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THE SYNTHESIS AND CHARACTERIZATION OF SOME NEW DIAZOAMINO DERIVATIVES

The sulfonamidic moiety is much encountered in structures of bioactive compounds. In the present paper the studies on the sulfonamidated aryloxyalkylcarboxylic acids are extended by their attaching on certain substrata able to confer some special biological properties to the final products, such as anti-tumor and antioxidant actions useful in treating inflammatory processes, ulcer, convulsions and diabetes, as well as a herbicidal action. The stepwise syntheses of the sulfonamidated aryloxyalkylcarboxylic acid derivatives and their characterization by elemental analysis data and IR, ^{1}H-NMR and UV-Vis spectral measurements are described. The newly obtained compounds could show potential pharmaceutical and herbicide properties.

Key words: hydrazide; coupling components; diazoamino derivatives; elemental analysis; IR; ^{1}H-NMR; UV-Vis; spectral measurements.

The sulphonamides belong to an important class of therapeutical agents applied in the modern medical science. The sulfonamidic moiety is much used for designing new biologically active compounds [1-3]. Modifications of the sulfonamide structure afforded new compounds showing improved biological properties, such as hypoglicemic, diuretic, antihypertensive activities acting also as carbonic anhydrase inhibitors. The *in vivo* and *in vitro* antitumoural activities found recently with these new compounds are particularly interesting. Although their structures have an aromatic/heterocyclic residue and a sulphonamidic group in common, their action mechanisms and pharmacological effects are different [4-6].

A particular attention is paid to the derivatives acting in microbial/viral infections, to those foavouring the immunity system and also to the sulphonamides with immunomodulating effect [7-9], blocking effects in viral infections [10], inhibiting effects on the non-peptidic proteases and HIV proteases being applied as anti-HIV drugs [11-14]. Many sulfonamidic derivatives can be used as drugs with the anti-cancer anti-inflammatory double action. For these reasons, the researches are directed to finding new sulfonamides of low toxicity and antidepressive and anticonvulsive properties and new alkyl-aryl-sulfonamides, sulphonylureides, thiosulphonylureides [15], indazole sulfonamides [16], and imido-benzene-sulphonyl-azirides [17,18].

The derivatives of the aryloxyalkylcarboxylic acids have a particularly high biological potential. In recent years, their hydrazides and the corresponding alkylhydrazones as condensation products draw the attention in the field of synthetic chemistry and inorganic chemistry due to their various potential biological actions as pesticides (*e.g.* herbicides, plant growth stimulators) [19-28] or drugs with a pharmacological action [29-35]. By taking this fact into account we obtained some diazoaminoderivatives, starting from hydrazides and several coupling components.

EXPERIMENTAL

General procedure

The diazotization was carried out by treating hydrazide with aqueous hydrogen chloride, in a 1:4.5 or 1:5 HCl/amine ratio. The suspension was cooled at 0-5 °C, and the required amount of 10% $NaNO_2$ solution then added stepwise, under stirring. The mixture was stirred for another 15 min at the same temperature. Diazotizations are usually carried out at low temperatures (0-5 °C), sometimes below 0 °C, in order to avoid the degradation of diazonium salts although the reaction rate increases a lot with increasing the temperature.

The coupling was made in the following variants:

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- The addition of the diazonium salt solution on the coupling component solved in ethanol followed by the diazoderivative precipitation with sodium acetate.

- The buffering of the diazonium salt solution with sodium acetate followed by treating with the coupling component as an aqueous solution or NaOH solution.

- The addition of the diazonium salt solution on the alcalinized coupling component followed by pH adjustment by Na_2CO_3.

Diazoaminoderivatives were purified from organic solvents such as ethanol, toluene, o-xylene or two combined solvents (DMF-ethylic ether, ethylacetate--ethylic ether, toluene-petroleum ether). The purification by column chromatography with aluminium oxide was simultaneously applied. CH_2Cl_2-isopropanol (9:1(v/v)) or ethyl acetate-hexane mixtures were used.

