Arenesulfonylimines of methyl trifluoropyruvate in the cyclocondensation reactions with 1,3-C,N- and -N,N-binucleophiles

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Reaction of arenesulfonylimines of methyl trifluoropyruvate with 1,3-C,N- and -N,N-binucleophiles led to a variety of *N*-sulfonylated fluorine-containing heterocycles, including the fused ones.

Key words: arenesulfonamides of methyl trifluoropyruvate, sulfonylimines, nitrile of 2-aminocrotonic acid, 6-aminouracils, *N*-substituted 3-aminocyclohexenones, benzamidine, 2-aminothiazoline, *N*-substituted ureas, 2,3-dihydro-1*H*-pyrroles, hexahydro-1*H*-pyrrolo[2,3-*d*]pyrimidines, hexahydro-1*H*-indoles, 4,5-dihydro-1*H*-imidazoles, tetrahydroimidazo[2,1-*b*]thiazoles, imidazolidines, cyclocondensation.

Sulfanylamide drugs, despite of the discovery of penicillin and other antibiotics, did not lose their value and in number of cases are successfully used for the treatment of infectious diseases. The nowadays used sulfanylamide drugs significantly differ in their pharmacological parameters (absorption, accumulation in blood and organs in bacteriostatic concentrations, surmounting of the hematoencephalitic barrier, time of the removal from organism, *etc*.), which is caused to a considerable extent by the nature of the substituents at the nitrogen atom.[1] The variation of substituents at the nitrogen atom has been and, apparently, remains the main approach to the chemical design of sulfanylamides, including ones with fluorine-containing substituents.[2,3] Below, we consider from this point of view a developed by us approach to the introduction of various heterocycles with fluorine-containing substituents at the nitrogen atom of arenesulfanylamide molecules.

A systematic study of behavior of acylimines of methyl trifluoropyruvate (MTFP) in the cyclocondensation reaction with 1,3-binucleophiles, which enabled us to propose an original approach to the synthesis of five- and six-membered heterocycles with fluorine-containing substituents, served as the prerequisite to this research.[4—9] In the present work, new data on the synthetic potentialities of arenesulfonylimines of MTFP **1** in the preparation of fluorine-containing *N*-arenesulfonylated heterocycles by the cyclocondensation of compounds **1** with 1,3-C,N- and -N,N-binucleophiles are presented. It should be noted that earlier, sulfonylimines of polyfluoroketones were used in the synthesis of heterocycles with fluorine-containing substituents (the aza-Diels—Alder reaction),[10] as well as of biologically active fluorine-containing aminophosphonates, the serine hydrolase inhibitors.[11]

In contrast to acylimines of MTFP, showing properties of 1,2- and 1,3-bielectrophiles in the cyclocondensation reactions, arenesulfonylimines of MTFP **1** exclusively play the role of 1,2-bielectrophiles in the reactions with 1,3-binucleophiles. These transformations, similarly to the reactions studied by us earlier,[5,7—9] proceed according to the two-step scheme: addition of the binucleophile at the C=N bond of arenesulfonylimine and subsequent cyclization with elimination of MeOH.

In the cyclocondensation with compounds **1a—d**, nitrile of 2-aminocrotonic acid (**2**), 6-aminouracils **3a**,**b**, and *N*-substituted 3-aminocyclohexenones **4a—d** were studied as the C,N-binucleophiles, whereas benzamidine **5**, 2-aminothiazoline **6**, and *N*-substituted ureas **7a—d**, as the N,N-binucleophiles (Scheme 1). The reaction was carried out by the heating of a mixture of reagents in DMF for 1 h at 90—100 °C and, in case of nucleophiles **3**, **4**, and **7**, in the presence of catalytic amount of Et_3N. All the transformations considered above led to the formation of the corresponding *N*-arenesulfonylated heterocycles with fluorine-containing substituents: 2,3-dihydro-1*H*-pyrroles **8a—c**, 2,3,4,5,6,7-hexahydro-1*H*-pyrrolo[2,3-*d*]pyrimidines **9a**,**b**, 2,3,4,5,6,7-hexahydro-1*H*-indoles **10a—d**, 4,5-dihydro-1*H*-imidazoles **11a**,**b**, 2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazoles **12a**,**b**, and imidazolidines **13a—e**.

