

TRIFLUOROACETIC ACID CATALYZED ONE-POT FOUR-COMPONENT DOMINO REACTION FOR THE SYNTHESIS OF SUBSTITUTED DIHYDRO 2-OXYPYRROLES

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ABSTRACT

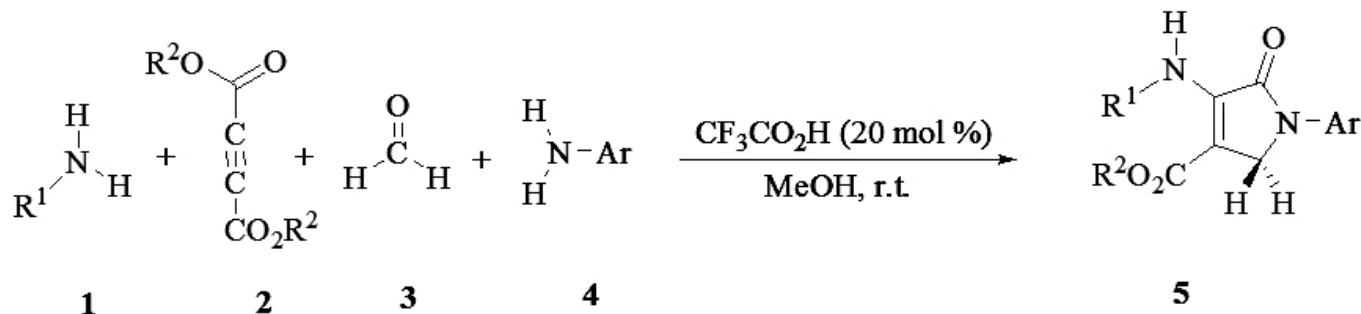
Trifluoroacetic acid was applied as an efficient catalyst for the one-pot four-component synthesis of N-aryl/alkyl-3-aminodihydropyrrol-2-one-4-carboxylates via the domino reaction of amines, formaldehyde and dialkyl acetylenedicarboxylates at ambient temperature in methanol. This methodology includes number of advantages such as: short reaction time, clean work-up, use of inexpensive catalyst, high yields and clean work-up. The work-up of this reaction involves only a filtration and a simple washing step with MeOH, and there is no need for column chromatography.

Keywords: Trifluoroacetic acid; N-aryl/alkyl-3-aminodihydropyrrol-2-one-4-carboxylates; ambient temperature

INTRODUCTION

In recent years, growing attention has been paid to the synthesis of N-heterocycles due to diverse biological and pharmaceutical applications [1-3]. In this respect, the presence of pyrrol-2-ones (5-lactams or γ -lactams) in pharmaceuticals and natural products has continued to stimulate a great deal of interest in the development of new methodologies for their synthesis [4-6]. There are several bioactive natural molecules with a pyrrol-2-one moiety, such as holomycin and thiolutin [7], thiomarinol A4 [8], oteromycin [9], pyrrocidine A [10], quinolactacin C [11], and ypaamide [12]. On the other hand, dihydropyrrol-2-ones have been successfully used as peptidomimetic [13], HIV integrase [14], herbicidal [15], DNA polymerase inhibitors [16], caspase-3 inhibitors [17] cytotoxic and antitumor agents [18], antibiotics [19], and also inhibitors of the annexin A2-S100A10 protein interaction [20]. Recently, a few methods have been reported for the synthesis of highly

substituted dihydropyrrol-2-ones using one-pot, four-component reactions in the presence of catalyst, such as AcOH [21], I₂ [22], benzoic acid [23], TiO₂ nanopowder [24] or Cu(OAc)₂·H₂O [25]. However, some of these methods have drawbacks, such as high temperature and utilize a chlorinated solvent. Therefore, the development of a milder and more efficient route for one-pot synthesis of these important heterocycles is still in demand. In continue of our ongoing program on multi-component reactions [26-31], an efficient and convenient synthesis of N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates has been accomplished using trifluoroacetic acid as an efficient catalyst in MeOH at ambient temperature, with good yields (Schemes 1). Trifluoroacetic acid is widely utilized in organic synthesis as a solvent [32] or as an acid catalyst for different organic transformations such as rearrangements [33], functional group deprotections [34], reductions [35], oxidations [36], hydroarylations [37], condensations [38], and also trifluoromethylation reactions [39].



Scheme 1. Synthesis of N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates in the presence of trifluoroacetic acid as catalyst in MeOH at ambient temperature

RESULTS AND DISCUSSION

Formaldehyde, aniline, and dimethyl acetylenedicarboxylate were taken as model compounds for the optimization of the reaction conditions. For this purpose, the reaction was initially carried out in different conditions (Table 1). As can be seen, trifluoroacetic acid (10 mol %) was found to be the most effective catalyst for the reaction at room temperature.

