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SYNTHESIS OF 3-METHYLISOXAZOLE- 5-CARBOXAMIDES AND 5-[(1H-PYRAZOL-1-YL)CARBONYL]- 3-METHYLISOXAZOLES

Marcos A. P. Martins^a, Marcelo Neto^a, Adilson P. Sinhorin^a, Giovani P. Bastos^a, Nilo E. K. Zimmermann^a, Adriano Rosa^a, Helio G. Bonacorso^a & Nilo Zanatta^a

^a Departamento de Química , Núcleo de Química de Heterociclos (NUQUIMHE) , Universidade Federal de Santa Maria , Santa Maria, RS, 97.105-900, Brazil Published online: 16 Aug 2006.

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SYNTHESIS OF 3-METHYLISOXAZOLE-5-CARBOXAMIDES AND 5-[(1*H*-PYRAZOL-1-YL)CARBONYL]-3-METHYLISOXAZOLES

Marcos A. P. Martins,* Marcelo Neto, Adilson P. Sinhorin, Giovani P. Bastos, Nilo E. K. Zimmermann, Adriano Rosa, Helio G. Bonacorso, and Nilo Zanatta

Departamento de Química, Núcleo de Química de Heterociclos (NUQUIMHE), Universidade Federal de Santa Maria, 97.105-900 Santa Maria, RS, Brazil

ABSTRACT

The one-pot synthesis of six 3-methylisoxazole-5-carboxamides **2** [where the *N*-substituents are $R^1 = H$, Me and $R^2 = Ph$, CH₂Ph, *n*-Bu, C(CH₃)₂Et, 3-methylisoxazol-5-yl] and twelve 5-[(1*H*-pyrazol-1-yl)carbonyl]-3-methyl isoxazoles **3** and **4** [where the pyrazole substituents are R^3 (C5/C3) = CO₂Et, CF₃ and R^5 (C3/C5) = H, Me, Et, Ph] from the 3-methyl isoxazole-5-carboxylic acid, thionyl chloride and the corresponding amine or pyrazole is reported. In the synthesis of 5-[(1*H*-pyrazol-1-yl)carbonyl]-3-methylisoxazoles we obtained a mixture of 1,3- and 1,5-isomers **3** and **4** in variable

425

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^{*}Corresponding author. E-mail: mmartins@base.ufsm.br; http://www.ufsm.br/ nuquimhe

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ratios, i.e., the substituent R^3 (CO₂Et and CF₃) are present in position 3 or 5 on the pyrazole ring.

Isoxazole derivatives possess very interesting pharmacological properties, especially isoxazolecarbonyl compounds that show marked action as diuretic,¹ antiinflammatory, analgesic, antipyretic, anticoagulant, and antireumatic. Isoxazolecarbonyl compounds are also important precursors for the synthesis of other compounds such as agrochemicals and microbicides.² As a part of our research program, we developed a general onestep procedure for preparing a series of analytically pure 4-alkoxy-1,1, 1-trihalo-3-alken-2-ones, from the acylation of several enol ethers (or acetals), in molar quantities.³ These compounds have been used as precursors of a variety of substituted five-, six- and seven-membered heterocyclic compounds, e.g., isoxazoles,^{3,4} pyrazoles,⁵ pyrimidines⁶ and diazepines.⁷ Recently,⁸ we reported a one-pot synthesis of isoxazole-5-carboxylic acids from the cyclocondensation of 4-alkoxy-1,1,1-trichloro-3-alken-2-ones with hydroxylamine, in hydrochloric acid or sulfuric acid medium. The aim of this work is to show the one-pot synthesis of a series 3-methylisoxazole-5-carboxamides (2) and 5-[(1H-pyrazol-1-yl)carbonyl]-3-methylisoxazoles (3,4) from the 3-methyl isoxazole-5-carboxylic acid (1), thionyl chloride and the corresponding amine or pyrazole (scheme).

