylacetamides gave rise to alkenylation products *E*-/*Z*-(**IIId–IIIf**) in reactions with acetylene compounds **Ib** and **Id** in HSO_3F (Scheme 11). From *N*-4-methylphenyl-acetamide formed the products of substitution in the *ortho*-position to the methyl group *E*-**IIId** and *Z*-**IIIe**

Scheme 9.



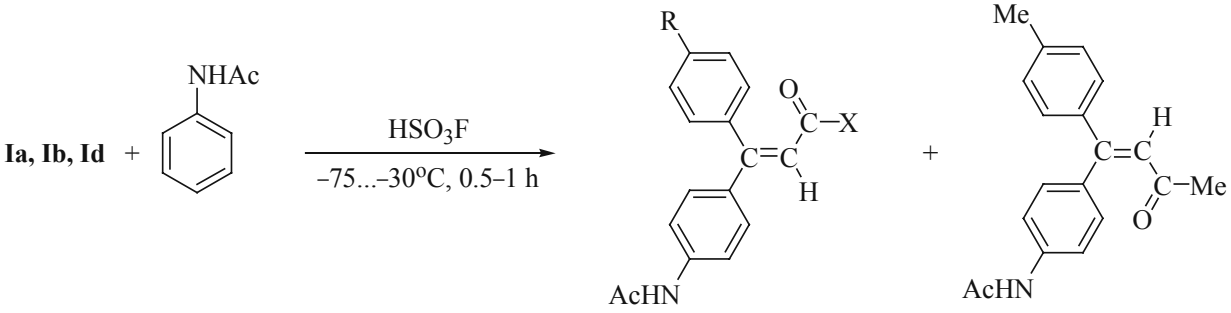
X = OMe (**x**), Me (**y**).

(Scheme 11) as had been previously observed in the trifluoromethylation of this amide in the system HF–SbF_5 [12].

Analogously to arylammonium ions (Schemes 6–8) the sterically hindered N-arylacetamide also reacts with cations of vinyl type to give alkenylation products *E*-/*Z*-(**IIIg–IIIm**) in 29–67% yield (see Experimental, Schemes 12–14). In the ^{1}H NMR spectra of compounds *Z*-(**IIIh** and **IIIi**) the signal from the proton attached to the nitrogen in the NHAc group was not observed, in the spectra of the other acetamides *E*-/*Z*-(**IIIa–IIIm**) the signal of this proton appeared as a broadened singlet in the region δ 6.6–7.6 ppm (see Experimental).

The reaction of *N*-2,4,5-trimethylphenylacetamide with acetylene ester **Ib** in HSO_3F at –75°C for 0.5 h resulted in the formation of substitution products at each

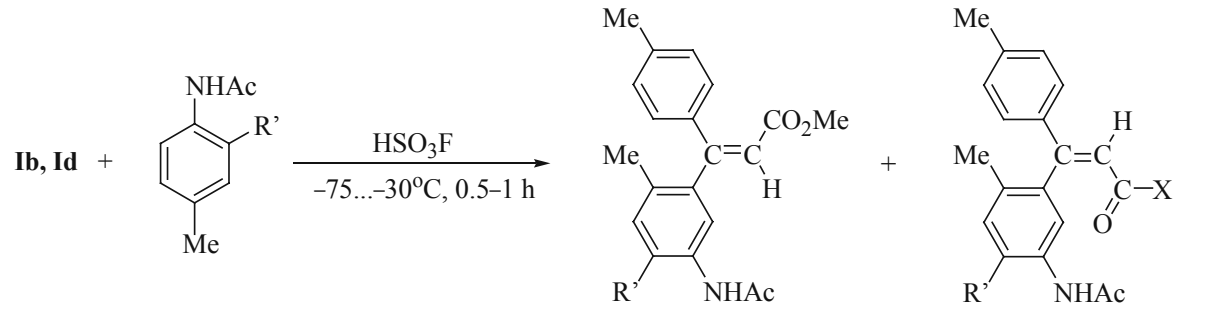
Scheme 10.



syn-E-**IIIa** (4%), **IIIb** (18%), **IIIc** (3%) *anti-Z*-**IIIc** (3%)

X = OMe, R = H (**a**), Me (**b**); X = R = Me (**c**).

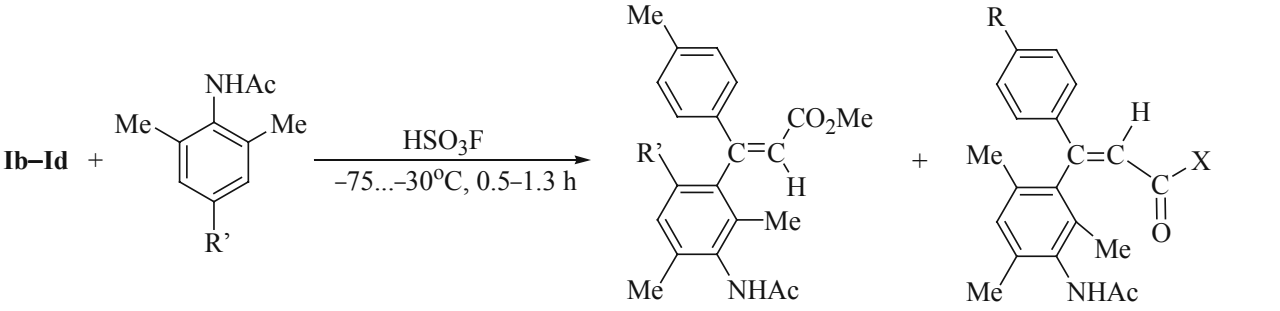
Scheme 11.



syn-E-**IIId** (29%), **IIIf** (13%) *anti-Z*-**IIIe** (17%), **IIIf** (13%)

R' = H (**d**); X = Me, R' = H (**e**); X = OMe, R' = Me (**f**).

Scheme 12.



syn-E-**IIIg** (51%), **IIIj** (67%) *anti-Z*-**IIIh** (52%), **IIIi** (29%)

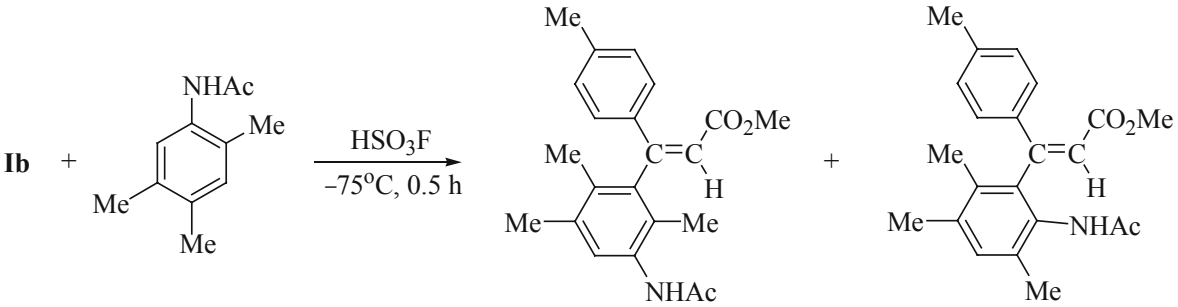
R' = Me (**g**), *t*-Bu (**j**); X = R = OMe (**h**), Me (**i**).

position in the aromatic ring of the initial *N*-arylacetamide, substances *E*-(**IIIk and IIIl**) (Scheme 13) in a ratio ~1:1 in keeping with the *ortho*-, *para*-director behavior of the NHAc group.

The study of protonation of compound *E*-**IIp** by ^{1}H NMR spectroscopy showed the formation of dication **IV** in HSO_3F at –80°C (Scheme 15). In the spectrum of ion **IV** signals were observed from the NH_3^+ group at δ 7.39 ppm [9] and of a proton which added to the carbonyl oxygen (O-protonation) at δ 12.49 ppm [6, 11] (see Experimental). In these conditions the protonation of the C^2 atom of double bond did not occur (cf. with data in [17]). O-Protonation in ion **IV** weakens the double bond character of the fragment C^2=C^3 (resonance structure **V** in Scheme 15) and facilitates the rotation around this bond favoring the transformation of *E*-forms into their *Z*-analogs at higher temperature (–30°C), as has been shown by an example of isomerization of compounds *E*-**IIv**→*Z*-**IIv** (Scheme 8).