Diazoamino derivatives obtained from 2-methyl-4--sulfonamido-phenoxyacetyl hidrazide

Diazoaminoderivative synthesized with N,N-dimethylaniline. The hydrazide (0.65 g; 2.5 mmol) was treated with conc. HCl (0.36 g; 0.31 ml; 10 mmol) and then diluted with water (10 ml). The hydrochloride suspension was cooled to 0-5 °C and then submitted to diazotization under stirring by a stepwise addition of $NaNO_2$ (0.17 g; 2.5 mmol) dissolved in water (2-2.5 ml) keeping the temperature below 3 °C. The reaction mixture was then stirred for another 10 min. Finally, the acidity was verified with Congo red test paper and the nitrite excess with starch-iodide paper.

Coupling: *N,N*-dimethylaniline 0.30 g (2.2 mmol) was dissolved in ethanol (10 ml) and the previously prepared diazonium sodium salt added under stirring. The reaction was completed by stirring for one hour at low temperature. At the same time sodium acetate (3 g) was added in order to have a weak acid pH value. The resulting precipitate was filtered off and washed with water on the filter paper. The crude product was purified by recrystallization in ethanol. Higher purification was made by column chromatography on a column containing alluminium oxide and using a CH_2Cl_2-isopropanol mixture (9:1, vol.), η = 70%.

Diazoaminoderivative synthesized with resorcine. Hydrazide (0.65 g; 2.5 mmol) was treated with conc. HCl (0.36 g; 0.31 mL; 10 mmol) and then diluted with water (10 ml). After cooling the reaction mixture to 0-5 °C with ice a sodium nitrite (0.17 g; 2.5 mmol) solution in 2 mL water was added drop-wise on the resulting amine hydrochloride. The acidity was finally verified (pH 2-3).

Coupling: The resorcine (0.28 g; 2.5 mmol) dissolved in water (5 ml) was added to the diazonium salt previously obtained and the mixture heated for one hour. The suspension of the diazoamino derivative was filtered off and the solid product thoroughly washed with water, η = 73%.

Diazoaminoderivative synthesized with Cu β--naphtol. The β-naphtol (0.36 g; 2.5 mmol) was dissolved in the solution prepared with NaOH (0.2 g; 5 mmol) in 3.5 mL water. After cooling at about 5 °C the diazonium salt solution was added under stirring. The pH value was verified during coupling and maintained at 8-8.5 by adding Na_2CO_3 (0.5 g), η = 75%.

Diazoaminoderivative synthesized with Schaffer acid. The hydrazide (0.65 g; 2.5 mmol) was treated with conc. HCl (0.36 g; 10 mmol; 0.31 ml) and 2 mL water and then again with 10 mL water. After cooling with ice at 0-5 °C a solution of sodium nitrite (0.17 g; 2.5 mmol) in 2 ml water was added drop-wise to amine hydrochloride The acidity was finally checked (pH 2-3).

Coupling: The Schaffer acid (0.56 g; 2.5 mmol) was treated with NaOH (0.2 g; 5 mmol) and 4 ml water. The resulting sodium salt diluted with another 5 ml of water was cooled at 10 °C. The diazoderivative was added and also solid Na_2CO_3 to maintain the alkaline pH (8-8.5). The pH had turned into acid before filtration, η = 72%.

Diazoamino derivative synthesized with H acid. The hydrazide (0.65 g; 2.5 mmol) was treated with conc. HCl (0.36 g; 10 mmol; 0.31 ml) and 2 ml water and then with another 10 ml water. After cooling with ice a solution of sodium nitrite (0.172 g; 2.5 mmol) in 2 ml water was added dropwise. The acidity was finally verified (pH 2-3).

Coupling: The salicilic acid (0.61 g; 5.0 mmol) was treated with NaOH (0.40 g; 10 mmol) and water (5 ml). The diazonium salt was added stepwise to the coupling component. Solid Na_2CO_3 was added for maintaining the pH of 8.5-9. The reaction mixture was allowed to stay overnight and then diazoamino derivative was filtered and washed with water being finally submitted to purification in ethanol, η = 74%.

Diazoaminoderivative synthesized with BON acid. Diazotization was carried out as described above and the coupling at pH 8-9. The reaction mixture was finally acidified by diluted HCl, η = 71%.