Table 1. Optimization of the reaction conditions for the synthesis of 5a ^a

Entry	Catalyst (mol%)	Solvent	Time (h)	Isolated yields (%)
1	TiO ₂	MeOH	15	25
2	Zn(SO ₄) ₂ ·7H ₂ O	MeOH	15	25
3	Zr(NO ₃) ₄	MeOH	12	30
5	HClO ₄ -SiO ₂	MeOH	12	20
6	KHSO ₄	MeOH	15	26
7	NH ₄ HSO ₄	MeOH	15	40
8	Trifluoroacetic acid (5 mol %)	MeOH	2.5	74
9	Trifluoroacetic acid (10 mol %)	MeOH	2	74
10	Trifluoroacetic acid (15 mol %)	MeOH	2	83
11	Trifluoroacetic acid (20 mol %)	MeOH	1.5	93
12	Trifluoroacetic acid (30 mol %)	MeOH	1.5	81

^a Reaction conditions: aniline (2.0 mmol), DMAD (1.0 mmol), and formaldehyde (1.5 mmol) at room temperature in the presence of catalyst

To demonstrate the utility and generality of this method, the various substituted anilines, dimethyl and/or diethyl acetylenedicarboxylate and formaldehyde were employed successfully to generate the desired N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates **5a-h** (Table 2). Encouraged by these results, different poly functionalized dihydropyrrol-2-ones **5i-q** were

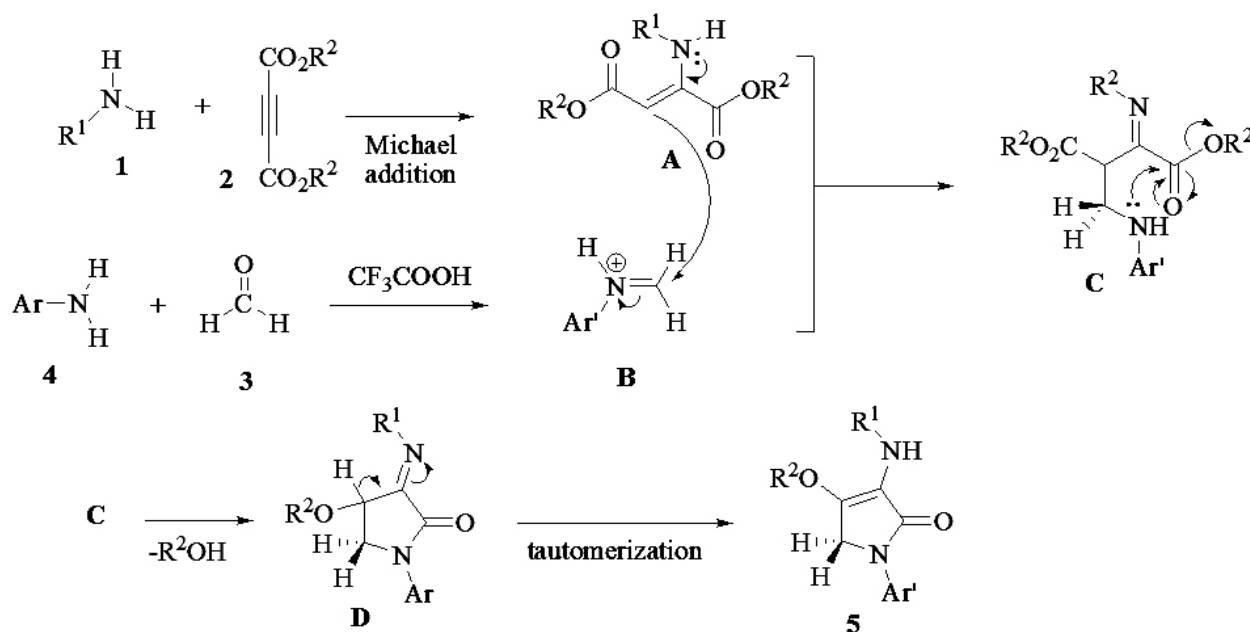
synthesized using two different amines. Aliphatic amines, such as benzyl amine, 1-(pyridin-2-yl)methanamine and *n*-butyl amine, were reacted with dialkyl acetylenedicarboxylates, anilines and formaldehyde to produce the corresponding products in good to high yields.

Table 2. Synthesis of N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates **5**.