3-Methylisoxazole-5-carboxylic acid (1) was synthesized from the cyclocondensation reaction of 4-alkoxy-1,1,1-trichloro-3-alken-2-ones with hydroxylamine in sulfuric acid.⁸ The 3(5)-trifluoromethylpyrazoles and pyrazole-3(5)-carboxylic acid ethyl ester were synthesized according as previous report.⁵ The preparation of 3-methylisoxazole-5-carbonyl chloride from 1 was carried out with an excess of thionyl chloride and catalytic amounts of pyridine, following by reflux for 16 h.

Compounds **2a–f** were obtained by addition of amines to equimolar amounts of 3-methylisoxazole-5-carbonyl chloride, obtained *in situ*. The mixture was stirred for 12 h at 60°C, and isolated products were recrystallized. Compounds **3a–d** and **4a–d** were obtained by addition of pyrazoles to 3-methylisoxazole-5-carbonyl chloride as described for **2** with chloroform as solvent. The mixture was stirred for 12 h at reflux. The recrystallized products were identified by ¹H and ¹³C NMR spectroscopy.

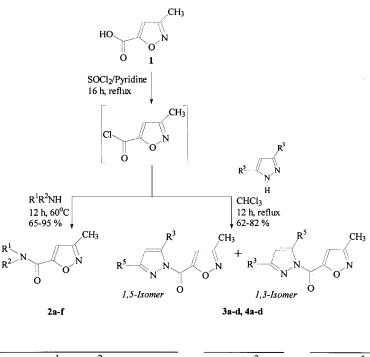
The assignments of Z- and E-isomers in the carboxamide group of 2a-f were done based on the literature data.⁹ Then, we assign compounds 2a-e as E-isomer and 2f shown a 1:1 mixture of Z- and E-isomers.

The assignments of 1,3- and 1,5-isomers of compounds 3 and 4 were done experimentally by the aromatic solvent induced shifts $(ASIS)^{5,10}$ and a two-dimensional correlation spectrum HMBC (*Heteronuclear Multiple Bond*



ORDER		REPRINTS
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Compd.	Rl	R ²	Compd	l. R ³	Compd	. R ⁵
2					3, 4	
а	Н	Ph	3	COOEt	а	Н
b	Н	CH ₂ Ph	4	CF3	b	Me
c	Н	C(CH ₃) ₂ Et			с	Et
d	Н	5-methylisoxazol-3-yl			d	Ph
e	Me	Ph				
f	Me	<i>n</i> -Bu				

Scheme.

Correlation).^{11,12} In the ASIS experiment, the ¹H NMR spectra are recorded in chloroform-d₁ and benzene-d₆ as solvents and all ¹H signals were shifted to upfield in benzene-d₆ when compared with the initial positions in chloroform-d₁. Due to the anisotropy *solvatation*^{9,5} of the aromatic solvent, the hydrogens closer to N-1 (an amidic nitrogen, with partial positive charge) experience more shielding than the others hydrogens of the molecule. Then we observed that \mathbb{R}^5 (hydrogen or alkyl groups) in compounds **3a–c** and **4a–c** was shifted upfield more in one isomer than in the other.



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MARTINS ET AL.

Thus, it was possible to assign the isomer that resonates more upfield as the *1,3-isomer* (\mathbb{R}^5 is closer to N-1). The assignment proposed were confirmed with the HMBC method. In the HMBC experiment, the long range coupling constant (${}^{2,3}J_{\text{C-H}}$) was optimized for 7 Hz. The experiment showed that the carbonyl carbon (attached on N-1) presented a cross-peak with the hydrogen attached on C-5 ($\mathbb{R}^5 = H$) for compounds **3a** and **4a** which were used as standard compounds. This fact identified the *1,3-isomers*, where $\mathbb{R}^5 = H$ is three bonds from the N-1 carbonyl carbon.

EXPERIMENTAL

Unless otherwise indicated all common reagents and solvents were used as obtained from commercial suppliers without further purification. Yields listed in Table 1 are of isolated compounds and all melting points were taken on a melting point microscope Reichert-Thermovar. Elemental analysis was carried out on an Elemental Analysensysteme Vario EL equipment. ¹H and ¹³C NMR spectra were recorded on a Bruker DPX 400, at 298 K, digital resolution of ± 0.01 ppm, 0.5 M in chloroform-d₁/TMS.