Amidosulfonates **8a—c**, **9a**,**b**, **10a—d**, **11a**,**b**, **12a**,**b**, and **13a—e**, obtained in 65—81% yield, are solid crystalline substances. Their composition and structures were

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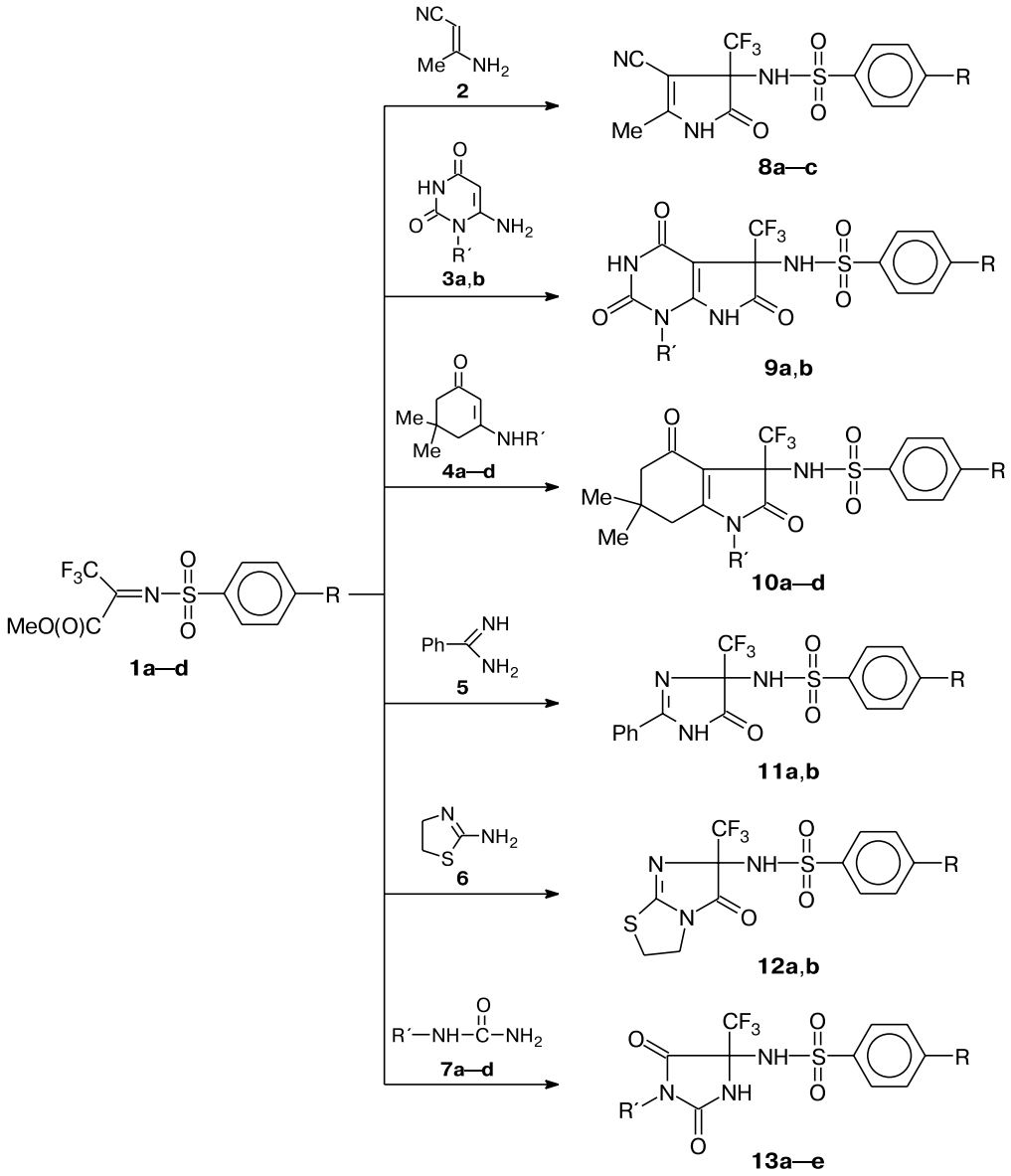
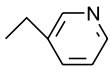
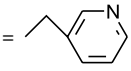
Scheme 1