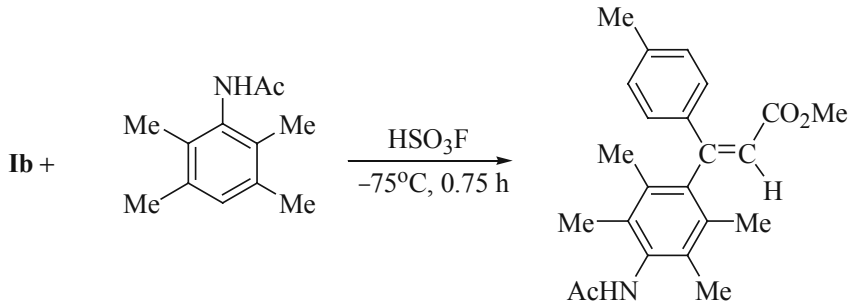
In the same way according to ^{1}H NMR spectrum compound **VI** exists in HSO_3F at –80°C as O-protonated at the carbonyl group ion **VII** (see Experimental, Scheme

Scheme 13.



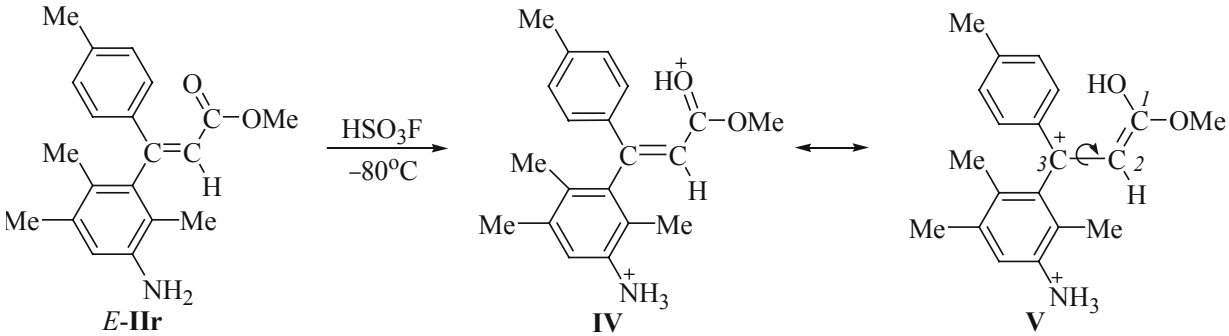
syn-*E*-**IIIk** (20%) *anti*-*E*-**IIIl** (16%)

Scheme 14.



syn-*E*-**IIIm** (32%)

Scheme 15.



16). The protonation of the carbon atom of the double bond the propenone fragment of molecule **VI** also was not observed. The formation in HSO_3F of *O*-proton-ated form **IV** and **VII** underlies the *E*-/*Z*-isomerization in superacids of 3,3-diarylpropenone structures *E*-/*Z*-(**IIa–IIz** and **IIIa–IIIm**) considered in this study and the other propenone systems [7, 8, 10, 11].

As a result of this investigation a new method was developed of regio- and stereoselective alkenylation of arylamines (or *N*-arylacetamides) based on the reaction of vinyl type cations with arylammonium ions (or *N*-aryl-acetamides) in superacids.

EXPERIMENTAL

^{1}H NMR spectra were registered on a spectrometer Bruker AM-500 (operating frequency 500 MHz) from solutions in $CDCl_3$. The residual signal of $CHCl_3$ (δ_H 7.25 ppm) was used as a reference. IR spectra were recorded on a spectrophotometer Specord 75IR from solutions in $CHCl_3$. Mass spectra were taken on MKh-1321 instrument, ionizing electrons energy 70 eV, direct admission of the sample into the ion source at 100–120°C. ^{1}H NMR spectra in HSO_3F at –80°C were measured on a spectrometer Bruker Avance 400 (operating frequency 400 MHz) with respect to internal reference CH_2Cl_2 (δ_H 5.32 ppm).

X-ray diffraction experiment was performed on an automatic diffractometer Smart APEX (graphite monochromator, MoK_α radiation, ω-θ scanning). The structure was solved by the direct method and refined by the least-squares method by F^2_{hkl} in an anisotropic approximation for all nonhydrogen atoms. Hydrogen atoms were found from the difference Fourier synthesis and refined isotropically. All calculations were carried out using software package SHELXTL v. 6.10 [18].

The single crystal of compound ***E*-IIIj** of the size 0.65×0.09×0.05 mm was obtained for XRD measurements by slow evaporation at room temperature of a solution of the substance in methanol within several days. Crystal of $C_{25}H_{31}NO_3$ at 100 K orthorhombic, *a* 9.3382(8), *b* 14.1577(10), *c* 17.0069(10) Å, $\alpha = \beta = \gamma = 90°$, *V* 2248.4(3) Å^3, *Z* 4, space group P-2, d_{calc} 1.162 g/cm^3, μ 0.075 mm^{-1}, $1.87 \le \theta \le 29.37°$, 23856 reflexions were measured, 6086 among them independent (R_{int} 0.1193), R_1 0.1247 [$I > 2\sigma(I)$], wR_2 0.1098 (for all reflexions).

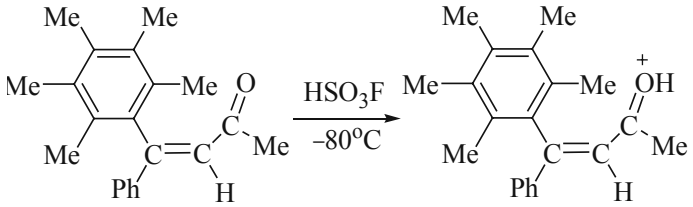
The preparation and properties of methyl 3-phenylpropionate (**Ia**), methyl 3-(4-methylphenyl)propionate (**Ib**) are given in [19], methyl 3-(4-methoxyphenyl)-propionate (**Ic**), in [20], 4-(4-methylphenyl)but-3-yn-2-one (**Id**), in [21].

Arylamines and *N*-arylacetamides were commercial products. In alkenylation reactions arylamines proper were used or their sulfates in an equivalent amount.

Alkenylation of arylamines and *N*-arylacetamides with acetylene derivatives Ia–Id in HSO_3F. General procedure. To a solution of 0.1–0.8 mmol of arylamine (or *N*-arylacetamide) in 0.7–1.0 ml of HSO_3F at –75... –30°C was added gradually in 10–20 min while vigorous stirring 0.09–0.32 mmol of acetylene derivative **Ia–Id**. The reaction mixture was stirred for 20–60 min more and then it was poured into 15–20 ml of concn. HCl cooled to –75°C. The mixture obtained after quenching was diluted with water (30–50 ml), warmed to room temperature, and extracted with chloroform (3×30 ml). The combined extracts were washed with water, with a saturated solution of $NaHCO_3$, again with water, and dried with Na_2SO_4, the solvent was distilled off in a vacuum of a water-jet pump, the residue was subjected to column chromatography on silica gel (eluent petroleum ether–ethyl acetate). The yield of final products *E*-/*Z*-(**IIa–IIz**) and *E*-/*Z*-(**IIIa–IIIm**) was evaluated by the weight of fractions obtained by chromatography.

(*E*)-Methyl 3-(3-amino-4-methylphenyl)-3-(4-methylphenyl)propenoate (IIa) was obtained from 30 mg (0.17 mmol) of compound **Ib** and 24 mg (0.23 mmol) of 2-methylphenylamine in 1 ml of HSO_3F

Scheme 16.