Synthesis of 3-Methylisoxazole-5-carboxamides 2a-f

General Procedure

A mixture of **1** (10 mmol), with catalytic amounts of pyridine, and thionyl chloride (1.43 g, 12 mmol) was refluxed for 12–16 h. The mixture was cooled to room temperature, the amine (R^1R^2NH , 12 mmol) added slowly, and the mixture stirred for 8–12 h at 60°C. The crude product was washed with water and then diluted in chloroform. The solvent was evaporated and the products **2a–f** were recrystallized from a mixture of hexane/ethyl acetate. Yields and selected physical and spectroscopic data are presented in Tables 1 and 2.

Synthesis of 5-[(1*H*-Pyrazol-1-yl)carbonyl]-3-methylisoxazoles 3a-d and 4a-d

General Procedure

A mixture of 1 (10 mmol), with catalytic amounts of pyridine, and thionyl chloride (1.43 g, 12 mmol) was refluxed for 12-16 h. The mixture





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Table 1. Selected Physical and Elemental Analysis Data of Compounds 2a–f, 3a–d and 4a–d

	Molecular Formula	Isomer Ratio	Yield	M.P.	Elemental (% Calcd./	(o)
Compound	(M. wt.)	1,3:1,5 ^a	(%) ^b	(°C)	С	Н
2a	$C_{11}H_{10}N_2O_2$	_	95	155–156	65.34	4.98
2b	$(202.21) \\ C_{12}H_{12}N_2O_2 \\ (216.23)$	_	88	148–150	65.16 66.66 66.60	4.97 5.59 5.58
2c	$\begin{array}{c} (210.23) \\ C_{10}H_{16}N_2O_2 \\ (196.24) \end{array}$	_	72	140–141	61.21 61.04	8.22 8.20
2d	C ₉ H ₉ N ₃ O ₃ (207.18)	—	78	149–153	52.18 51.90	4.38 4.35
2e	$\begin{array}{c} C_{12}H_{12}N_2O_2\\ (216.23)\end{array}$	-	88	158–160	66.66 66.43	5.59 5.57
2f	$C_{10}H_{16}N_2O_2$ (196.24)	-	55	164–166	61.21 61.00	8.22 8.19
3a	$C_{11}H_{11}N_3O_4$ (249.21)	4:1	73	122–124	53.02 52.82	4.45 4.40
3b	C ₁₂ H ₁₃ N ₃ O ₄ (263.24)	2:3	72	84–85	54.75 54.55	4.98 4.95
3c	C ₁₃ H ₁₅ N ₃ O ₄ (277.26)	4:5	70	98–99	56.32 56.10	5.45 5.43
3d	C ₁₇ H ₁₅ N ₃ O ₄ (325.31)	3:5	62	111–113	62.77 62.53	4.65 4.63
4 a	$C_9H_6F_3N_3O_2$ (245.15)	2:1	82	102–103	44.09 43.95	2.47 2.46
4b	$\begin{array}{c} C_{10}H_8F_3N_3O_2\\ (259.18) \end{array}$	4:1	80	108–109	46.34 46.19	3.11 3.09
4c	$\begin{array}{c} C_{11}H_{10}F_{3}N_{3}O_{2}\\ (273.21)\end{array}$	3:1	78	148–150	48.36 48.20	3.69 3.67
4d	$\begin{array}{c} C_{15}H_{10}F_{3}N_{3}O_{2}\\ (321.25)\end{array}$	5:1	78	99–100	56.08 55.90	3.14 3.13

^aIsomer ratio obtained from ¹H NMR data. ^bYields of isolated products.

was cooled to room temperature, the pyrazole (10 mmol) in chloroform (5 mL) added slowly, and the mixture stirred for 8–12 h reflux. The crude product was washed with water and then diluted in chloroform. The solvent was evaporated and the products 2a-f were recrystallized from a mixture of

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MARTINS ET AL.