Entry	Product	R ¹	R ²	Ar	Time (h)	Yield (%) ^a	m.p (°C)	Lit.mp (°C) [Ref]
1	5a	Ph	Me	Ph	5	91	156–157	155–156 [22]
2	5b	4-Me-C ₆ H ₄	Me	4-Me-C ₆ H ₄	6	85	177–179	177–178 [22]
34	5c	Ph	Et	Ph	5	89	138–140	138–140 [21]
5	5d	4-Me-C ₆ H ₄	Et	4-Me-C ₆ H ₄	7	85	128–130	131–132 [21]
6	5e	4-OMe-C ₆ H ₄	Et	4-OMe-C ₆ H ₄	7	92	152–154	152–154 [31]
7	5f	4-Br-C ₆ H ₄	Et	4-Br-C ₆ H ₄	7	91	171–172	169–171 [21]
8	5g	4-F-C ₆ H ₄	Me	4-F-C ₆ H ₄	6	78	163–165	163–165 [30]
9	5h	4-Cl-C ₆ H ₄	Et	4-Cl-C ₆ H ₄	6	85	167–170	168–170 [30]
10	5i	PhCH ₂	Me	Ph	7	84	140–141	140–141 [21]
11	5j	PhCH ₂	Me	4-F-C ₆ H ₄	7	85	168–170	166–168 [31]
12	5k	PhCH ₂	Et	Ph	8	77	127–129	129 130 [21]
13	5l	PhCH ₂	Me	4-Br-C ₆ H ₄	12	76	119–121	120–121 [22]
14	5m	PhCH ₂	Me	4-Me-C ₆ H ₄	6	85	144–146	144–146 [30]
15	5n	C ₅ H ₄ N ₂ -CH ₂	Me	4-Me-C ₆ H ₄	6	80	104–106	106–108 [29]
16	5o	<i>n</i> -C ₄ H ₉	Me	Ph	6	80	60–62	60 [22]
17	5p	<i>n</i> -C ₄ H ₉	Et	4-Br-C ₆ H ₄	6	85	94–97	94–96 [30]
18	5q	4-F-C ₆ H ₄	Et	4-F-C ₆ H ₄	6	89	170–172	172–173[21]
19	5r	4-Cl-C ₆ H ₄	Et	Me-C ₆ H ₄	5	93	169–170	168–170[30]
20	5s	4-Cl-C ₆ H ₄	Me	4-Cl-C ₆ H ₄	6	90	174–175	173–174[22]
21	5t	PhCH ₂	Me	3,4-Cl ₂ -C ₆ H ₃	8	89	165–167	162–164[40]
22	5u	PhCH ₂	Me	4-Cl-C ₆ H ₄	6	93	143–146	147–147[22]
23	5v	PhCH ₂	Me	4-OMe-C ₆ H ₄	8	84	123–126	129–130[22]
24	5w	4-Br-C ₆ H ₄	Me	4-Br-C ₆ H ₄	6	90	176–179	179–180 [22]
25	5x	4-Br-C ₆ H ₄	Me	4-Cl-C ₆ H ₄	6	89	157–160	160–162 [41]
26	5y	Ph	Et	4-Cl-C ₆ H ₄	7	84	199	202 [42]

^a Isolated Yield

base on the literature, this synthetic method can be combination of Michael, formation of imine, Mannich-type and cyclization reactions (Scheme 3).



Scheme 3. Proposed mechanism for the synthesis of N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates **5**

To compare the efficiency and applicability of $\text{CF}_3\text{CO}_2\text{H}$ with the reported catalysts and conditions in the literature for the synthesis of N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates, we have tabulated the results of these catalysts in Table 3. As shown in Table 3, $\text{CF}_3\text{CO}_2\text{H}$ can act as efficient catalyst with respect to reaction times and yields of products.

Table 3. Comparison result of $\text{CF}_3\text{CO}_2\text{H}$ with the reported catalysts in literature for the synthesis of N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates **5c**.

Entry	Catalyst	Conditions	Time (h)	Yield (%)	Ref.
1	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	MeOH, rt	5	85	25
2	I_2	MeOH, rt	1	81	22
3	AcOH	EtOH/ 70 °C	4	85	21
4	Oxalic acid dehydrate	MeOH, rt	2	87	43
5	$[\text{Hpyro}][\text{HSO}_4]$	MeOH, rt	6	80	31
6	$\text{Al}(\text{H}_2\text{PO}_4)_3$	MeOH, rt	5	80	29
7	InCl_3	MeOH, rt	3	85	44
8	$[\text{n-Bu}_4\text{N}][\text{HSO}_4]$	MeOH, rt	4	86	30
9	$\text{CF}_3\text{CO}_2\text{H}$	MeOH, rt	5	89	This work

EXPERIMENTAL

Melting points were taken on an Electrothermal 9100 apparatus. IR spectra were obtained on a JASCO FT/IR-460 plus spectrometer. The ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker DRX-400 Avance instrument with CDCl_3 as solvent and using TMS as internal reference at 400 MHz and 100 MHz, respectively. Chemicals were purchased from Merck (Darmstadt, Germany), Acros (Geel, Belgium) and Fluka (Buchs, Switzerland), and used without further purification.