VI **VII**

at –75°C in 1 h. Yield 8 mg (17%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 2.15 s (3H, Me), 2.38 s (3H, Me), 3.59 br.s (2H, NH_2), 3.60 s (3H, OMe), 6.28 s (1H, =CH–), 6.55 d (1H_{arom}, *J* 1.7 Hz), 6.67 d.d (1H_{arom}, *J* 7.8, 1.7 Hz), 6.99 d (1H_{arom}, *J* 7.8 Hz), 7.09 d (2H_{arom}, *J* 8 Hz), 7.17 d (2H_{arom}, *J* 8 Hz). Mass spectrum, *m/z* (I_{rel}, %): 281 (100) [*M*]$^+$, 266 (3), 250 (28). Found, %: C 76.60; H 7.15; N 4.87. $C_{18}H_{19}NO_2$. Calculated, %: C 76.84; H 6.81; N 4.98. *M* 281.14.

(*E*)- and (*Z*)-Methyl 3-(5-amino-2-methylphenyl)-3-phenylpropenoates (IIb) were obtained from 50 mg (0.31 mmol) of compound **Ia** and 83 mg (0.775 mmol) of 4-methylphenylamine in 1 ml HSO_3F at –30°C in 30 min as an oily isomers mixture.

Compound *E*-**IIb**. Yield 11 mg (12%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.94 s (3H, Me), 3.64 s (3H, OMe), 3.58 br.s (2H, NH_2), 5.98 s (1H, =CH–), 6.54 d (1H_{arom}, *J* 2.5 Hz), 6.59 d.d (1H_{arom}, *J* 8.1, 2.5 Hz), 6.92 d (1H_{arom}, *J* 8.1 Hz), 7.23–7.30 m (5H_{arom}).

Compound *Z*-**IIb**. Yield 1.5 mg (2%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.95 s (3H, Me), 3.60 s (3H, OMe), 3.58 br.s (2H, NH_2), 6.41 d (1H_{arom}, *J* 2.5 Hz), 6.48 s (1H, =CH–), 6.63 d.d (1H_{arom}, *J* 8.1, 2.5 Hz), 7.02 d (1H_{arom}, *J* 8.1 Hz), 7.23–7.30 m (5H_{arom}). Mass spectrum (isomers mixture), *m/z* (I_{rel}, %): 267 (100) [*M*]$^+$, 236 (33), 207 (56), 206 (47), 197 (47), 130 (33), 115 (28). Found, %: C 76.47; H 6.30; N 5.43. $C_{17}H_{17}NO_2$. Calculated, %: C 76.38; H 6.41; N 5.24. *M* 267.13.

(*Z*)-4-(5-Amino-2-methylphenyl)-4-(4-methylphenyl)but-3-en-2-one (IIc) was obtained from 50 mg (0.32 mmol) of compound **Id** and 86 mg (0.8 mmol) of 4-methylphenylamine in 1 ml of HSO_3F at –30°C in 1 h. Yield 8 mg (10%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.82 s (3H, Me), 1.95 s (3H, Me), 2.34 s (3H, Me), 3.62 br.s (2H, NH_2), 6.45 d (1H_{arom}, *J* 2.4 Hz), 6.64 s (1H, =CH–), 6.66 d.d (1H_{arom}, *J* 8.0, 2.4 Hz), 7.03 d (1H_{arom}, *J* 8.0 Hz), 7.11 d (2H_{arom}, *J* 8.1 Hz), 7.22 d (2H_{arom}, *J* 8.1 Hz). Mass spectrum, *m/z* (I_{rel}, %): 265 (70) [*M*]$^+$, 250 (100), 222 (33), 207 (20), 130 (25), 43 (20). Found, %: C 81.64; H 7.36; N 5.20. $C_{18}H_{19}NO$. Calculated, % : C 81.47; H 7.22; N 5.28. *M* 265.15.

(*E*)-Methyl 3-(5-amino-2,4-dimethylphenyl)-3-phenylpropenoate (IId) was obtained from 50 mg (0.31 mmol) of compound **Ia** and 94 mg (0.78 mmol) of 2,4-dimethylphenylamine in 1 ml of HSO_3F at –30°C in 30 min. Yield 12 mg (14%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.93 s (3H, Me), 2.14 s (3H, Me), 3.50 br.s (2H, NH_2), 3.64 s (3H, OMe), 5.98 s (1H, =CH–), 6.52 s (1H_{arom}), 6.83 s (1H_{arom}), 7.23–7.30 m (5H_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 281 (100) [*M*]$^+$, 256 (30), 221 (30), 220 (20), 207 (40). Found, %: C 76.61; H 7.02; N 4.95. $C_{18}H_{19}NO_2$. Calculated, %: C 76.84; H 6.81; N 4.98. *M* 281.14.

(*E*)- and (*Z*)-Methyl 3-(5-amino-2,4-dimethylphenyl)-3-(4-methylphenyl)propenoates (IIe) were obtained from 30 mg (0.17 mmol) of compound **Ib** and 21 mg (0.17 mmol) of 2,4-dimethylphenylamine in 1 ml of HSO_3F at –75°C in 1 h as an oily isomers mixture.

Compound *E*-**IIe**. Yield 13 mg (24%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.93 s (3H, Me), 2.14 s (3H, Me), 2.34 s (3H, Me), 3.47 s (2H, NH_2), 3.65 s (3H, OMe), 5.93 s (1H, =CH–), 6.51 s (1H_{arom}), 6.82 s (1H_{arom}), 7.09 d (2H_{arom}, *J* 8 Hz), 7.14 d (2H_{arom}, *J* 8 Hz).

Compound *Z*-**IIe**. Yield 6 mg (13%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.94 s (3H, Me), 2.17 s(3H, Me), 2.34 s (3H, Me), 3.60 s (3H, OMe), 3.69 s (2H, NH_2), 6.38 s (1H_{arom}), 6.45 s (1H, =CH–), 6.92 s (1H_{arom}), 7.10 d (2H_{arom}, *J* 7.8 Hz), 7.23 d (2H_{arom}, *J* 7.8 Hz). Mass spectrum (isomers mixture), *m/z* (I_{rel}, %): 295 (100) [*M*]$^+$, 280 (9), 264 (26), 235 (22), 221 (38), 147.5 (5) [*M*]$^{2+}$. Found, %: C 77.41; H 7.05; N 4.73. $C_{19}H_{21}NO_2$. Calculated, %: C 77.26; H 7.17; N 4.74. *M* 295.16.

(*Z*)-4-(5-Amino-2,4-dimethylphenyl)-4-(4-methylphenyl)but-3-en-2-one (IIf) was obtained from 50 mg (0.32 mmol) of compound **Id** and 77 mg (0.64 mmol) of 2,4-dimethylphenylamine in 1 ml of HSO_3F at –30°C in 1 h. Yield 49 mg (56%). Oily substance. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.80 s (3H, Me), 1.93 s (3H, Me), 2.19 s (3H, Me), 2.33 s (3H, Me), 3.55 br.s (2H, NH_2), 6.43 s (1H_{arom}), 6.63 s (1H, =CH–), 6.93 s (1H_{arom}), 7.10 d (2H_{arom}, *J* 8.3 Hz), 7.22 d (2H_{arom}, *J* 8.3 Hz). Mass spectrum, *m/z* (I_{rel}, %): 279 (53) [*M*]$^+$, 264 (100), 236 (30), 221 (20). Found, %: C 81.61; H 8.02; N 4.93. $C_{19}H_{21}NO$. Calculated, %: C 81.68; H 7.58; N 5.01. *M* 279.16.

(*E*)-Methyl 3-(2-amino-4,5-dimethylphenyl)-3-(4-methylphenyl)propenoate (IIg) was obtained from 35 mg (0.2 mmol) of compound **Ib** and 49 mg (0.4 mmol) of 3,4-dimethylphenylamine in 1 ml of HSO_3F at –75°C in 0.75 h. Yield 20 mg (34%), mp 200°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 2.13 s (3H, Me), 2.17 s (3H, Me), 2.35 s (3H, Me), 3.42 br.s (2H, NH_2), 3.64 s (3H,

OMe), 6.10 s (1H, =CH–), 6.43 s (1H_{arom}), 6.83 s (1H_{arom}), 7.14 d (2H_{arom}, *J* 8 Hz), 7.21 d (2H_{arom}, *J* 8 Hz). Mass spectrum, *m/z* (I_{rel}, %): 295 (59) $[M]^+$, 264 (70), 263 (88), 248 (26), 236 (100), 222 (23). Found, %: C 77.21; H 7.17; N 4.82. $C_{19}H_{21}NO_2$. Calculated, %: C 77.26; H 7.17; N 4.74. *M* 295.16.

