SYNTHETIC APPROACHES TO 6-ARYL-4-[(2-FURYL)METHYLIDENE]-1-(TOSYLAMINOCARBONYL)-1,2,3,4-TETRAHYDROPYRIDAZINE-3-ONES AND 5-[1-AROYLMETHYL-2-(2-FURYL)-1-ETHENYL]-2-(TOSYLAMINO)-1,3,4-OXADIAZOLES

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Abstract

Novel of 6-aryl-4-[(2-furyl)methylidene]-1-(tosylaminocarbonyl)-1,2,3,4-tetrahydropyridazine-3-ones **4a-d** were prepared by the reaction of α-aracyl-β-(2-furyl)acrylic acid hydrazides **2a-d** with tosylisocyanate. This has been shown to occur by initial formation of N^1-[2-aroylmethyl-3-(2-furyl)acroyloyl]-N^2-(tosylaminocarbonyl)hydrazines **3a-d** followed by acid catalyzed cyclization to afford 5-[1-aroylmethyl-2-(2-furyl)-1-ethenyl]-2-(tosylamino)-1,3,4-oxadiazoles **5a-d**.

Key words; furanones, pyridazinones, 1,3,4-oxadiazoles, tosylisocyanate.

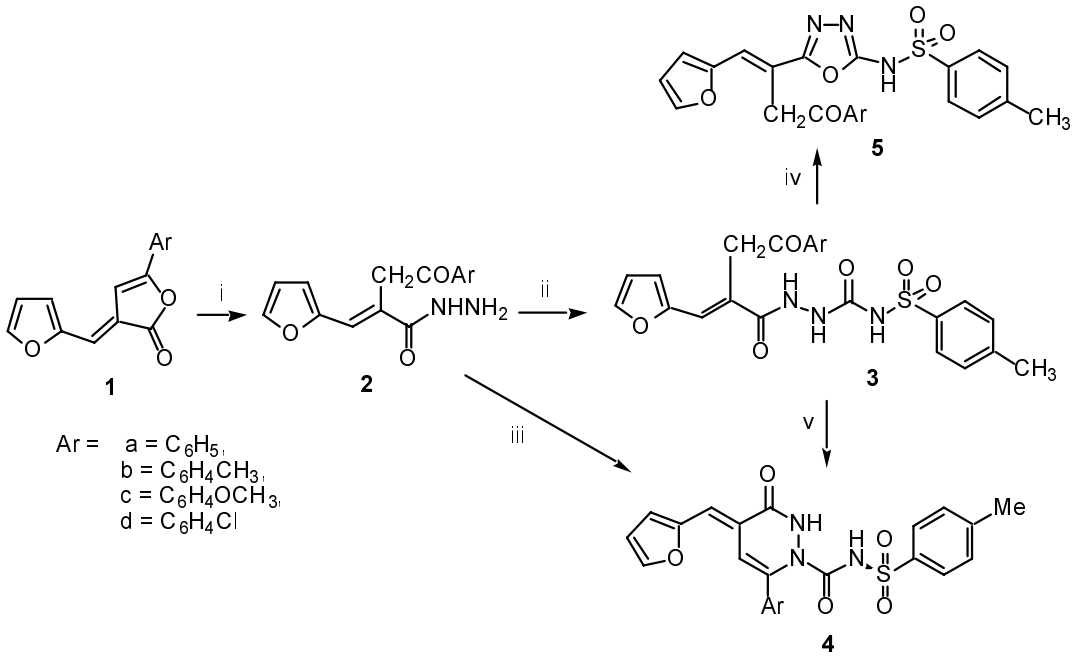
Introduction

In connection with our synthetic approach to the 5-aryl-3-[(-2-furyl)methylidene]-2(3*H*)-furanones **1a-d**,[1-4] these compounds were isolated in almost quantitative yield upon condensation reaction of furan-2-carboxaldehyde with 3-aroylpropionic acids under Perkin conditions.[5] Furanones **1a-d** react with hydrazine hydrate in ethanol to give α-aracyl-β-(2-furyl)acrylic acid hydrazides **2a-b**, which were found to be useful precursors in the synthesis of several heterocyclic compounds.[4] The conversion of the 2(3*H*)-furanones **1a-d** into the corresponding pyridazones **4a-d** and oxadiazoles **5a-d** is of potential biological interest.[6-10] Usually, 3(2*H*)-pyridazinones are prepared from β-oxoalkanoic acid derivatives and hydrazines[11] and 1,3,4-oxadiazoles are prepared from 1,2-diacylhydrazines.[12] We report here the preparation of N^1-[-2-aroylmethyl-3-(2-furyl)acroyloyl]-N^2-(tosylaminocarbonyl)hydrazines **3a-d**, which, upon acid catalyzed cyclization, furnished 6-aryl-4-[(2-furyl)methylidene]-1-(tosylaminocarbonyl)-1,2,3,4-tetrahydropyridazine-3-ones **4a-d** and 5-[1-aroylmethyl-2-(2-furyl)-1-ethenyl]-2-(tosylamino)-1,3,4-oxadiazoles **5a-d**.

Results and Discussion