RESULTS AND DISCUSSION

Synthesis of diazoaminoderivatives

The syntheses of dizoaminoderivatives were carried out in two stages beginning with the synthesis of sulfonamidated phenoxyacetic acid hydrazides according to Figure 1. In the second stage (Figure 2),

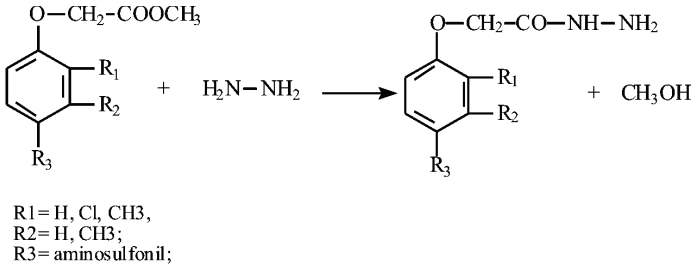


Figure 1. Syntheses of hydrazides.

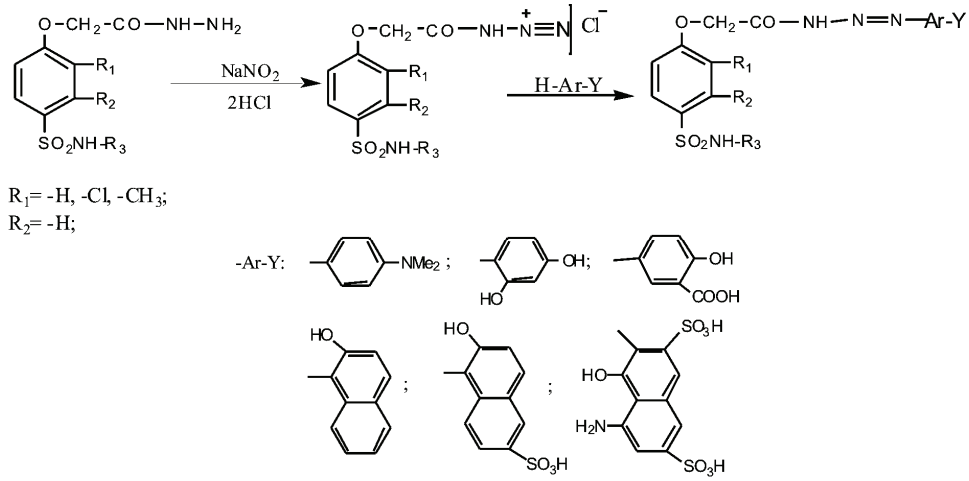


Figure 2. Syntheses of diazoamino derivatives.

the above mentioned hydrazides were submitted to the diazotization reaction followed by couplings with the following components: *N,N*-dimethylaniline, resorcine, *β*-naphthol, Schaffer acid, H acid, BON acid. The newly synthesized diazoaminoderivatives (Figure 3), their denominations, obtaining reaction mechanism as well as some of the physico-chemical characteristics and elemental analysis data are given below.

Spectral characterization

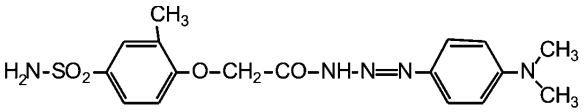
Physico-chemical characteristics and elemental analysis data of new diazoaminoderivatives are given in Table 1.

The structures of the newly obtained derivatives were elucidated by IR (Table 2), ^{1}H-NMR (Table 3) and UV-Vis (Table 4) spectral measurements.

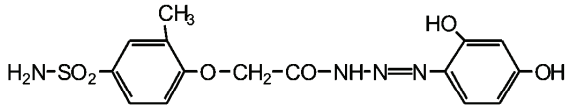
The IR spectra show the band characteristic of the ν(C=N) vibration at 1639.49 cm^{-1} (*β*-naphtol dye) or the lower wavenumber (1602.84 cm^{-1}) superimposed on the ν(C-C) absorption in aromatic rings. The azo group gives weak infrared absorptions for even unsymmetrical molecules. With the aromatic azo derivatives absorption bands at 1566±8 cm^{-1} and 1053±14 cm^{-1} are to be found but their position in spectra is rather difficult to be exactly identified. Since the diazoamino derivatives are of an aromatic structure, their vibrations are to be found especially within the ν(C-H) (2991.59-3057.17 cm^{-1}) and ν(C-H) (1454.32-1654.92 cm^{-1} valence vibration or ν(C-H) (646.15-883.40 cm^{-1}) deformation vibration ranges [36-43].