(*E*)-Methyl 3-(5-amino-2,3-dimethylphenyl)-3-(4-methylphenyl)propenoate (IIh) was obtained from 35 mg (0.2 mmol) of compound **Ib** and 49 mg (0.4 mmol) of 3,4-dimethylphenylamine in 1 ml of HSO_3F at –75°C in 0.75 h. Yield 4 mg (7%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.89 s (3H, Me), 2.15 s (3H, Me), 2.32 s (3H, Me), 3.65 s (5H, OMe, NH_2), 5.91 s (1H, =CH–), 6.39 d (1H_{arom}, *J* 2.3 Hz), 6.51 d (1H_{arom}, *J* 2.3 Hz), 7.09 d (2H_{arom}, *J* 8 Hz), 7.14 d (2H_{arom}, *J* 8 Hz). Mass spectrum, *m/z* (I_{rel}, %): 295 (100) $[M]^+$, 280 (14), 264 (25), 236 (35), 221 (39). Found, %: C 77.08; H 7.45; N 4.76. $C_{19}H_{21}NO_2$. Calculated, %: C 77.26; H 7.17; N 4.74. *M* 295.16.

(*E*)-Methyl 3-(3-amino-2,4,6-trimethylphenyl)-3-phenylpropenoate (IIi) was obtained from 30 mg (0.19 mmol) of compound **Ia** and 63 mg (0.47 mmol) 2,4,6-trimethylphenylamine in 1 ml of HSO_3F at –30°C in 0.5 h. Yield 11 mg (20%). Oily substance. IR spectrum, ν, cm^{-1}: 1620 (N–H), 1710 (C=O), 3300 (N–H), 3400 (N–H). ^{1}H NMR spectrum, δ, ppm: 2.07 s (3H, Me), 2.08 s (3H, Me), 2.17 s (3H, Me), 3.50 br.s (2H, NH_2), 3.67 s (3H, OMe), 5.87 s (1H, =CH–), 6.80 s (1H_{arom}), 7.26 s (5H_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 295 (100) $[M]^+$, 235 (21), 220 (43), 207 (15), 134 (21). Found, %: C 77.23; H 7.01; N 4.65. $C_{19}H_{21}NO_2$. Calculated, %: C 77.26; H 7.17; N 4.74. *M* 295.16.

(*E*)-Methyl 3-(3-amino-2,4,6-trimethylphenyl)-3-(4-methylphenyl)propenoate (IIj) was obtained from 15 mg (0.086 mmol) of compound **Ib** and 14 mg (0.1 mmol) of 2,4,6-trimethylphenylamine in 0.7 ml of HSO_3F at –75°C in 1 h. Yield 10 mg (38%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 2.05 s (3H, Me), 2.06 s (3H, Me), 2.16 s (3H, Me), 2.31 s (3H, Me), 3.51 br.s (2H, NH_2), 3.68 s (3H, OMe), 5.82 s (1H, =CH–), 6.79 s (1H_{arom}), 7.06 d (2H_{arom}, *J* 8.1 Hz), 7.15 d (2H_{arom}, *J* 8.1 Hz). Mass spectrum, *m/z* (I_{rel}, %): 309 (100) $[M]^+$, 278 (7), 249 (13), 234 (25), 221 (14). Found, %: C 77.48; H 7.47; N 4.53. $C_{20}H_{23}NO_2$. Calculated, %: C 77.64; H 7.49; N 4.53. *M* 295.16.

(*Z*)-4-(3-Amino-2,4,6-trimethylphenyl)-4-(4-methylphenyl)but-3-en-2-one (IIk) was obtained from 50 mg (0.32 mmol) of compound **Id** and 106 mg (0.79 mmol) of 2,4,6-trimethylphenylamine in 1 ml of HSO_3F at –30°C in 1.25 h. Yield 41 mg (44%). Oily substance. IR spectrum, ν, Cm^{-1}: 1590 (N–H), 1630 (C=O), 3300 (N–H), 3400 (N–H). ^{1}H NMR spectrum, δ, ppm: 1.72 s (3H, Me), 1.94 s (3H, Me), 1.95 s (3H, Me), 2.21 s (3H, Me), 2.33 s (3H, Me), 3.56 br.s (2H, NH_2), 6.80 s (1H, =CH–), 6.86 s (1H_{arom}), 7.10 d (2H_{arom}, *J* 8.1 Hz), 7.24 d (2H_{arom}, *J* 8.1 Hz). Mass spectrum, *m/z* (I_{rel}, %): 293 (100) $[M]^+$, 278 (96), 263 (10), 234 (31), 220 (21). Found, %: C 81.90; H 7.68; N 4.71. $C_{20}H_{23}NO$. Calculated, %: C 81.87; H 7.90; N 4.77. *M* 293.18.

(*E*)-Methyl 3-(3-amino-6-*tert*-butyl-2,4-dimethylphenyl)-3-phenylpropenoate (IIl) was obtained from 29 mg (0.18 mmol) of compound **Ia** and 32 mg (0.18 mmol) of 4-*tert*-butyl-2,6-dimethylphenylamine in 1 ml HSO_3F at –30°C in 1.5 h. Yield 10 mg (16%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.22 s (9H, CMe_3), 2.01 s (3H, Me), 2.22 s (3H, Me), 3.62 br.s (2H, NH_2), 3.72 s (3H, OMe), 5.91 s (1H, =CH–), 7.13 s (1H_{arom}), 7.25 m (3H_{arom}), 7.30 m (2H_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 337 (100) $[M]^+$, 322 (87), 290 (21), 280 (7), 262 (43), 248 (47). Found, %: C 78.40; H 8.14; N 4.15. $C_{22}H_{27}NO_2$. Calculated, %: C 78.30; H 8.06; N 4.15. *M* 337.2.

(*E*)-Methyl 3-(3-amino-6-*tert*-butyl-2,4-dimethylphenyl)-3-(4-methylphenyl)propenoate (IIm) was obtained from 22 mg (0.13 mmol) of compound **Ib** and 22 mg (0.13 mmol) of 4-*tert*-butyl-2,6-dimethylphenylamine in 0.75 ml of HSO_3F of –75°C in 1 h. Yield 15 mg (34%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.22 s (9H, CMe_3), 2.00 s (3H, Me), 2.22 s (3H, Me), 2.31 s (3H, Me), 3.60 br.s (2H, NH_2), 3.72 s (3H, OMe), 5.86 s (1H, =CH–), 7.05 d (2H_{arom}, *J* 8.3 Hz), 7.13 s (1H_{arom}), 7.20 d (2H_{arom}, *J* 8.3 Hz). Mass spectrum, *m/z* (I_{rel}, %): 351 (100) $[M]^+$, 336 (83), 304 (17), 294 (14), 276 (34), 262 (43). Found, %: C 78.65; H 8.27; N 4.01. $C_{23}H_{29}NO_2$. Calculated, %: C 78.59; H 8.32; N 3.99. *M* 351.22.

(*E*)- and (*Z*)-4-(3-Amino-6-*tert*-butyl-2,4-dimethylphenyl)-4-(4-methylphenyl)but-3-en-2-ones (IIn) were obtained from 27 mg (0.17 mmol) of compound **Id** and 30 mg (0.17 mmol) of 4-*tert*-butyl-2,6-dimethylphenylamine in 1 ml of HSO_3F at –30°C in 1 h as a crystalline isomers mixture of mp 148–151°C.

Compound *E*-**IIn**. Yield 1 mg (2%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.23 s (9H, CMe_3), 1.99 s (3H, Me), 2.21 s (3H, Me), 2.22 s (3H, Me), 2.32 s (3H, Me), 3.58 br.s (2H, NH_2), 6.05 s (1H, =CH–), 7.06 d (2H_{arom}, *J* 8.2 Hz), 7.13 s (1H_{arom}), 7.15 d (2H_{arom}, *J* 8.2 Hz).