Reaction of α-aracyl-β-(2-furyl)acrylic acid hydrazides **2a-d** with tosylisocyanate at 0–25 °C gave N^1-[-2-aroylmethyl-3-(2-furyl)acroyloyl]-N^2-(tosylaminocarbonyl) hydrazines **3a-d**, and 6-aryl-4-[(2-furyl)methylidene]-1-(tosylaminocarbonyl)-1,2,3,4-tetrahydropyridazine-3-ones **4a-d**. Formation of products **3a-d** and **4a-d** was dependent on the reaction conditions. When the reaction was carried out at 0–5 °C for 12 h, N^1-[-2-aroylmethyl-3-(2-furyl)acroyloyl]-N^2-(tosylaminocarbonyl)hydrazines **3a-d** were obtained in 31–45% yield. However, treatment of **2a-d** with tosylisocyanate at 25 °C for 48 h gave 6-aryl-4-[(2-furyl)methylidene]-1-(tosylaminocarbonyl)-1,2,3,4-tetrahydro-pyridazine-3-ones **4a-d** in 75–82% yield. Pyridazinones **4a-d** were also obtained by cyclization of hydrazides **3a-d** in a mixture of acetonitrile and 1 N hydrochloric acid. Treatment of hyrazides **3a-d** with $POCl_3$ under reflux furnished the corresponding 5-[1-aroylmethyl-2-(2-furyl)-1-ethenyl]-2-(tosylamino)-1,3,4-oxadiazoles **5a-d** in 75–81% yield (Scheme 1).

Scheme 1



Reagents and conditions; i) NH_2NH_2 . H_2O/ethanol, stirring at RT for 2 days; ii) Stirring with tosylisocyanate in acetonitrile at 0-5 °C for 12hrs; iii) Stirring with tosylisocyanate in acetonitrile at 25 °C for 2 days. iv) Refluxing with $POCl_3$ for 20 min; v) Stirring with 1.0 N HCl in acetonitrile for 1h.

The structures of novel compounds **3-5** were determined by spectroscopic methods and analyses for C, H, N and S. The IR spectral data for compounds **4b,d** are in agreement with the data of related 1-benzoyl-6-aryl-4-thienylidene-1,6-dihydropyridazin-3-(2*H*)ones.[13]

Experimental

^{1}H NMR spectra were recorded on Varian Plus 300 (300 MHz) or Bruker XL 300 (300 MHz) instruments, the ^{13}C NMR spectra (with DEPT 135) on a Bruker WP80 or XL 300 instrument. Infrared Red spectra were taken on a Perkin Elmer 1600 FT-IR spectrometer. Mass points were recorded on a Kratos Concept instrument. Melting points were measured on an Electrothermal digital melting point apparatus and are uncorrected. The R_f values reported for TLC analyses were determined on Macherey-Nagel 0.25 mm layer fluorescent UV_{254} plates with the indicated solvent system. Elemental analyses were performed by M-H-W Laboratories (Phoenix, AZ) at University of Minho, Braga, Portugal.