The fact can be noticed that by carrying out the diazotization-coupling pair operations the possibility of preparing diazoaminoderivatives of a structure similar to azomethines has risen. Their only structural difference is the imino group (-N=CH-) replacement by the azo group. This structure modification causes significant physico-chemical features for every compound. For instance, while azomethines are easily splitted hidrolitically in diluted strong acid media, the diazoamino derivatives are rather stable under such conditions.

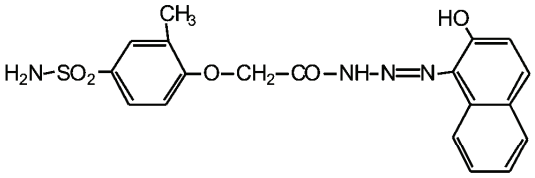
The ^{1}H-NMR spectra confirm the presence of the characteristic structural elements in every compound. The spectrum aliphatic region shows all types of methyl groups (NH_2, N-CH_3, N$(CH_3)_2$, O-CH_3, CH_3, Ar-CH_3) at the corresponding *δ* values. The aromatic protons in the phenyl residue could be differentiated according to their vicinities and couplings. With the compounds containing asymmetrically substituted benzene ring, two singlets of very close values correspond to them [37-40]. The values of the chemical shifts and the peak intensities in the ^{1}H-NMR spectra are in total agreement with the proton types and num-



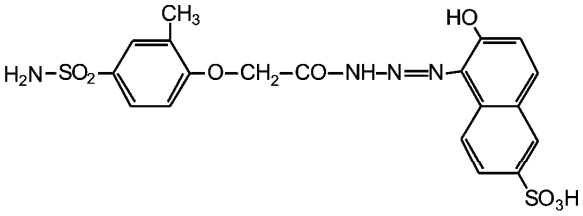
(2-methyl-4-sulfonamido-phenoxymethylen-ceto-tryaz)-N,N-dimethylaniline (1)



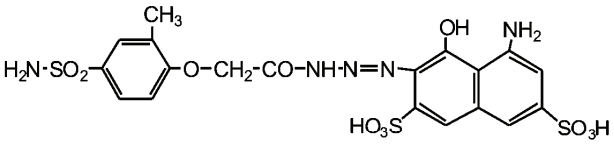
(2-methyl-4-sulfonamido-phenoxymethylen-ceto-tryaz)-resorcine (2)



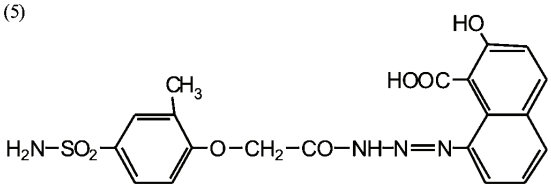
(2-methyl-4-sulfonamido-phenoxymethylen-ceto-tryaz)-2-naphthol (3)



(2-methyl-4-sulfonamido-phenoxymethylen-ceto-tryaz)-Schaffer acid (4)



(2-methyl-4-sulfonamido-phenoxymethylen-ceto-tryaz)-H acid (5)



(2-methyl-4-sulfonamido-phenoxymethylen-ceto-tryaz)- BON acid (6)

Figure 3. Structures of diazoamino derivatives.

ber in every diazoamino derivative. The ^{1}H-NMR spectra undoubtedly confirm the structures of the newly obtained diazoaminoderivatives.

The UV-Vis absorption spectra of the synthesized diazoamino derivatives were recorded on a RFS 100/S Raman spectrophotometer by using solutions in various solvents of concentrations between 2×10^{-5} M and 1×10^{-4} M and quartz cells of 0.5 and 1 cm.

Since the diazoaminoderivatives are not soluble in certain spectral solvents, the measurements were made with the 95% ethanol solution and dimethylsulfoxide solution which restricted the spectroscopic investigations.

The results and discussion on the structure-reactivity-applicability were made on each chromofore type and spectral shifting in function of the number and position of the substitutes in the aromatic moieties made evident.

