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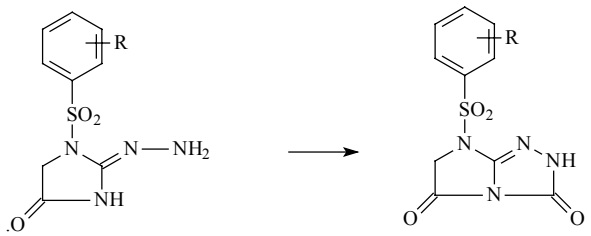
Synthesis of derivatives of 7-arylsulfonyl-3,5 (2H)-dioxo-6,7-dihydroimidazo[2,1-c] triazole. Part II

Synteza pochodnych 7-arylosulfonylo-3,5 (2H)-dioxo-6,7-dihydroimidazo[2,1-c] triazolu. Część II

Some derivatives of imidazo[2,1-c][1,2,4]triazole have been received and tested as antifungal [3], cardiovascular [2] drugs and as inhibitors of protein kinases [6]. The other derivatives of this ring system have been preparated as possible antibacterial agents [4].Based on this fact and continuing previous research works concerning synthesis of imidazo[2,1-c][1,2,4]triazoles [1,5] it seemed worthwhile to obtain derivatives of 7-arylsulfonyl-3,5(2H)-dioxo-6,7-dihydroimidazo[2,1-c][1,2,4]triazole. Compounds I ,III,V,VI were received for authors earlier [1]-by condensation of 1-arylsulfonyl-2-hydrazinoimidazolidin-4-ones with urea.Compounds II,IV were obtained with a new method and so were compounds I, III,V,VI.

RESULTS AND DISCUSSION

The desired compounds were received by condensation of 1-arylsulfonyl-2-hydrazinoimidazolidin-4-ones with 1,1'-carbonyldiimidazole-CDI. The reaction sequence leading to the formation of [I-VI] is outlined in Scheme 1



R = H, 2-CH_3, 4-CH_3, 2-Cl, 4-Cl, 4-$NHCOCH_3$

The physical data of new compounds are shown in Table 1

Table 1. The Physical data of compounds

Comp. .	R	Formula (mol.wt.)	M.p. (°C) Yield (%)	Analysis (calctd/found)				
				% C	% H	% Cl	% N	% S
I	H	$C_{10}H_8N_4O_4S$ 280.30	250-52 32	42.85 42.63	2.88 2.60		20.00 20.52	11.44 11.97
II	2-CH_3	$C_{11}H_{10}N_4O_4S$ 294.30	284-85 35	44.90 44.80	3.43 3.73		19.04 19.06	10.89 10.69
III	4-CH_3	$C_{11}H_{10}N_4O_4S$ 294.30	305-06 43	44.90 44.73	3.43 3.66		19.04 19.26	10.89 10.93
IV	2-Cl	$C_{10}H_7ClN_4O_4S$ 314.70	280-81 47	38.17 38.43	2.24 2.29	10.08 10.10	17.18 17.43	11.30 11.40
V	4-Cl	$C_{10}H_7ClN_4O_4S$ 314.70	255-57 52	38.17 38.23	2.24 2.34	10.08 10.11	17.18 17.85	11.30 11.63
VI	4-NHAc	$C_{12}H_{11}N_5O_5S$ 337.30	240-42 40	42.73 42.61	3.29 3.30		20.76 20.66	9.50 9.81

^{1}HNMR (DMSO-d_6); ppm for

Comp. I: 8.4 (s, 1H, NH-C=O); 6.9-7.5 (m, 5H, CH_{arom}); 3.72 (s, 2H, CH_2)

Comp. II: 8.2 (s, 1H, NH-C=O); 7.1-7.4 (m, 4H, CH_{arom}); 3.7 (s, 2H, CH_2); 2.34 (s, 3H, CH_3)

Comp. III: 8.3 (s, 1H, NH-C=O); 7.0-7.6 (m, 4H, CH_{arom}); 3.6 (s, 2H, CH_2); 2.4 (s, 3H, CH_3)

Comp. IV: 8.15 (s, 1H, NH-C=O); 6.8-7.2 (m, 4H, CH_{arom}); 3.65 (s, 2H, CH_2)

Comp. V: 8.2 (s, 1H, NH-C=O); 6.9-7.3 (m, 4H, CH_{arom}); 3.6 (s, 2H, CH_2)

EXPERIMENTAL DESIGN

M.ps were determined on Boetius apparatus and uncorrected ^{1}HNMR spectra chemical shifts were measured on a Tesla BS 567 A in $CDCl_3$ with an internal standard. All the synthesized compounds were checked by thin layer chromatography (TLC). The proton magnetic resonance (^{1}H NMR) spectra confirmed the presence of characteristic absorption band of two protons derived from imidazolidine formation occurring in the form of singlet at the range 3.6–3.72 ppm. The multiplet at 6.8–7.6 ppm was characteristic of aromatic protons. The singlet at about 8.3 ppm was characteristic of one proton derived from 1,2,4-triazole ring. Besides ^{1}H NMR spectra displayed singlet at about 2.4 ppm which characterizes the presence of methyl group Elemental analysis and spectral data are on request from the author. All compounds were recrystallized from ethanol.

SYNTHESIS OF 7-ARYLSULFONYL-3,5 (2H)-DIOXO-6,7-DIHYDROIMIDAZO-[2,1-C] TRIAZOLES [I-VI]

An appriopriate 7-arylsulfonyl-3,5 (2H)-dioxo-6,7-dihydroimidazo [2,1-c][1,2,4] triazole was obtained by cyclization of proper 1-arylsulfonyl-2-hydrazinoimidazolidin-4-one with 1,1'-carbonyldiimidazole(CDI). The CDI (0,01 mole) was added to 1-arylsulfonyl-2-hydrazinoimidazolidin-4-one (0,01 mole) dissolved in 50 cm^3 of DMF. The mixture was heated under reflux for 10–12 h. After cooling, the precipitate was separated and finally purified by recrystallization from ethanol.

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SUMMARY

The series of derivatives of 7-arylsulfonyl-3,5(2H)-dioxo-6,7-dihydroimidazo [2,1-c] [1,2,4]triazole was obtained as a result of condensation of 1-arylsulfonyl-2-hydrazinoimidazolidin-4-ones with 1,1'-carbonyldiimidazole. Based on the structure of imidazotriazole derivatives it is likely to happen that these compounds should reveal antifungal activity.

STRESZCZENIE

Otrzymano szereg pochodnych 7-arylosulfonylo-3,5(2H)-diokso-6,7-dihydroimidazo [2,1-c][1,2,4] triazolu w wyniku kondensacji 1-arylosulfonylo-2-hydrazynoimidazolidyno-4-onów z karbonylodiimidazolem. Na podstawie budowy pochodnych imidazotriazolu należy spodziewać się, że związki te powinny wykazywać działanie przeciwgrzybicze.