***N*^{1}-[-2-Aroylmethyl-3-(2-furyl)acroyloyl]-*N*^{2}-(tosylaminocarbonyl)hydrazines 3a-d. General Procedure A.** To a suspension of α-aracyl-β-(2-furyl)acrylic acid hydrazide **2a-b** (2 mmole) in CH_3CN (2 mL) under nitrogen atmosphere at 0–5°, *p*-toluenesulfonylisocyanate (0.3 mL, 2.1 mmole) was added and the mixture was stirred at 0–5° for 12 h. The precipitate was collected by filtration to give **3a-d**. Experimental and analytical data for compounds **3a-d** are given in Tables 1 and 2.

6-Aryl-4-[2-furyl)methylidene]-1-(tosylaminocarbonyl)-1,2,3,4-tetrahydro-pyridazine-3-ones 4a-d.

General Procedure B. To a suspension of α-aracyl-β-(2-furyl)acrylic acid hydrazide **2a-b** (2 mmole) in CH_3CN (2 mL) under nitrogen atmosphere at 0–5°, *p*-toluenesulfonylisocyanate (0.3 mL, 2.1 mmole) was added and the mixture was stirred at 0–5° for 12 hours and then at 25°for 48 h. The solvent was evaporated *in vacuo* and the solid residue was crystallized from ethanol to give **4a-d**.

General Procedure C. To a suspension of *N*^{1}-[2-aroylmethyl-3-(2-furyl)acroyloyl]-*N*^{2}-(tosylaminocarbonyl)hydrazine **3a-d** (1 mmole) in acetonitrile (4 mL), 1.0 N HCl (1

mL) was added and the mixture was stirred at 25° for one hour. The solvent was evaporated *in vacuo* and the solid residue was crystallized from ethanol to give **4a-d**. Experimental and analytical data for compounds **4a-d** are given in Tables 1, 2.

5-[1-Aroylmethyl-2-(2-furyl)-1-ethenyl]-2-(tosylamino)-1,3,4-oxadiazoles 5a-d.

General Procedure D. A mixture of N^1-[2-aroylmethyl-3-(2-furyl)acroyloyl]-N^2-(tosylaminocarbonyl)hydrazine **3a-d** (2 mmole) and $POCl_3$ (10 mL) was heated at 106 ^oC for 1 h. The mixture was cooled, poured into crushed ice (20 mL), neutralized with 1.0 N aqueous solution of $NaHCO_3$. The yellowish precipitate was collected by filtration, washed with water and crystallized from acetonitrile to give **5a-d**. Experimental and analytical data for compounds **5a-d** are given in Tables 1, 2.

Table 1. Physical data, yields and elemental analysis data for compounds **3-5**.

Cpd No.	*Aryl group*	*m.p* ^oC	*Yield*	*Recryst. Solv.*	*MF*	*Analysis [Calc./found]* *C* *H* *N* *S*
3a	C_6H_5	110-112	45 %	CH_3CN	$C_{23}H_{21}N_3O_6S$	59.09/59.55 4.52/4.62 8.98/9.78 6.85/6.73
3b	$C_6H_5CH_3$	133-135	32 %		$C_{24}H_{23}N_3O_6S$	59.98/59.86 4.82/4.85 8.74/8.74 6.67/6.68
3c	$C_6H_5OCH_3$	125-127	31 %		$C_{24}H_{23}N_3O_7S$	58.05/58.01 4.66/6.65 8.46/8.57 6.45/6.77
3d	C_6H_5Cl	190-192	34 %		$C_{23}H_{20}ClN_3O_6S$	55.03/55.15 4.01/3.99 8.37/8.36 6.38/6.42
4a	C_6H_5	156-158	75 %	EtOH	$C_{23}H_{19}N_3O_5S$	61.45/61.55 4.26/4.16 9.34/9.28 7.13/7.33
4b	$C_6H_5CH_3$	175-177	82 %		$C_{24}H_{21}N_3O_5S$	62.19/62.18 4.56/4.55 9.06/9.09 6.91/6.89
4c	$C_6H_5OCH_3$	168-170	82 %		$C_{24}H_{21}N_3O_6S$	60.11/60.15 4.41/4.45 8.76/8.76 6.68/6.77
4d	C_6H_5Cl	185-186	77 %		$C_{23}H_{18}ClN_3O_5S$	57.08/57.05 3.74/3.73 8.68/8.66 6.62/6.62
5a	C_6H_5	145-147	75%	CH_3CN	$C_{23}H_{19}N_3O_5S$	61.45/61.55 4.26/4.16 9.34/9.28 7.13/7.33
5b	$C_6H_5CH_3$	176-177	80 %		$C_{24}H_{21}N_3O_5S$	62.19/62.18 4.56/4.55 9.06/9.09 6.91/6.89
5c	$C_6H_5OCH_3$	178-180	81 %		$C_{24}H_{21}N_3O_6S$	60.11/60.15 4.41/4.45 8.76/8.76 6.68/6.77
5d	C_6H_5Cl	166-168	75 %		$C_{23}H_{18}ClN_3O_5S$	57.08/57.05 3.74/3.73 8.68/8.66 6.62/6.62