Table 2. Selected ¹H and ¹³C NMR Data^a of Compounds 2a-f, 3, 4a-d

1 ubic 2	. Selected II and CIAMIR Data	or compounds 2a 1, 3, 4a u
Compound	δ ¹ H	δ ¹³ C, J (Hz)
2a	8.29 (NH, s), 6.85 (H4, s), 2.38 (H6 s), 7.67, 7.22 (H phonyl)	161.2 (C3), 108.4 (C4), 153.7 (C5), 11.4 (C6), 163.0 (C7), 136.6–121.2
	(H6, s), 7.67–7.22 (H phenyl)	(C phenyl). $(C7)$, $150.0-121.2$
2b	9.48 (NH, s), 6.90 (H4, s), 2.35	160.5 (C3), 107.0 (C4), 155.8
	(H6, s), 4.38 (CH ₂ , s), 7.41–7.22	(C5), 11.0 (C6), 163.0 (C7), 138.7-
	(H phenyl)	126.9 (C phenyl), 42.2 (CH ₂).
2c	6.30 (NH, s), 6.70 (H4, s), 2.35	160.9 (C3), 107.3 (C4), 155.3 (C5),
	(H6, s), 1.82 (CH2, q), 1.43	11.4(C6), 163.8(C7), 55.1(CH28),
	(2CH3, s), 0.90 (CH3, t)	32.9 (CH29), 26.3 (CH210), 8.3
		(CH311).
2d	3.50 (NH, s), 7.25 (H4, s), 2.35	160.6 (C3), 108.4 (C4), 154.1
	(H6, s), 6.69 (H4, s), 2.52 (Me5, s)	(C5), 11.1 (C6), 161.5 (C7), 169.9
		(C8), 97.0 (C9), 157.5 (C10), 12.1
•		(Me11).
2e	7.20 (H4, s), 2.20 (H6, s), 3.45	159.0 (C3), 108.2 (C4), 157.5 (C5),
	(Me, s), 7.40–7.22 (H phenyl)	11.0 (C6), 162.7 (C7), 142.6–126.7
26	0.49 (70) (114 -) (74) (114' -)	(C phenyl). $1(0.5)(C2) = 107.2 (C4)$
2f	9.48; 6.79 (H4, s), 6.74 (H4', s),	160.5 (C3), 160.7 (C3'), 107.3 (C4),
	2.31 (H6, s), 2.30 (H6', s), 3.07 (Me, s), 2.98 (Me', s), 3.44 (CH2,	106.9(C4'), 157.8(C5), 157.7(C5'), 10.8(C6), 162.2(C7), 25.8(M2)
	t), 3.37 (CH2', t), 1.31 (CH2, qui),	10.8 (C6), 163.3 (C7), 35.8 (Me), 33.0 (Me'), 49.5 (C8), 47.0 (C8'),
	1.20 (CH2', qui), 1.55 (CH2, sex),	29.9 (CH2), 28.3 (CH2'), 19.4
	0.90 (CH3, t)	(2CH3), 19.0 (2CH2'), 13.6 (CH3),
	0.90 (CH3, t)	(2CH3), 19.0 (2CH2), 15.0 (CH3), 13.4 (CH3').
3a	7.67 (H4, s), 2.47 (H6, s), 2.40	160.8 (C3), 115.6 (C4), 158.0
Ja	(H6', s), 8.47 (H10, s), 7.60 (H10',	(C5), 159.4 (C5'), 11.5 (C6), 160.4
	s), 7.01 (H11, s), 6.90 (H11', s),	(C7), 159.4 (C7'), 149.0 (C10),
	4.47 (H15, s), 4.45 (H15', s), 1.45	153.5 (C10'), 111.6 (C11), 108.0
	(H16, s)	(C11'), 131.2 (C12), 132.5 (C12'),
	(, -)	161.0 (C13), 161.5 (C13'), 61.8
		(C15), 61.0 (C15'), 14.2 (C16).
3b	7.51 (H4, s), 2.43 (H6, s), 2.39	160.7 (C3), 115.1 (C4), 155.4
	(H6', s), 2.70 (Me, s), 6.71 (H11,	(C5), 11.2 (C6), 159.4 (C7), 158.8
	s), 6.60 (H11', s), 4.43 (H15, s),	(C7'), 14.0 (Me), 147.0 (C10),
	4.35 (H15', s), 1.41 (H16, s), 1.30	146.4 (C10'), 111.6 (C11), 107.4
	(H16′, s)	(C11'), 140.9 (C12), 143.0 (C12'),
		161.0 (C13), 161.2 (C13'), 61.5
		(C15), 61.0 (C15'), 14.1 (C16).
		(), •••• (••••), •••• (••••).