1, **8**: R = H (**a**), MeO (**b**), Cl (**c**), Me (**d**)
3, **9**: R´ = CH_2Ph (**a**), 4-ClC_6H_4 (**b**)

4: R´ = 3,4-$Me_2C_6H_3$ (**a**), CH_2Ph (**b**),  (**c**), CH_2CH_2Ph (**d**)

7: R´ = CH_2Ph (**a**), 4-ClC_6H_4 (**b**), Ph (**c**), 4-FC_6H_4 (**d**)

10: R = H, R´ = 3,4-$Me_2C_6H_3$ (**a**); R = Me, R´ = CH_2Ph (**b**); R = Me, R´ =  (**c**); R = Cl, R´ = CH_2CH_2Ph (**d**)

11: R = H (**a**), Cl (**b**)
12: R = H (**a**), MeO (**b**)
13: R = H, R´ = CH_2Ph (**a**); R = Me, R´ = CH_2Ph (**b**); R = Me, R´ = 4-ClC_6H_4 (**c**); R = Cl, R´ = Ph (**d**); R = H, R´ = 4-FC_6H_4 (**e**)

confirmed by elemental analysis and NMR spectroscopy data. In the ^{19}F NMR spectra, signals of the trifluoromethyl groups in the region δ −1—5 is characteristic of them.

It should be noted that in the transformations under consideration (taking into account the high electrophilicity of the C=N bond of the arenesulfonylimines of MTFP, which exothermically react with C,H- and N,H-nucleo-

philes),[12,13] the condensation is the determining step, rather than the *C*-alkylation, and these processes are under the decisive influence of the nucleophilic properties of the addition products of arenesulfonylimines of MTFP to 1,3-binucleophiles. Thus, the cyclocondensation with the least nucleophilic binucleophiles (**3**, **4**, and **7**) was successful only in the presence of catalytic amount of Et_3N.

In conclusion, starting from the fairly available arenesulfonylimines of MTFP, we synthesized a variety of the earlier unknown *N*-arenesulfonylated fluorine-containing 2,3-dihydro-1*H*-pyrroles, imidazoles, and imidazolidines, including the fused ones, which can be of interest as the potentially biologically active substances.

Experimental

^1H and ^{19}F NMR spectra were recorded on a Bruker DXP 200 spectrometer (200.13 and 188.29 MHz) relatively to Me_4Si and CF_3COOH as the internal and external standards, respectively. Melting points were determined in a glass capillary tube on a PTP (M) instrument and were uncorrected. The starting arenesulfonylimines of MTFP **1a—d** were synthesized according to the procedure described earlier,[14] 6-aminouracils **3a**,**b**, according to the known procedure,[15] *N*-substituted 3-aminocyclohexenones **4a—d**, according to the published procedure.[16] Nitrile of 2-aminocrotonic acid **2**, benzamidine **5**, 2-aminothiazoline **6**, and *N*-substituted ureas **7a—d** (all from Aldrich) were used without additional purification.

Table 1. The yields, melting points, and elemental analysis data of compounds **8a—c**, **9a**,**b**, **10a—d**, **11a**,**b**, **12a**,**b,** and **13a—e**