Table 2. Infrared (IR) and ^{1}H NMR (300 MHz) spectral data for compounds **3-5.**

Cpd. No	*Aryl group*	*(IR)$_{max}$(Nujol)/ cm^{-1}* $\upsilon C=O$; $\upsilon C=N$; υ-NH	*^{1}H NMR (300 MHz , DMSO-d_6); δH [DMSO-d_6]*
3a	C_6H_5	1667(s), 1725(w), 3112(w), 3263.6(s)	δ = 2.36 (s, 3H, Ar-C<u>H</u>$_3$), 3.85 (s, 2H, C<u>H</u>2COPh), 6.63 (dd, 1H, *J* = 1.8, 3.3 Hz, Furan-H4), 6.75 (d, 1H, *J* = 3 Hz, Furan-H3), 7.04 (s, 1H, furan-CH=C-), 7.35 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.37-7.43 (m, 5H, Ph-H), 7.64 (d, *J* = 1.8 Hz, Furan-H5), 7.75 (d, 2H, *J* = 8.1 Hz, Ar-H), 9.30 (s, 1H, -NH-NH-), 10.67 (s, 1H,-NH-NH-), 11.63 (s, 1H, -CONHSO2-Ar) ppm.
3b	$C_6H_5CH_3$	1667(s), 1699, 1725.8(w), 3099(w), 3266.6(S)	δ = 2.36 (s, 3H, Ar-C<u>H</u>$_3$), 2.37 (s, 3H, Ar-C<u>H</u>$_3$), 3.88 (s, 2H, C<u>H</u>2COAr), 6.70 (s, 1H, furan-CH=C-), 6.87 (d, 2H, *J* = 9 Hz, Ar-H), 7.00 (d, 1H, *J* = 3.6 Hz, Furan-H3), 7.14 (dd, 1H, J = 1.8, 3.6 Hz, Furan-H4), 7.32 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.45 (d, 2H, *J* = 9 Hz, Ar-H), 7.69 (d, 2H, *J* = 7.5 Hz, Ar-H), 7.82 (s, 1H, -NH-NH-), 7.96 (d, *J* = 1.5 Hz, Furan-H5), 8.28 (s, 1H, -NH-NH-), 10.68 (s, 1H, -CONHSO2-Ar) ppm.
3c	$C_6H_5OCH_3$	1651(s), 1680(s), 1737(w), 3214(s), 3376(w)	δ = 2.44 (s, 3H, Ar-C<u>H</u>$_3$), 3.80 (s, 3H, Ar-OC<u>H</u>$_3$), 3.85 (s, 2H, C<u>H</u>2COAr), 6.75 (d, 1H, *J* = 3 Hz, furan-CH=C-), 6.85 (d, 2H, *J* = 9 Hz, Ar-H), 7.05 (d, 1H, *J* = 3.6 Hz, Furan-H3), 7.12 (dd, 1H, *J* = 1.8, 3.6 Hz, Furan-H4), 7.37 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.41 (d, 2H, *J* = 9 Hz, Ar-H), 7.71 (d, 2H, *J* = 7.5 Hz, Ar-H), 7.81 (s, 1H, -NH-NH-), 7.95 (d, *J* = 1.5 Hz, Furan-H5), 8.27 (s, 1H, -NH-NH-), 10.68 (s, 1H, -CONHSO2-Ar) ppm.