Compound *Z*-**IIn**. Yield 8 mg (15%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.13 s (9H, CMe_3), 1.73 s (3H, Me), 1.90 s (3H, Me), 2.25 s (3H, Me), 2.33 s (3H, Me), 3.58 br.s (2H, NH_2), 6.88 s (1H, =CH–), 7.11 d (2H_{arom}, *J* 8.2 Hz), 7.17 C (1H_{arom}), 7.28 d (2H_{arom}, *J* 8.2 Hz). Mass spectrum (isomers mixture), *m/z* (I_{rel}, %): 335 (5) $[M]^+$, 320 (2), 278 (100). Found, %: C 82.45; H 8.73; N 4.20. $C_{23}H_{29}NO$. Calculated, %: C 82.34; H 8.71; N 4.18. *M* 335.22.

(*E*)-Methyl 3-(3-amino-2,5,6-trimethylphenyl)-3-phenylpropenoate (IIo) was obtained from 50 mg (0.31 mmol) of compound **Ia** and 50 mg (0.37 mmol) of 2,4,5-trimethylphenylamine in 1 ml of HSO_3F at –30°C in 1 h. Yield 38 mg (30%), mp 126–128°C. ^{1}H NMR spectrum, δ, ppm: 2.02 s (3H, Me), 2.03 s (3H, Me), 2.17 s (3H, Me), 3.48 br.s (2H, NH_2), 3.68 s (3H, OMe), 5.87 s (1H, =CH–), 6.53 s (1H_{arom}), 7.27 m (5H_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 295 (100) $[M]^+$, 264 (10), 248 (11), 236 (29), 220 (42), 134 (23). Found, %: C 77.35; H 7.21; N 4.69. $C_{19}H_{21}NO_2$. Calculated, %: C 77.26; H 7.17; N 4.74. *M* 295.16.

(*E*)-Methyl 3-(3-amino-2,5,6-trimethylphenyl)-3-(4-methylphenyl)propenoate (IIp) was obtained from 30 mg (0.17 mmol) of compound **Ib** and 29 mg (0.22 mmol) of 2,4,5-trimethylphenylamine in 1 ml of HSO_3F at –75°C in 1 h. Yield 6 mg (11%), mp 116–118°C. ^{1}H NMR spectrum, δ, ppm: 2.02 s (6H, 2Me), 2.17 s (3H, Me), 2.31 s (3H, Me), 3.50 br.s (2H, NH_2), 3.68 s (3H, OMe), 5.82 s (1H, =CH–), 6.53 s (1H_{arom}), 7.07 d (2H_{arom}, *J* 8.2 Hz), 7.17 d (2H_{arom}, *J* 8.2 Hz). Mass spectrum, *m/z* (I_{rel}, %): 309 (100) $[M]^+$, 294 (8), 278 (10), 262 (11), 250 (21), 234 (36). Found, %: C 77.55; H 7.40; N 4.56. $C_{20}H_{23}NO_2$. Calculated, %: C 77.64; H 7.49; N 4.53. *M* 309.17.

(*E*)-Methyl 3-(3-amino-2,5,6-trimethylphenyl)-3-(4-methoxyphenyl)propenoate (IIq) was obtained from 37 mg (0.19 mmol) of compound **Ic** and 34 mg (0.25 mmol) of 2,4,5-trimethylphenylamine in 1 ml of HSO_3F at –75°C in 1 h. Yield 11 mg (17%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.99 s (3H, Me), 2.00 s (3H, Me), 2.17 s (3H, Me), 3.48 br.s (2H, NH_2), 3.69 s (3H, OMe), 3.78 s (3H, OMe), 5.78 s (1H, =CH–), 6.53 s (1H_{arom}), 6.79 d (2H_{arom}, *J* 8.8 Hz), 7.24 d (2H_{arom}, *J* 8.8 Hz). Mass spectrum, *m/z* (I_{rel}, %): 325 (100) $[M]^+$, 250 (34), 237 (22), 134 (28). Found, %: C 73.75; H 7.15; N 4.36. $C_{20}H_{23}NO_3$. Calculated, %: C 73.82; H 7.12; N 4.30. *M* 325.17.

(*Z*)-Methyl 3-(3-amino-2,5,6-trimethylphenyl)-3-(4-methoxyphenyl)propenoate (IIq) was obtained from 37 mg (0.19 mmol) of compound **Ic** and 34 mg (0.25 mmol) of 2,4,5-trimethylphenylaminea in 1 ml of HSO_3F at –75°C in 1 h. Yield 5.5 mg (9%), mp 108–112°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.85 s (3H, Me), 1.86 s (3H, Me), 2.19 s (3H, Me), 3.48 br.s (2H, NH_2), 3.57 s (3H, OMe), 3.79 s (3H, OMe), 6.55 s (1H, =CH–), 6.56 s (1H_{arom}), 6.81 d (2H_{arom}, *J* 8.9 Hz), 7.29 d (2H_{arom}, *J* 8.9 Hz). Mass spectrum, *m/z* (I_{rel}, %): 325 (100) $[M]^+$, 250 (33), 237 (20), 134 (27). Found, %: C 73.86; H 7.11; N 4.33. $C_{20}H_{23}NO_3$. Calculated, %: C 73.82; H 7.12; N 4.30. *M* 325.17.

(*Z*)-4-(3-Amino-2,5,6-trimethylphenyl)-4-(4-methylphenyl)but-3-en-2-one (IIr) was obtained from 42 mg (0.27 mmol) of compound **Id** and 47 mg (0.35 mmol) of 2,4,5-trimethylphenylamine in 1 ml of HSO_3F at –30°C in 1.25 h. Yield 12.5 mg (16%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.71 s (3H, Me), 1.89 s (3H, Me), 1.90 s (3H, Me), 2.20 s (3H, Me), 2.33 s (3H, Me), 3.52 br.s (2H, NH_2), 6.60 s (1H_{arom}), 6.80 s (1H, =CH–), 7.11 d (2H_{arom}, *J* 7.9 Hz), 7.24 d (2H_{arom}, *J* 7.9 Hz). Mass spectrum, *m/z* (I_{rel}, %): 293 (100) $[M]^+$, 278 (81), 258 (81), 234 (33). Found, %: C 81.80; H 7.92; N 4.75. $C_{20}H_{23}NO$. Calculated, %: C 81.87; H 7.90; N 4.77. *M* 293.18.

(*E*)-Methyl 3-(2-amino-3,5,6-trimethylphenyl)-3-phenylpropenoate (IIs) was obtained from 50 mg (0.31 mmol) of compound **Ia** and 50 mg (0.37 mmol) of 2,4,5-trimethylphenylamine in 1 ml of HSO_3F at –30°C in 1 h. Yield 22 mg (18%), mp 98–100°C. ^{1}H NMR spectrum, δ, ppm: 2.04 s (3H, Me), 2.13 s (3H, Me), 2.15 s (3H, Me), 3.57 br.s (2H, NH_2), 3.68 s (3H, OMe), 6.03 s (1H, =CH–), 6.86 s (1H_{arom}), 7.29–7.30 m (3H_{arom}), 7.35–7.37 m (2H_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 295 (70) $[M]^+$, 264 (29), 248 (8), 236 (100), 221 (29). Found, %: C 77.30; H 7.28; N 4.74. $C_{19}H_{21}NO_2$. Calculated, %: C 77.26; H 7.17; N 4.74. *M* 295.16.

(*E*)-Methyl 3-(2-amino-3,5,6-trimethylphenyl)-3-(4-methylphenyl)propenoate (IIt) was obtained from 30 mg (0.17 mmol) of compound **Ib** and 29 mg (0.22 mmol) of 2,4,5-trimethylphenylamine in 1 ml of HSO_3F at –75°C in 1 h. Yield 10 mg (19%), mp 83–84°C. ^{1}H NMR spectrum, δ, ppm: 2.03 s (3H, Me), 2.11 s (3H, Me), 2.14 s (3H, Me), 2.32 s (3H, Me), 3.54 br.s (2H, NH_2), 3.68 s (3H, OMe), 5.97 s (1H, =CH–), 6.84 s (1H_{arom}), 7.09 d (2H_{arom}, *J* 8.3 Hz), 7.25 d (2H_{arom}, *J* 8.3 Hz). Mass spectrum, *m/z* (I_{rel}, %): 309 (63) $[M]^+$, 293 (3), 278 (30), 262 (10), 250 (100), 234 (21).