Compound	Yield (%)	M.p./°C	Found / Calculated (%)			Molecular formula
			C	H	N	
8a	81	133—135	45.07 45.22	2.79 2.92	12.03 12.17	$C_{13}H_{10}F_3N_3O_3S$
8b	76	145—147	44.93 44.80	3.33 3.22	11.36 11.20	$C_{14}H_{12}F_3N_3O_4S$
8c	65	151—153	41.34 41.12	2.23 2.39	11.19 11.07	$C_{13}H_9ClF_3N_3O_3S$
9a	75	168—170	50.12 50.00	3.02 3.15	11.49 11.66	$C_{20}H_{15}F_3N_4O_5S$
9b	77	134—136	45.69 45.57	2.55 2.42	11.32 11.19	$C_{19}H_{12}ClF_3N_4O_5S$
10a	73	137—139	59.41 59.28	4.85 4.97	5.66 5.53	$C_{25}H_{25}F_3N_2O_4S$
10b	72	141—143	59.41 59.28	4.83 4.97	5.38 5.53	$C_{25}H_{25}F_3N_2O_4S$
10c	69	139—141	56.93 56.80	4.88 4.77	8.14 8.28	$C_{24}H_{24}F_3N_3O_4S$
10d	73	166—168	55.63 55.51	4.59 4.47	5.02 5.18	$C_{25}H_{24}ClF_3N_2O_4S$
11a	68	217—219	50.30 50.13	3.02 3.16	10.81 10.96	$C_{16}H_{12}F_3N_3O_3S$
11b	76	176—178	40.12 40.00	2.78 2.65	10.18 10.06	$C_{16}H_{11}ClF_3N_3O_3S$
12a	70	185—187	39.31 39.45	2.62 2.76	11.34 11.50	$C_{12}H_{10}F_3N_3O_3S_2$
12b	67	165—167	39.38 39.49	3.21 3.06	10.48 10.63	$C_{13}H_{12}F_3N_3O_4S_2$
13a	66	169—171	49.52 49.40	3.29 3.41	10.03 10.17	$C_{17}H_{14}F_3N_3O_4S$
13b	73	133—135	50.44 50.58	3.89 3.77	9.71 9.83	$C_{18}H_{16}F_3N_3O_3S$
13c	74	148—150	45.48 45.60	2.81 2.93	9.52 9.38	$C_{17}H_{13}ClF_3N_3O_4S$
13d	68	156—158	44.19 44.30	2.71 2.56	9.55 9.56	$C_{16}H_{11}ClF_3N_3O_4S$
13e	67	159—161	46.17 46.05	2.78 2.66	10.21 10.07	$C_{16}H_{11}F_4N_3O_4S$

***N*-(4-Cyano-5-methyl-2-oxo-3-trifluoromethyl-2,3-dihydro-1*H*-pyrrol-3-yl)benzenesulfonamide (8a), *N*-(4-cyano-5-methyl-2-oxo-3-trifluoromethyl-2,3-dihydro-1*H*-pyrrol-3-yl)-4-methoxybenzenesulfonamide (8b), *N*-(4-cyano-5-methyl-2-oxo-3-trifluoromethyl-2,3-dihydro-1*H*-pyrrol-3-yl)-4-chlorobenzenesulfonamide (8c), *N*-(5-oxo-2-phenyl-4-trifluoromethyl-4,5-dihydro-1*H*-imidazol-4-yl)benzenesulfonamide (11a), *N*-(5-oxo-2-phenyl-4-trifluoromethyl-4,5-dihydro-1*H*-imidazol-4-yl)-4-chlorobenzenesulfonamide (11b), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)benzenesulfonamide (12a),** and ***N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-methoxybenzenesulfonamide (12b) (general procedure).** A solution of the corresponding arenesulfonylimine of MTFP **1** (0.01 mol) and the corresponding binucleophile **2**, **5**, or **6** (0.01 mol) in DMF (10 mL) was stirred for 1 h at 20 °C and heated for 2 h at 90—100 °C. Then, the reaction mixture was cooled, poured in water (50 mL), the precipitate formed was filtered off and recrystallized from 50% aq. EtOH. The yields, melting points, and spectral characteristics of compounds **8a—c**, **11a**,**b**, and **12a**,**b** are given in Tables 1 and 2.

***N*-(1-Benzyl-5-trifluoromethyl-2,4,6-trioxo-2,3,4,5,6,7-hexahydro-1*H*-pyrrolo[2,3-*d*]pyrimidin-5-yl)benzenesulfonamide (9a), *N*-{1-(4-chlorophenyl)-5-trifluoromethyl-2,4,6-trioxo-2,3,4,5,6,7-hexahydro-1*H*-pyrrolo[2,3-*d*]pyrimidin-5-yl}benzenesulfonamide (9b), *N*-[6,6-dimethyl-1-(3,4-dimethylphenyl)-2,4-dioxo-3-trifluoromethyl-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl]benzenesulfonamide (10a), *N*-(1-benzyl-6,6-dimethyl-2,4-dioxo-3-trifluoromethyl-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl)-4-methylbenzenesulfonamide (10b), *N*-[6,6-dimethyl-2,4-dioxo-1-(pyridin-3-yl)methyl-3-trifluoromethyl-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl]-4-methylbenzenesulfonamide (10c), *N*-[6,6-dimethyl-2,4-dioxo-1-(2-phenylethyl)-3-trifluoromethyl-2,3,4,5,6,7-hexahydro-1*H*-indol-3-yl]-4-chlorobenzenesulfon-**