The spectral characteristics of the synthesized diazoamino derivatives are indicative of three spectral domains. The spectral domains influenced by the solvent type are represented by the following bands: band I - λ = 259-265 nm, band II - λ = 277-292 nm and band III - λ = 286-342 nm. The UV-Vis spectrum of diazoaminoderivatives was recorded in 95% ethanol solution and DMF solution. Although diazoamino

Table 1. Physico-chemical characteristics and elemental analysis data of the new diazoamino derivatives of the sulphonamidated phenoxyalchyl carboxylic acids (R_1 = CH_3, R_2 = H, R_3 = amino sulphonil)

Compound	Ar-Y	Empirical formula	*M* g/mol	M.p. °C	Colour	Calcd./found, %		
						C	H	N
1	DMA	$C_{17}H_{21}N_5O_4S$	391	220-222	White-greenish	52.17/52.06	5.37/5.44	17.90/18.01
2	Resorcine	$C_{15}H_{16}N_4O_6S$	380	258-260	Dark-grey	47.36/47.22	4.21/4.30	14.73/14.84
3	*β*-Naphtol	$C_{19}H_{18}N_4O_5S$	414	208-210	Redish-brown	55.07/54.92	4.34/4.42	13.52/13.64
4	Schaffer acid	$C_{19}H_{18}N_4O_8S_2$	494	215-216	Grey	46.15/46.02	3.64/3.75	11.33/11.45
5	H Acid	$C_{19}H_{19}N_5O_{11}S_3$	589	206-208	White	38.70/38.55	3.22/3.31	11.88/11.98
6	BON Acid	$C_{20}H_{18}N_4O_7S$	458	216-218	Cream-coloured	52.40/52.28	3.93/4.02	12.22/12.31

Table 2. IR spectra of the obtained diazoamino derivatives; characteristic bands and their intensity, (VS = very intense, S = intense, M = medium intensity, W = weak, VW = very weak)