Found, %: C 77.61; H 7.38; N 4.55. $C_{20}H_{23}NO_2$. Calculated, %: C 77.64; H 7.49; N 4.53. *M* 309.17.

(*E*)-Methyl 3-(4-amino-2,3,5,6-tetramethylphenyl)-3-phenylpropenoate (IIu) was obtained and characterized before [7].

(*E*)-Methyl 3-(4-amino-2,3,5,6-tetramethylphenyl)-3-(4-methylphenyl)propenoate (IIv) was obtained from 40 mg (0.23 mmol) of compound **Ib** and 41 mg (0.28 mmol) 2,3,5,6-tetramethylphenylamine in 1 ml of HSO_3F at –75°C in 1 h. Yield 19 mg (25%), mp 197–199°C. ^{1}H NMR spectrum, δ, ppm: 2.09 s (6H, 2Me), 2.13 s (6H, 2Me), 2.31 s (3H, Me), 3.62 br.s (2H, NH_2), 3.67 s (3H, OMe), 5.82 s (1H, =CH–), 7.06 d ($2H_{arom}$, *J* 7.5 Hz), 7.15 d ($2H_{arom}$, *J* 7.5 Hz). Mass spectrum, *m/z* (I_{rel}, %): 323 (100) $[M]^+$, 308 (24), 292 (12), 250 (47), 235 (52), 161.5 (10) $[M]^{2+}$. Found, %: C 78.01; H 7.70; N 4.35. $C_{21}H_{25}NO_2$. Calculated, %: C 77.98; H 7.79; N 4.33. *M* 323.19.

(*Z*)-Methyl 3-(4-amino-2,3,5,6-tetramethylphenyl)-3-(4-methylphenyl)propenoate (IIv) was obtained by keeping a solution of 10 mg (0.03 mmol) of compound *E*-**IIv** in 0.8 ml of HSO_3F at –30°C for 1 h. Yield 5 mg (50%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.97 s (6H, 2Me), 2.10 s (6H, 2Me), 2.33 s (3H, Me), 3.58 s (5H, OMe, NH_2), 6.60 s (1H, =CH–), 7.09 d ($2H_{arom}$, *J* 8.3 Hz), 7.23 d ($2H_{arom}$, *J* 8.3 Hz). Found, %: C 77.88; H 7.85; N 4.38. $C_{21}H_{25}NO_2$. Calculated, %: C 77.98; H 7.79; N 4.33. *M* 323.19.

(*Z*)-4-(4-Amino-2,3,5,6-tetramethylphenyl)-4-(4-methylphenyl)but-3-en-2-one (IIw) was obtained from 50 mg (0.32 mmol) of compound **Id** and 89 mg (0.60 mmol) of 2,3,5,6-tetramethylphenylamine in 1 ml of HSO_3F at –30°C in 1 h. Yield 30 mg (31%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.65 s (3H, Me), 2.00 s (6H, 2Me), 2.12 s (6H, 2Me), 2.33 s (3H, Me), 3.70 br.s (2H, NH_2), 6.80 s (1H, =CH–), 7.10 d ($2H_{arom}$, *J* 8.2 Hz), 7.24 d ($2H_{arom}$, *J* 8.2 Hz). Mass spectrum, *m/z* (I_{rel}, %): 307 (100) $[M]^+$, 292 (98), 264 (33), 248 (27), 234 (22). Found, %: C 82.28; H 8.21; N 4.48. $C_{21}H_{25}NO$. Calculated, %: C 82.04; H 8.20; N 4.56. *M* 307.19.

(*E*)-Methyl 3-(3-amino-4-methoxyphenyl)-3-(4-methylphenyl)propenoate (IIx) was obtained from 28 mg (0.16 mmol) of compound **Ib** and 25 mg (0.20 mmol) 2-methoxyphenylamine in 1 ml of HSO_3F at –75°C in 1 h. Yield 21.5 mg (45%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 2.34 s (3H, Me), 3.62 s (3H, OMe), 3.77 br.s (2H, NH_2), 3.87 s (3H, OMe), 6.22 s (1H, =CH–), 6.54 d ($1H_{arom}$, *J* 2 Hz), 6.62 d.d ($1H_{arom}$, *J* 8.2, 2 Hz), 6.78 d ($1H_{arom}$, *J* 8.2 Hz), 7.11 d ($2H_{arom}$, *J* 8.1 Hz), 7.20 d ($2H_{arom}$, *J* 8.1 Hz). Mass spectrum, *m/z* (I_{rel}, %): 297 (100) $[M]^+$, 282 (29), 266 (12), 222 (16), 194 (11). Found, %: C 72.70; H 6.41; N 4.65. $C_{18}H_{19}NO_3$. Calculated, %: C 72.71; H 6.44; N 4.71. *M* 297.14.

(*E*)- and (*Z*)-4-(3-Amino-4-methoxyphenyl)-4-(4-methylphenyl)but-3-en-2-ones (IIy) were obtained from 30 mg (0.19 mmol) of compound **Id** and 30 mg (0.25 mmol) of 2-methoxyphenylamine in 1 ml of HSO_3F at –30°C in 1 h as an oily isomers mixture.

Compound *E*-**IIy**. Yield 2 mg (4%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.88 s (3H, Me), 2.35 s (3H, Me), 3.80 br.s (2H, NH_2), 3.89 s (3H, OMe), 6.42 s (1H, =CH–), 6.53 d ($1H_{arom}$, *J* 2 Hz), 6.59 d.d ($1H_{arom}$, *J* 8.2, 2 Hz), 6.78 d ($1H_{arom}$, *J* 8.2 Hz), 7.11 d ($2H_{arom}$, *J* 8.2 Hz), 7.20 d ($2H_{arom}$, *J* 8.2 Hz).

Compound *Z*-**IIy**. Yield 10.5 mg (20%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.82 s (3H, Me), 2.40 s (3H, Me), 3.80 br.s (2H, NH_2), 3.85 s (3H, OMe), 6.47 s (1H, =CH–), 6.65 br.s ($1H_{arom}$), 6.66 d.d ($1H_{arom}$, *J* 8.2, 2.2 Hz), 6.70 d ($1H_{arom}$, *J* 8.2 Hz), 7.08 d ($2H_{arom}$, *J* 7.9 Hz), 7.19 d ($2H_{arom}$, *J* 7.9 Hz). Mass spectrum (isomers mixture), *m/z* (I_{rel}, %): 281 (100) $[M]^+$, 266 (69), 150 (38). Found, %: C 76.89; H 6.71; N 5.03. $C_{18}H_{19}NO_2$. Calculated, %: C 76.84; H 6.81; N 4.98. *M* 281.14.

(*E*)-Methyl 3-(3-amino-2-methoxyphenyl)-3-(4-methylphenyl)propenoate (IIz) was obtained from 28 mg (0.16 mmol) of compound **Ib** and 25 mg (0.20 mmol) of 2-methoxyphenylamine on 1 ml of HSO_3F at –75°C in 1 h as an oily mixture with compound *Z*-**IIx**. Yield 8 mg (17%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 2.38 s (3H, Me), 3.59 s (3H, OMe), 3.76 br.s (2H, NH_2), 3.85 s (3H, OMe), 6.24 s (1H, =CH–), 6.64–6.71 m ($3H_{arom}$), 7.08 d ($2H_{arom}$, *J* 7.9 Hz), 7.17 d ($2H_{arom}$, *J* 7.9 Hz). Mass spectrum, *m/z* (I_{rel}, %): 297 (100) $[M]^+$, 282 (29), 266 (12), 222 (16), 194 (11). Found, %: C 72.70; H 6.41; N 4.65. $C_{18}H_{19}NO_3$. Calculated, %: C 72.71; H 6.44; N 4.71. *M* 297.14.