Table 2. ^{1}H and ^{19}F NMR spectra of compounds **8a—c**, **9a**,**b**, **10a—d**, **11a**,**b**, **12a**,**b**, and **13a—e** in DMSO-d_6

Compound	δ_H (J/Hz)	δ_F
8a	2.19 (s, 3 H, Me); 7.57 (m, 3 H, CH_{Ar}); 7.84 (m, 2 H, CH_{Ar}); 7.58 (s, 1 H, NH); 11.21 (s, 1 H, NH)	2.71 (s)
8b	2.96 (s, 3 H, Me); 4.66 (s, 3 H, MeO); 7.78 (d, 2 H, CH_{Ar}, J = 9.8); 8.52 (d, 2 H, CH_{Ar}, J = 9.8); 10.15, 11.99 (both s, 1 H each, NH)	2.31 (s)
8c	2.28 (s, 3 H, Me); 7.58, 7.81 (both d, 2 H each, CH_{Ar}, J = 8.5); 9.70, 11.29 (both s, 1 H each, NH)	2.69 (s)
9a	5.17 (m, 2 H, AB system, J = 19); 7.36 (m, 7 H, CH_{Ar}); 7.51 (t, 1 H, J = 7.2); 7.64 (d, 2 H, J = 7.2); 9.40, 10.71, 12.28 (all s, 1 H each, NH)	4.45 (s)
9b	7.40 (m, 2 H, CH_{Ar}); 7.51 (m, 3 H, CH_{Ar}); 7.59 (m, 2 H, CH_{Ar}); 7.81 (d, 2 H, CH_{Ar}, J = 8.3); 9.43, 10.82, 11.51 (all s, 1 H each, NH)	4.33 (s)
10a	1.02, 1.08 (both s, 3 H each, Me); 2.15 (AB system, 2 H, CH_2, J = 10.6); 2.23, 2.38 (both s, 3 H each, Me); 2.46 (s, 2 H, CH_2); 7.18—7.36 (m, 5 H, CH_{Ar}); 7.73 (m, 3 H, CH_{Ar}); 9.83 (s, 1 H, NH)	4.05 (s)
10b	1.05, 1.11 (both s, 3 H each, Me); 2.11 (AB system, 2 H, CH_2, J = 10.9); 2.36 (s, 3 H, Me); 2.49 (s, 2 H, CH_2); 4.88 (s, 2 H, CH_2N); 7.36 (m, 7 H, CH_{Ar}); 7.95 (d, 2 H, CH_{Ar}, J = 8.1); 9.74 (s, 1 H, NH)	4.62 (s)
10c	0.98, 1.06 (both s, 3 H each, Me); 2.15 (AB system, 2 H, CH_2, J = 10.6); 2.33 (s, 3 H, Me); 2.46 (s, 2 H, CH_2); 4.88 (s, 2 H, CH_2N); 7.36 (m, 6 H, CH_{Ar}); 7.95 (d, 2 H, CH_{Ar}, J = 8.1); 9.74 (s, 1 H, NH)	4.51 (s)
10d	0.96, 1.02 (both s, 3 H each, Me); 2.12 (s, 2 H, CH_2); 2.16 (AB system, 2 H, CH_2, J = 17.4); 2.94 (m, 2 H, CH_2); 3.84 (m, 2 H, CH_2N); 7.22—7.38 (m, 5 H, CH_{Ar}); 7.46 (m, 2 H, CH_{Ar}); 7.94 (d, 2 H, CH_{Ar}, J = 8.4); 9.62 (s, 1 H, NH)	4.33 (s)
11a	7.46 (m, 6 H, CH_{Ar}); 7.82 (m, 4 H, CH_{Ar}); 9.72, 12.20 (both br.s, 1 H each, NH)	0.40 (s)
11b	7.44 (d, 2 H, CH_{Ar}, J = 8.3); 7.49 (m, 2 H, CH_{Ar}); 7.58 (m, 1 H, CH_{Ar}); 7.73, 7.84 (both d, 2 H each, CH_{Ar}, J = 8.2); 9.80, 12.19 (both s, 1 H each, NH)	0.24 (s)
12a	3.81 (m, 3 H, CH_2N + CH_2S); 3.99 (m, 1 H, CH_2S); 7.53 (m, 3 H, CH_{Ar}); 7.81 (m, 2 H, CH_{Ar}); 9.88 (s, 1 H, NH)	0.44 (s)
12b	3.80 (m, 3 H, CH_2N + CH_2S); 3.87 (s, 3 H, MeO); 3.97 (m, 1 H, CH_2S); 6.99, 7.81 (both d, 2 H each, CH_{Ar}, J = 8.1); 9.77 (s, 1 H, NH)	0.31 (s)
13a	4.58 (m, 2 H, AB system, J = 18.2); 7.27 (m, 5 H, CH_{Ar}); 7.53 (m, 3 H, CH_{Ar}); 7.77 (d, 2 H, CH_{Ar}, J = 7.9); 9.78, 9.84 (both s, 1 H each, NH)	−0.48 (s)
13b	2.47 (s, 3 H, Me); 4.59 (m, AB system, 2 H, J = 16.8); 7.31 (m, 7 H, CH_{Ar}); 7.65 (d, 2 H, CH_{Ar}, J = 9.2); 9.70 (br.s, 2 H, NH)	−0.38 (s)
13c	2.47 (s, 3 H, Me); 7.34—7.55 (m, 6 H, CH_{Ar}); 7.77 (d, 2 H, CH_{Ar}, J = 8.6); 9.90, 10.09 (both s, 1 H each, NH)	0.07 (s)
13d	7.31 (t, 2 H, CH_{Ar}, J = 7.9); 7.44 (m, 5 H, CH_{Ar}); 7.82 (d, 2 H, CH_{Ar}, J = 8.4); 9.82, 10.07 (both s, 1 H each, NH)	0.10 (s)
13e	7.22 (t, 2 H, CH_{Ar}, J = 8.2); 7.34 (m, 2 H, CH_{Ar}); 7.58 (m, 3 H, CH_{Ar}); 7.87 (d, 2 H, CH_{Ar}, J = 8.2); 9.92, 9.97 (both s, 1 H each, NH)	0.02 (s, 3 F, CF_3); −31.52 (m, 1 F, CF_{Ar})