General procedure for the synthesis of N-aryl-3-aminodihydropyrrol-2-one-4-carboxylates 5

A mixture of amine **1** (1.0 mmol) and dialkyl acetylenedicarboxylate **2** (1.0 mmol) in MeOH (3 mL) was stirred for 25 min. Next, amine **3** (1.0 mmol), formaldehyde **4** (37% solution, 1.5 mmol) and trifluoroacetic acid (20 mol %) were added in successively. The reaction mixture was allowed to stir at ambient temperature for appropriate time. After completion of the reaction (monitored by TLC), the water was added to produce solid precipitate, and the precipitate was filtered off and washed with methanol (3×2 mL) to give the pure product **5**. The structures of the synthesized compounds were characterized by their IR, ^1H NMR and ^{13}C NMR spectra and were found to be identical with data

described in the literature [21,22].

Characterization data of some compounds

Methyl 2,5-dihydro-5-oxo-1-phenyl-4-(phenylamino)-1H-pyrrole-3-carboxylate (5a)

White solid, IR (KBr) (δ max, cm^{-1}): 3310 (NH), 1705, 1684, 1645; ^1H NMR (400 MHz, CDCl_3): δ 3.76 (3H, s, OCH_3), 4.57 (2H, s, CH_2), 7.16-7.23 (4H, m, ArH), 7.34 (2H, t, $J = 8.0$ Hz, ArH), 7.42 (2H, t, $J = 8.0$ Hz, ArH), 7.81 (2H, d, $J = 8.0$ Hz, ArH), 8.05 (1H, br s, NH).

Ethyl 4-(p-tolylamino)-2,5-dihydro-5-oxo-1-p-tolyl-1H-pyrrole-3-carboxylate (5d)

Yellow solid, IR (KBr) (δ max, cm^{-1}): 3310 (NH), 1707, 1682, 1649; ^1H NMR (400 MHz, CDCl_3): δ 1.25 (3H, t, $J = 7.2$ Hz, OCH_2CH_3), 2.36 (3H, s, CH_3), 2.37 (3H, s, CH_3), 4.23 (2H, t, $J = 7.2$ Hz, OCH_2CH_3), 4.52 (2H, s, CH_2), 7.06 (2H, d, $J = 8.4$ Hz, ArH), 7.14 (2H, d, $J = 8.0$ Hz, ArH), 7.21 (2H, d, $J = 8.4$ Hz, ArH), 7.69 (2H, d, $J = 8.8$ Hz, ArH), 8.01 (1H, brs, NH); ^{13}C NMR (100 MHz, CDCl_3): δ 14.2, 20.9, 21.0, 48.3, 60.2, 102.4, 119.1, 122.9, 128.9, 129.6, 134.2, 134.6, 136.2, 136.3, 143.1, 163.7, 164.7.

Methyl 4-(benzylamino)-1-p-tolyl-2,5-dihydro-5-oxo-1H-pyrrole-3-carboxylate (5m)

White solid IR (KBr) (δ max, cm^{-1}): 3310 (NH), 1704, 1682, 1646; ^1H NMR (400 MHz, CDCl_3): δ 1.34 (3H, t, $J = 7.2$ Hz, OCH_2CH_3), 4.27 (2H, t, J

= 7.2 Hz, OCH₂CH₃), 4.41 (2H, s, CH₂-N), 5.12 (2H, d, *J* = 6.4 Hz, CH₂-NH), 6.90 (1H, br, NH), 7.28-7.37 (5H, m, ArH), 7.52 (2H, d, *J* = 8.8 Hz, ArH), 7.70 (2H, d, *J* = 8.8 Hz, ArH); ¹³C NMR (100 MHz, CDCl₃): δ 20.9, 46.6, 48.0, 51.0, 97.1, 119.4, 127.3, 127.5, 128.7, 129.6, 134.8, 136.2, 139.5, 164.3, 165.6.

CONCLUSION

In conclusion we have identified an efficient and simple one-pot reaction for the synthesis of N-aryl-3-aminodihydropyrrrol-2-one-4-carboxylates derivatives using trifluoroacetic acid as an economic catalyst at ambient conditions. This methodology has several advantages such as simplified workup procedures, mild conditions, and high yields

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