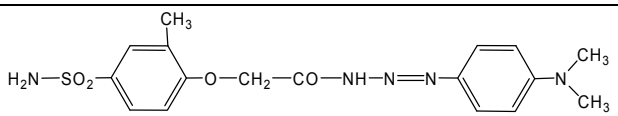
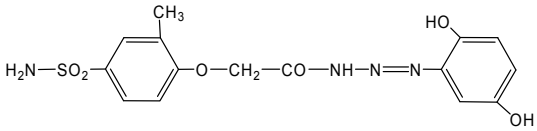
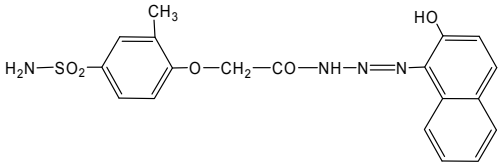
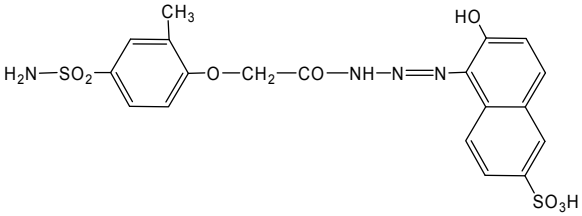
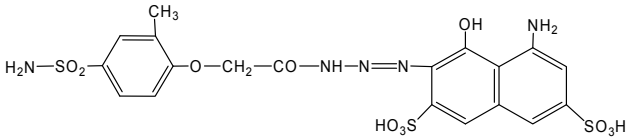
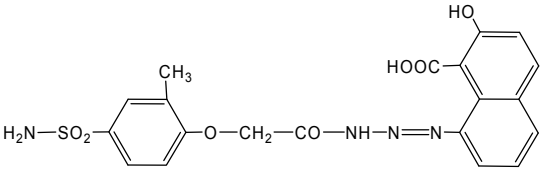
Compound	Characteristic bands, cm^{-1}
1	439.77 M, 457.13 W, 499.56 S, 513.06 S, 522.71 S, 530.42 S, 563.21 S, 584.43 S, 594.07 S, 721.38 VS, 812.03 VS, 912.33 VS, 954.76 W, 977.91 M, 1004.91 W, 1053.13 S, 1136.07 S, 1207.44 S, 1265.30 S, 1288.45 M, 1311.59 M, 1396.46 M, 1438.89 M, 1500.62 M, 1585.48 M, 1597.06 M, 1681.92 S, 2852.71W, 2914.43 M, 2993.52 M, 3082.24 M.
2	433.98.M, 443.63 M, 457.13 M, 466.77M, 499.56 S, 518.85 S, 538.14 S, 547.78 S, 592.15 S, 752.24 M, 806.24 M, 974.05 M, 999.12 M, 1010.70 M, 1066.63 S, 1099.42 S,1138.00 S, 1157.29 S, 1203.58 S, 1263.37 S, 1280.73 S, 1305.81 S, 1496.76 S,1583.55 S, 1598.98 S,1662.64 S, 2773.63 W, 2864.29 M, 2920.22 S, 2997.37 S, 3059.09 S, 3358.06 S.
3	439.77 M, 474.49 M, 495.70 M, 522.71 M, 545.85 M, 582.50 M, 590.22 M, 621.08 M, 663.51 M, 719.45 M, 742.59 M, 754.17 M, 1043.49 M, 1064.70 M, 1101.35 M, 1116.78 M, 1132.21 M, 1153.43 M, 1203.58 W,1217.08 M, 1251.80 M, 1265.30 M, 1315.45 M, 1381.03 M, 1600.91 M, 1633.70 S, 2916.36 M, 2947.22 M, 2993.52 M, 3383.14 M.
4	433.98 M, 443.63 M, 464.84 M, 499.56 S, 518.85 S, 547.78 M, 592.15 S, 642.29 M, 642.29 M, 723.30 M, 825.53 VS, 881.47 M, 891.11 S, 1097.49 S, 1136.07 S, 1157.29 S, 1224.80 S, 1265.30 S, 1323.16 S, 1371.39 M, 1381.M, 1442.75 S, 1496.76 S, 1552.69 S, 1583.55 M, 2864.29 M, 2958.80 W, 2999.30 M, 3676.31 W.
5	433.98 M, 443.63 M, 464.84 M, 499.56 M, 518.85 M, 547.78 W, 594.07 M, 688.59 M, 723.30 M, 825.53 S, 891.11 S, 929.69 S, 1157.29 M, 1224.80 M, 1263.37 M, 1321.24 M, 1369.46 M, 1381.03 M, 1442.68 M, 1498.69 M, 1558.48 S, 1583.55 S, 1598.98 S, 2864.29 M, 2958.80 M, 3689.82 W.
6	441.70 M, 457.13 W, 480.27 M, 505.35 M, 561.28 M, 584.43 M, 592.15 M, 630.72 W, 678.94 M, 721.38 W, 742.59 W, 759.95 W, 1097.49 S, 1138.00 S, 1188.15 S, 1226.72 M, 1263.37 M, 1328.95 S, 1386.82 W, 1431.18 M, 1456.25 W, 1496.76 M, 1662.64 W, 2927.94 M, 3371.56 VS.

Table 3. ^{1}H-NMR spectra of the diazoamino derivatives

Compound	Characteristic bands (DMSO), δ/ ppm
1	2.08 (s, 2H, $-NH_2$); 2,11 (s, 3H, $-CH_3$); 4.94 (s, 2H,$-CH_2-$); 5.36 (d, 6H, $-CH_3$); 7.08 (s, 1H, Ar); 7.29 (d, 2H, Ar); 7.49 (d, 2H, Ar); 7.73 (s, 2H, Ar); 8.16 (s, 1H, -NH-)
2	2.02 (s, 2H, $-NH_2$); 2.3 (s, 3H, $-CH_3$); 4.8 (s, 2H,$-CH_2-$); 4.98 (d, 2H, $(-OH)_2$); 6.99 (s, 1H, Ar); 7.10 (d, 2H, Ar); 7.25 (s, 1H, Ar); 7.33 (s, 1H, Ar); 7.47 (s, 1H, Ar); 8.09 (s, 1H, -NH-)
3	2.07 (s, 2H, $-NH_2$); 2.4 (s, 3H, $-CH_3$); 4.83 (d, 2H,$-CH_2-$); 5.21 (s, 1H, -OH); 6.98 (s, 1H, Ar); 7.10 (d, 2H, Ar); 7.17 (s, 1H, Ar); 7.29 (s, 1H, Ar); 7.48 (d, 2H, Ar); 7.83 (d, 2H, Ar); 8.1 (s,1H, -NH-)
4	2.07 (s, 2H, $-NH_2$); 2.21 (s, 3H, $-CH_3$); 4.83 (s, 2H, $-CH_2-$); 5.22 (s, 1H, -OH); 6.98 (s, 1H, Ar); 7.10 (d, 2H, Ar); 7.17 (s, 1H, Ar); 7.29 (s, 1H, Ar); 7.49 (d, 2H, Ar); 7.83 (s, 1H, Ar); 8,1 (s, 1H, -NH-); 10.7 (s, 1H, $-SO_3H$);
5	2.02 (s, 2H,$-NH_2$); 2.16 (s, 3H, $-CH_3$); 4.89 (s, 2H,$-CH_2-$); 5.08 (s, 1H, -OH); 6.21 (s, 2H, $-NH_2$), 6.98 (s, 1H, Ar); 7.1 (d, 2H, Ar); 7.17 (d, 2H, Ar); 7.81 (s, 1H, Ar); 8.11 (s, 1H, -NH-); 10.6 (d, 2H, $-SO_3H$)
6	2.04 (s, 2H, $-NH_2$), 2.25 (s, 3H,$-CH_3$); 4.92 (s, 2H,$-CH_2-$); 5.67 (s, 1H, -OH); 6.88 (s, 1H, Ar); 7.15 (d, 2H, Ar); 7.6 (s, 1H, Ar); 7.82 (d, 2H, Ar); 7.94 (d, 2H, Ar); 8.08 (s, 1H, -NH-); 11.8 (s, 1H, -COOH)