amide (10d), *N*-[1-benzyl-2,3-dioxo-4-(trifluoromethyl)imidazolidin-4-yl]benzenesulfonamide (13a), *N*-[1-benzyl-2,3-dioxo-4-(trifluoromethyl)imidazolidin-4-yl]-4-methylbenzenesulfonamide (13b), *N*-[1-(4-chlorophenyl)-2,3-dioxo-4-(trifluoromethyl)imidazolidin-4-yl]-4-methylbenzenesulfonamide (13c), *N*-[2,5-dioxo-1-phenyl-4-(trifluoromethyl)imidazolidin-4-yl]-4-chlorobenzenesulfonamide (13d), and ***N*-[2,5-dioxo-1-(4-fluorophenyl)-4-(trifluoromethyl)imidazolidin-4-yl]benzenesulfonamide (13e) (general procedure).** A solution of the corresponding arenesulfonylimine of MTFP **1** (0.01 mol) and the corresponding binucleophile **3a**,**b**, **4a**—**d**, or **7a**—**d** (0.01 mol) in DMF (10 mL) was stirred for 1 h at 20 °C, after that, Et_3N (0.2 mL) was added, and this was heated for 2 h at 90—100 °C. The reaction mixture was cooled, poured in water (50 mL), the precipitate formed was filtered off and recrystallized from 50% aq. EtOH. The yields, melting points, and spectral characteristics of compounds **9a**,**b**, **10a**—**d**, and **13a**—**e** are given in Tables 1 and 2.

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