3d	C_6H_5Cl	1663(s), 1702(s), 1726.1(w), 3233(s), 3316(w)	δ = 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 3.79 (s, 2H, C<u>H</u>2COAr), 6.61 (s, 1H, furan-CH=C-), 6.85 (d, 2H, *J* = 9 Hz, Ar-H), 7.1 (d, 1H, *J* = 3.6 Hz, Furan-H3), 7.2 (dd, 1H, *J* = 1.8, 3.6 Hz, Furan-H4), 7.38 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.42 (d, 2H, *J* = 9 Hz, Ar-H), 7.73 (d, 2H, *J* = 7.5 Hz, Ar-H), 7.85 (s, 1H, -NH-NH-), 7.97 (d, *J* = 1.5 Hz, Furan-H5), 8.57 (s, 1H, -NH-NH-), 10.72 (s, 1H, -CONHSO2-Ar) ppm.
4a	C_6H_5	1662(s),1699(w) 3112(w), 3263.6(s)	δ = 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 6.55 (s, 1H, -CH=C-Ar), 6.70 (dd, 1H, *J* = 1.8, 3.3 Hz, Furan-H4), 7.06 (s, 1H, furan-CH=C-), 7.15 (d, 1H, *J* = 3.3 Hz, Furan-H3), 7.30-7.42 (m, 5H, Ph-H), 7.46 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.69 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.95 (d, *J* = 1.5 Hz, Furan-H5), 9.32 (s, 1H, NH), 11.49 (s, 1H, -CONHSO2-Ar) ppm.
4b	$C_6H_5CH_3$	1668(s), 1725.8(w), 3099(w), 3266.6(S)	δ = 2.35 (s, 3H, Ar-C<u>H</u>$_3$), 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 6.48 (s, 1H, -CH=C-Ar), 6.71 (dd, 1H, *J* = 1.8, 3.6 Hz, Furan-H4), 6.85 (d, 2H, *J* = 8.7 Hz, Ar-H), 6.98 (s, 1H, furan-CH=C-), 7.18 (d, 1H, *J* = 3.6 Hz, furan-H3), 7.36 (d, 2H, *J* = 8.4, Ar-H), 7.42 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.68 (d, 2H, J = 8.4 Hz, Ar-H), 7.92 (d, J = 1.5 Hz, furan-H5), 8.32 (s, 1H, NH), 10.49 (s, 1H, -CONHSO2-Ar) ppm.