(*E*)-Methyl 3-(4-acetylaminophenyl)-3-phenylpropenoate (IIIa) was obtained from 50 mg (0.31 mmol) of compound **Ia** and 50 mg (0.37 mmol) of *N*-phenylacetamide in 1 ml of HSO_3F at –50°C in 1 h. Yield 4 mg (4%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 2.15 s (3H, Me), 3.63 s (3H, OMe), 6.31 s (1H, =CH–), 7.05–

7.55 m (10H, 9H_{arom}, NH). Mass spectrum, *m/z* (I_{rel}, %): 295 $[M]^+$. Found, %: C 73.35; H 5.82; N 4.71. $C_{18}H_{17}O_3$. Calculated, %: C 73.20; H 5.80; N 4.74. *M* 295.12.

(*E*)-Methyl 3-(4-acetylaminophenyl)-3-(4-methylphenyl)propenoate (IIIb) was obtained from 30 mg (0.17 mmol) of compound **Ib** and 22 mg (0.16 mmol) of *N*-phenylacetamide in 1 ml of HSO_3F at –75°C in 0.75 h. Yield 9 mg (18%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 2.15 s (3H, Me), 2.34 s (3H, Me), 3.61 s (3H, OMe), 6.30 s (1H, =CH–), 7.06–7.52 m (9H, 8H_{arom}, NH). Mass spectrum, *m/z* (I_{rel}, %): 309 (100) $[M]^+$, 278 (21), 267 (85), 254 (18), 236 (63), 209 (37). Found, %: C 73.70; H 6.15; N 4.56. $C_{19}H_{19}NO_3$. Calculated, %: C 73.77; H 6.19; N 4.53. *M* 309.14.

(*E*)- and (*Z*)-*N*-{4-[1-(4-Methylphenyl)-3-oxobut-1-enyl]phenyl}acetamides (IIIc) were obtained from 30 mg (0.19 mmol) of compound **Id** and 24 mg (0.18 mmol) *N*-phenylacetamide in 1 ml of HSO_3F at –30°C in 1 h as an oily isomers mixture. Yield *E*-**IIIc** 2 mg (3%), *Z*-**IIIc** 2 mg (3%). ^{1}H NMR spectrum, δ, ppm (isomers mixture): 1.86 s (3H, Me), 1.91 s (3H, Me), 2.17 s (3H, Me), 2.19 s (3H, Me), 2.35 s (3H, Me), 2.40 s (3H, Me), 6.50 s (1H, =CH–), 6.52 s (1H, =CH–), 7.05–7.30 m (18H, 16H_{arom}, 2NH). Mass spectrum (isomers mixture), *m/z* (I_{rel}, %): 293 (100) $[M]^+$, 292 (56), 278 (100), 250 (25), 236 (63), 208 (21), 43 (91). Found, %: C 77.70; H 6.52; N 4.68. $C_{19}H_{19}NO_2$. Calculated, %: C 77.79; H 6.53; N 4.77. *M* 293.14.

(*E*)-Methyl 3-(5-acetylamino-2-methylphenyl)-3-(4-methylphenyl)propenoate (IIId) was obtained from 30 mg (0.17 mmol) of compound **Ib** and 27 mg (0.18 mmol) of *N*-4-methylphenylacetamide in 1 ml of HSO_3F at –75°C in 0.75 h. Yield 16 mg (29%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.98 s (3H, Me), 2.11 s (3H, Me), 2.31 s (3H, Me), 3.65 s (3H, OMe), 5.93 s (1H, =CH–), 7.05–7.11 m (6H, 5H_{arom}, NH), 7.15 d (1H_{arom}, *J* 2.1 Hz), 7.52 d.d (1H_{arom}, *J* 8, 2.1 Hz). Mass spectrum, *m/z* (I_{rel}, %): 323 (100) $[M]^+$, 308 (8), 291 (64), 263 (23), 249 (80), 234 (60), 221 (60), 207 (49), 43 (64). Found, %: C 74.05; H 6.58; N 4.30. $C_{20}H_{21}NO_3$. Calculated, %: C 74.28; H 6.55; N 4.33. *M* 323.15.

***N*-{4-Methyl-3-[(*Z*)-1-(4-methylphenyl)-3-oxobut-1-enyl]phenyl}acetamide (IIIe)** was obtained from 37 mg (0.23 mmol) of compound **Id** and 45 mg (0.30 mmol) of *N*-4-methylphenylacetamide in 1 ml of HSO_3F at –30°C in 1 h. Yield 12 mg (17%), mp 168–170°C. ^{1}H NMR spectrum, δ, ppm: 1.85 s (3H, Me), 2.01 s (3H, Me), 2.12 s (3H, Me), 2.33 s (3H, Me), 6.70 s (1H, =CH–), 7.10 d (2H_{arom}, *J* 7.9 Hz), 7.14 d (1H_{arom}, *J* 2 Hz), 7.17–7.20 m (3H_{arom}), 7.31 br.s (1H, NH), 7.56 d.d (1H_{arom}, *J* 8.3, 2 Hz). Mass spectrum, *m/z* (I_{rel}, %): 307 (37) $[M]^+$, 292 (100), 264 (37), 250 (53), 222 (18), 130 (21), 43 (84). Found, %: C 78.38; H 6.80; N 4.56. $C_{20}H_{21}NO_2$. Calculated, %: C 78.15; H 6.89; N 4.56. *M* 307.16.

(*E*)- and (*Z*)-Methyl 3-(5-acetylamino-2,4-dimethylphenyl)-3-(4-methylphenyl)propenoates (IIIf) were obtained from 30 mg (0.17 mmol) of compound **Ib** and 27 mg (0.16 mmol) of *N*-2,4-dimethylphenylacetamide in 1 ml of HSO_3F at –75°C in 0.75 h as a crystalline isomers mixture of mp 190–193°C. Yield *E*-**IIIf** 7 mg (13%), *Z*-**IIIf** 7 mg (13%). ^{1}H NMR spectrum, δ, ppm (isomers mixture): 1.96 s (3H, Me), 1.98 s (3H, Me), 2.10 s (3H, Me), 2.14 s (3H, Me), 2.20 s (3H, Me), 2.23 s (3H, Me), 2.32 s (6H, 2Me), 3.58 s (3H, OMe), 3.63 s (3H, OMe), 5.95 s (1H, =CH–), 6.46 s (1H, =CH–), 6.95 s (1H_{arom}), 7.02 br.c (1H, NH), 7.04–7.14 m (8H, 7H_{arom}, NH), 7.22 d (2H_{arom}, *J* 8.3 Hz), 7.39 s (1H_{arom}), 7.46 s (1H_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 337 (73) $[M]^+$, 322 (5), 305 (45), 277 (15), 263 (100), 248 (35), 221 (29), 220 (30), 43 (31). Found, %: C 74.85; H 6.84; N 4.20. $C_{21}H_{23}NO_3$. Calculated, %: C 74.75; H 6.87; N 4.15. *M* 337.17.

(*E*)-Methyl-3 (3-acetylamino-2,4,6-trimethylphenyl)-3-(4-methylphenyl)propenoate (IIIg) was obtained from 30 mg (0.17 mmol) of compound **Ib** and 33 mg (0.19 mmol) of *N*-2,4,6-trimethylphenylacetamide in 1 ml of HSO_3F at –75°C in 0.75 h. Yield 31 mg (51%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 2.08 s (3H, Me), 2.13 s (3H, Me), 2.16 s (3H, Me), 2.19 s (3H, Me), 2.30 s (3H, Me), 3.67 s (3H, OMe), 5.82 s (1H, =CH–), 6.80 br.s (1H, NH), 6.94 s (1H-$_{arom}$), 7.05 d (2H_{arom}, *J* 8.1 Hz), 7.13 d (2H_{arom}, *J* 8.1 Hz). Mass spectrum, *m/z* (I_{rel}, %): 351 (100) $[M]^+$, 336 (11), 319 (82), 309 (23), 304 (52), 291 (68), 277 (77). Found, %: C 75.28; H 7.24; N 4.03. $C_{22}H_{25}NO_3$. Calculated, %: C 75.19; H 7.17; N 3.99. *M* 351.18.