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Table 2. Continued

Compound	$\delta^{1}H$	δ ¹³ C, J (Hz)
3c	7.53 (H4, s), 2.44 (H6, s), 2.38 (H6', s), 3.71 (CH2, q), 1.40 (CH3, t), 6.75 (H11, s), 6.60 (H11', s), 4.43 (H15, s), 4.35 (H15', s), 1.41 (H16, s), 1.30	161.7 (C3), 115.0 (C4), 156.8 (C5), 11.3 (C6), 159.5 (C7), 19.2 (CH2), 13.1 (CH3), 149.0 (C10), 105.8 (C11), 141.3 (C12), 160.3 (C13), 60.9
3d	(H16', s) 7.67 (H4, s), 2.40 (H6, s), 2.30 (H6', s), 7.76–7.40 (H phenyl), 7.20 (H11, s), 7.03 (H11', s), 4.39 (H15, s), 4.31 (H15', s), 1.36 (H16, s), 1.32 (H16', s)	(C12), 14.1 (C16). 160.0 (C3), 110.3 (C4), 159.4 (C5), 11.4 (C6), 160.0 (C7), 129.1–126.0 (C phenyl), 148.0 (C10), 105 (C11), 140.0 (C12), 160.0 (C13), 61.4 (C15), 13.9 (C16).
4a	7.60 (H4, s), 2.49 (H6, s), 2.37 (H6', s), 8.53 (H10, s), 7.76 (H10', s), 6.83 (H11, s), 6.70 (H11', s)	$\begin{array}{c} (C10).\\ 160.1 & (C3), \ 115.6 & (C4), \ 153.1 \\ (C5), \ 11.4 & (C6), \ 157.7 & (C7), \\ 131.8 & (C10), \ 130.5 & (C10'), \\ 104.4 & (C11), \ 108.0 & (C11'), \\ 147.0 & (C12 \ J=39.3), \ 120.0 \\ (C13 \ J=270.1). \end{array}$
4b	7.43 (H4, s), 2.45 (H6, s), 2.34 (H6', s), 2.73 (Me, s)	160.6 (C3), 115.1 (C4), 155.2 (C5), 11.4 (C6), 159.3 (C7), 14.3 (Me), 147.2 (C10), 143.0 (C10'), 108.1 (C11), 102.9 (C11'), 146.0 (C12 $J=39.1$), 118.0 (C13).
4c	7.43 (H4, s), 2.45 (H6, s), 2.47 (H6', s), 3.16 (CH2, q), 2.75 (CH2', q), 1.35 (CH3, t), 1.32 (CH3', t)	160.8 (C3), 115.0 (C4), 155.1 (C5), 11.3 (C6), 158.8 (C7), 21.6 (CH2), 18.6 (CH2'), 11.9 (CH3), 12.7 (CH3'), 153.6 (C10), 148.1 (C10'), 106.3 (C11), 104.3 (C11'), 145.0 (C12 $J=39.0$), 118.0 (C13 $J=270.0$).
4d	7.54 (H4, s), 2.46 (H6, s), 2.34 (H6', s), 7.06 (H11, s), 6.97 (H11', s), 7.85–7.54 (H phenyl)	J = 270.0). 162.0 (C3), 116.0 (C4), 155.5 (C5), 11.5 (C6), 159.7 (C7), 131.2–127.6 (C phenyl), 154.0 (C10), 113.8 (C11), 136.3 (C12), 120.0 (C13).

^{a 1}H and ¹³C NMR spectra were recorded on a Bruker DPX 400, at 298 K, 0.5 M in chloroform- d_1 /TMS. For compound **2f** the data labeled as (') refers to *E*-isomer (R² is syn to carbonyl oxygen in the carboxamide group) and for 3,4 the data refers to 1,5-isomer (see scheme).



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MARTINS ET AL.

hexane/ethyl acetate. Yields and selected physical and spectroscopic data are presented in Tables 1 and 2.

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