Table 2 Continued.

4c	$C_6H_5OCH_3$	1679(s), 1737(w), 3214(s), 3376(w)	δ = 2.36 (s, 3H, Ar-C<u>H</u>$_3$), 3.78 (s, 3H, Ar-OC<u>H</u>$_3$), 6.46 (s, 1H, -CH=C-Ar), 6.68 (dd, 1H, *J* = 1.8, 3.3 Hz, Furan-H4), 6.86 (d, 2H, *J* = 8.7 Hz, Ar-H), 6.99 (s, 1H, furan-CH=C-), 7.1 (d, 1H, *J* = 3.3 Hz, furan-H3), 7.35 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.42 (d, 2H, *J* = 9 Hz, Ar-H), 7.69 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.92 (d, *J* = 1.5 Hz, furan-H5), 8.27 (s, 1H, NH), 10.67 (s, 1H, -CONHSO2-Ar) ppm.
4d	C_6H_5Cl	1665(s), 1728(w), 3233(s), 3316(w)	δ = 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 6.54 (s, 1H, -CH=C-Ar), 6.71 (dd, 1H, *J* = 1.8, 3.3 Hz, Furan-H4), 6.87 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.1 (s, 1H, furan-CH=C-), 7.18 (d, 1H, *J* = 3.3 Hz, furan-H3), 7.37 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.47 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.69 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.95 (d, *J* = 1.5 Hz, furan-H5), 9.34 (s, 1H, NH), 10.86 (s, 1H, -CONHSO2-Ar) ppm.
5a	C_6H_5	1698.2(s), 1578-1615, 3263	δ = 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 4.36 (s, 2H, -C<u>H</u>2COPh), 6.66 (dd, 1H, *J* = 1.8, 3.3 Hz, Furan-H4), 7.1 (s, 1H, furan-CH=C-), 7.15 (d, 1H, *J* = 3.3 Hz, Furan-H3), 7.35-7.46 (m, 5H, Ph-H), 7.48 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.69 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.95 (d, *J* = 1.5 Hz, Furan-H5), 11.49 (s, 1H, -CONHSO2-Ar) ppm.
5b	$C_6H_5CH_3$	1692(s), 1587-1613, 3216(s)	δ = 2.35 (s, 3H, Ar-C<u>H</u>$_3$), 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 4.36 (s, 2H, -C<u>H</u>2COAr), 6.67 (dd, 1H, *J* = 1.8, 3.6 Hz, Furan-H4), 6.82 (d, 2H, *J* = 8.4 Hz, Ar-H), 6.98 (s, 1H, furan-CH=C-), 7.17 (d, 1H, *J* = 3.3 Hz, furan-H3), 7.35 (d, 2H, *J* = 8.7, Ar-H), 7.44 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.67 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.93 (d, *J* = 1.5 Hz, furan-H5), 10.49 (s, 1H, -CONHSO2-Ar) ppm.
5c	$C_6H_5OCH_3$	1692(s), 1589–1610, 3220(s)	δ = 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 3.81 (s, 3H, Ar-OC<u>H</u>$_3$), 4.47 (s, 2H, -C<u>H</u>2COAr),, 6.71 (dd, 1H, *J* = 1.8, 3.3 Hz, Furan-H4), 6.87 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.1 (s, 1H, furan-CH=C-), 7.18 (d, 1H, *J* = 3.3 Hz, Furan-H3), 7.37 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.47 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.69 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.95 (d, *J* = 1.5 Hz, Furan-H5), 10.86 (s, 1H, -CONHSO2-Ar) ppm.
5d	C_6H_5Cl	1689(s), 1590-1620, 3217(s)	δ = 2.38 (s, 3H, Ar-C<u>H</u>$_3$), 4.47 (s, 2H, -C<u>H</u>$_2$COAr),, 6.71 (dd, 1H, *J* = 1.8, 3.3 Hz, Furan-H4), 6.87 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.1 (s, 1H, furan-CH=C-), 7.18 (d, 1H, *J* = 3.3 Hz, Furan-H3), 7.37 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.47 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.69 (d, 2H, *J* = 8.7 Hz, Ar-H), 7.95 (d, *J* = 1.5 Hz, Furan-H5), 10.86 (s, 1H, -CONHSO2-Ar) ppm.

Conclusion

6-Aryl-4-[(2-furyl)methylidene]-1-(tosylaminocarbonyl)-1,2,3,4-tetrahydropyridazin-3-ones **4a-d** and 5-[1-aroylmethyl-2-(2-furyl)-1-ethenyl]-2-(tosylamino)-1,3,4-oxadiazoles **5a-d** were prepared in one step by acid catalyzed cyclization of N^1-[2-aroylmethyl-3-(2-furyl)acroyloyl]-N^2-(tosylaminocarbonyl)hydrazines **3a-d** in good yields.

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Povzetek

Nove derivate 6-aril-4-[(2-furil)metiliden]-1-(tozilaminokarbonil)-1,2,3,4-tetrahidro-piridazin-3-onov **4a-d** smo pripravili z reakcijami α-aracil-β-(2-furil)akrilohidrazidov **2a-d** s tozilisocianatom in jih nato pretvorili v 5-[1-aroilmetil-2-(2-furil)-1-etenil]-2-(tozilamino)-1,3,4-oksadiazole **5a-d**. Strukture spojin in intermediatov smo potrdili z analiznimi in spektroskopskimi podatki.