(*Z*)-Methyl 3-(3-acetylamino-2,4,6-trimethylphenyl)-3-(4-methoxyphenyl)propenoate (IIIh) was obtained from 30 mg (0.16 mmol) of compound **Ic** and 36 mg (0.20 mmol) of *N*-2,4,6-trimethylphenylacetamide in 1 ml of HSO_3F at –75°C in 1 h. Yield 30 mg (52%), mp 185–187°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.95 s (3H, Me), 1.98 s (3H, Me), 2.18 s (3H, Me), 2.24 s (3H, Me), 3.57 s (3H, OMe), 3.78 s (3H, OMe), 6.54 s (1H, =CH–), 6.82 d (2H_{arom}, *J* 9 Hz), 6.82 s (1H_{arom}), 7.30 d (2H_{arom}, *J* 9 Hz). Mass spectrum, *m/z* (I_{rel}, %): 367 $[M]^+$ (96), 352 (18), 335 (100), 320 (54),

307 (61), 293 (78), 250 (57), 43 (83). Found, %: C 72.06; H 6.86; N 3.92. $C_{22}H_{25}NO_4$. Calculated, %: C 71.91; H 6.86; N 3.81. *M* 367.18.

***N*-{2,4,6-Trimethyl-3-[(*Z*)-1-(4-methylphenyl)-3-oxobut-1-enyl]phenyl}acetamide (IIIi)** was obtained from 32 mg (0.20 mmol) of compound **Id** and 47 mg (0.26 mmol) of *N*-2,4,6-trimethylphenylacetamide in 1 ml of HSO_3F at –30°C in 1.25 h. Yield 20 mg (29%). Oily substance. ^{1}H NMR spectrum, δ, ppm: 1.80 s (3H, Me), 1.97 s (3H, Me), 2.01 s (3H, Me), 2.18 s (3H, Me), 2.25 s (3H, Me), 2.33 s (3H, Me), 6.82 s (1H, =CH–), 6.87 s (1H_{arom}), 7.10 d (2H_{arom}, *J* 8.3 Hz), 7.23 d (2H-$_{arom}$, *J* 8.3 Hz). Mass spectrum, *m/z* (I_{rel}, %): 335 (40) $[M]^+$, 320 (47), 317 (23), 292 (100), 278 (34), 250 (47), 234 (32), 135 (82). Found, %: C 78.56; H 7.54; N 4.10. $C_{22}H_{25}NO_2$. Calculated, %: C 78.77; H 7.51; N 4.18. *M* 335.19.

(*E*)-Methyl 3-(6-*tert*-butyl-3-acetylamino-2,4-dimethylphenyl)-3-(4-methylphenyl)propenoate (IIIj) was obtained from 25 mg (0.14 mmol) of compound **Ib** and 29 mg (0.13 mmol) of *N*-4-*tert.*-butyl-2,6-dimethylphenylacetamide in 1 ml of HSO_3F at –75°C in 0.75 h. Yield 35 mg (67%), mp 150°C (decomp.). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.24 s (9H, CMe_3), 2.03 s (3H, Me), 2.18 s (3H, Me), 2.25 s (3H, Me), 2.30 s (3H, Me), 3.71 s (3H, OMe), 5.84 s (1H, =CH–), 6.74 br.s (1H, NH), 7.04 d (2H_{arom}, *J* 8.3 Hz), 7.17 d (2H_{arom}, *J* 8.3 Hz), 7.28 s (1H-$_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 393 (25) $[M]^+$, 336 (100), 333 (17), 304 (20), 219 (22), 43 (77). Found, %: C 76.47; H 7.90; N 3.56. $C_{25}H_{31}NO_3$. Calculated, %: C 76.30; H 7.94; N 3.56. *M* 393.23.

(*E*)-Methyl 3-(3-acetylamino-2,5,6-trimethylphenyl)-3-(4-methylphenyl)propenoate (IIIk) and **(*E*)-methyl 3-(2-acetylamino-3,5,6-trimethylphenyl)-3-(4-methylphenyl)propenoate (IIIl)** were obtained from 30 mg (0.17 mmol) of compound **Ib** and 31 mg (0.17 mmol) of N-2,4,5-trimethylphenylacetamide in 1 ml of HSO_3F at –75°C in 0.75 h as a crystalline isomers mixture of mp 148–152°C.

Compound *E*-**IIIk**. Yield 12 mg (20%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 2.13 s (3H, Me), 2.15 s (6H, 2Me), 2.24 s (3H, Me), 2.32 s (3H, Me), 3.69 s (3H, OMe), 5.80 s (1H, =CH-), 6.96 br.s (1H, NH), 7.04 s (1H-$_{arom}$), 7.05–7.10 m (2H_{arom}), 7.13 d (2H_{arom}, *J* 8.1 Hz).

Compound *E*-**IIIl**. Yield 10 mg (16%). ^{1}H NMR spectrum, δ, ppm (extracted from the spectrum of isomers mixture): 1.94 s (3H, Me), 2.06 s (3H, Me), 2.09 s (3H, Me), 2.23 s (3H, Me), 2.31 s (3H, Me), 3.67 s (3H, OMe), 5.86 s (1H, =CH–), 6.56 br.s (1H, NH), 7.05–7.10 m (3H_{arom}), 7.13 d (2H_{arom}, *J* 8.1 Hz). Mass spectrum (isomers mixture), *m/z* (I_{rel}, %): 351 (46) $[M]^+$, 308 (100), 276 (36), 249 (48), 234 (24), 43 (20). Found, %: C 75.36; H 7.23; N 3.95. $C_{22}H_{25}NO_3$. Calculated, %: C 75.19; H 7.17; N 3.99. *M* 351.18.

(*E*)-Methyl 3-(4-acetylamino-2,3,5,6-tetramethylphenyl)-3-(4-methylphenyl)propenoate (IIIm) was obtained from 30 mg (0.17 mmol) of compound **Ib** and 31 mg (0.16 mmol) of *N*-2,3,5,6-tetramethylphenylacetamide in 1 ml of HSO_3F at –75°C in 0.75 h. Yield 19 mg (32%), mp 180–185°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 2.13 s (12H, 4Me), 2.23 s (3H, Me), 2.33 s (3H, Me), 3.68 s (3H, OMe), 5.81 s (1H, =CH–), 6.69 br.s (1H, NH), 7.07 d (2H_{arom}, *J* 8 Hz), 7.16 d (2H_{arom}, *J* 8 Hz). Mass spectrum, *m/z* (I_{rel}, %): 365 (100) $[M]^+$, 350 (34), 334 (13), 322 (30), 290 (21), 248 (25). Found, %: C 75.58; H 7.60; N 3.79. $C_{23}H_{27}NO_3$. Calculated, %: C 75.59; H 7.45; N 3.83. *M* 365.20.

The generation and registering at –80°C of ^{1}H NMR spectra in HSO_3F of ions **IV** and **VII** obtained from compounds *E*-**IIp** and **VI** respectively was performed as in [6]. The preparation and characteristics of (*Z*)-4-(pentamethylphenyl)-4-phenylbut-3-en-2-one (**VI**) were described before [8].

3-(3-Ammonio-2,5,6-trimethylphenyl)-1-hydroxy-3-(4-methylphenyl)-1-methoxyprop-2-en-1-ylium IV). ^{1}H NMR spectrum (HSO_3F), δ, ppm: 2.20 s (6H, 2Me), 2.38 s (3H, Me), 2.81 s (3H, Me), 4.41 s (3H, OMe), 6.41 s (1H, =CH–), 7.34 s (1H_{arom}), 7.39 s (3H, NH_3^+), 7.62 s (1H_{arom}), 7.69 s (1H_{arom}), 8.22 s (1H_{arom}), 8.30 s (1H_{arom}), 12.49 s (1H, C^+–OH).

2-Hydroxy-4-pentamethylphenyl-4-phenylbut-3-en-2-ylium (VII). ^{1}H NMR spectrum (HSO_3F), δ, ppm: 2.16 s (6H, 2Me), 2.49 s (6H, 2Me), 2.26 s (3H, Me), 3.32 s (3H, Me), 7.66 d (2H^o, *J* 7 Hz), 7.72 t (2H^m, *J* 7 Hz), 8.01 t (1H^n, *J* 7 Hz), 8.33 s (1H, =CH–).

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