derivatives is soluble in both solvents it seems that DMF is a better solvent than ethanol. The UV-Vis spectra show strong π-π* bands at 265 and 292 nm and n-π* band at 342 nm. The spectral shifting are explained based on the type, number and position of the substitutes, the capacity of forming inter- and intra-molecular hydrogen bonds as well as the solvent polarity [41-43].

Table 4. UV-Vis spectral characteristics of some diazoamino derivatives

Compound	Structure	Solvent	λ_{max} / nm (ε)		
			Band I	Band II	Band III
1		DMSO	265	281	342
		Ethanol	-	278	-
2		DMSO	263	277	286
		Ethanol	-	269	-
3		DMSO	264	278	334
		Ethanol	261	-	329
4		DMSO	259	290	328
		Ethanol	-	290	324
5		DMSO	261	292	332
		Ethanol	259	289	329
6		DMSO	260	291	335
		Ethanol	259	287	327

CONCLUSIONS

Due to the particular biological potential of the sulfonamidated substituted phenoxyacetic acids, our researches have been extended to obtaining new derivatives able to show such properties with respect to their possible applications as pharmaceuticals as well as selective herbicides. The place, role and importance of the phenoxyacetic acid derivatives among the biologically active compounds were made evident. Diazotization reactions of the amidosulphonyl-R_1,R_2--phenoxyacetic acids hydrazides followed by coupling reactions with several components afforded the corresponding diazoamino-derivatives to be obtained. The newly obtained final products were characterized by means of elemental analysis data and spectral measurements (IR, ^{1}H-NMR, UV-Vis) which have undoubtedly confirmed the advanced structures of diazoamino derivatives.

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NAUČNI RAD

SINTEZA I KARAKTERIZACIJA NEKIH NOVIH DIAZOAMINODERIVATA

Sulfonamidne grupe su najčešće sastavni delovi strukture bioaktivnih jedinjenja. U ovom radu ispitivanje sulfonamidne ariloksiakilkarboksilne kiseline je prošireno njenim vezivanjem za različite supstrate kako bi se potvrdile neke biološke karakteristike dobijenih proizvoda, kao što su anti-tumorna i antioksidativna dejstva u tretiranju inflamatornih procesa, čira, konvulzija i dijabetesa, kao i herbicidalno dejstvo. U radu su opisani postupak sinteze derivata sulfonamidne ariloksiakilkarboksilne kiseline i njihova karakterizacija pomoću elementarne mikroanalize, IR, ^{1}H-NMR i UV-VIS spektralnih metoda. Novodobijena jedinjenja pokazuju potencijalne farmaceutske i herbicidne karakteristike.

Ključne reči: hidrazidi; komponente za kuplovanje; diazoaminoderivati, elementalna mikroanaliza, IR, ^{1}H-NMR, UV-VIS.