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Synthesis of functionalized 2-pyrrolidinones via domino reactions of arylamines, ethyl glyoxylate and acetylenedicarboxylates

Hong Gao, Jing Sun, Chao-Guo Yan *

College of Chemistry & Chemical Engineering, Yangzhou University, Yangzhou 225002, China

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ABSTRACT

An efficient and practical synthetic method for the functionalized 2-pyrrolidinones was successfully developed via the domino reactions of ethyl glyoxylate and acetylenedicarboxylate with 2 M arylamines in the presence of benzoic acid as catalyst. When two kinds of amines were used in the reaction, the stronger nucleophilic aliphatic amine preferentially added to acetylenedicarboxylate to form the reactive intermediate β-enamino ester and the reaction showed high regioselectivity.

1. Introduction

Pyrrole and its derivatives are the important structural motifs that are widely present in numerous natural products, synthetic pharmaceuticals and molecular metarials.[1,2] As a consequence, versatile synthetic methods have been developed for the synthesis of these privileged molecules.[3,4] Recently, The multicomponent reactions containing active Huisgen's *1,4*-dipoles, which were generated in situ from the addition of nucleophiles to electron-deficient alkynes, have become one of the efficient methods for preparation diverse substituted pyrroles.[5–8] As for some examples, Huisgen's *1,4*-dipoles derived from additions of alkyl(aryl) isocyanides to dimethyl acetylenedicarboxylate reacted with succinimide,[9] cyclobutene-1,2-diones,[10] benzoyl chloride[11] all resulted in functionalized pyrroles. Triarylphosphine induced domino reactions of acetylenedicarboxylates with some unsaturated compounds[12–15] and tertiary amines as well as other catalyst assisted domino reactions[16–20] are also efficient synthetic methods for the functionalized pyrroles. Very recently, Jiang reported three-component reactions of aromatic aldehydes, arylamines and acetylenedicarboxylates to give 3-arylamino-2-pyrrolidinones.[21] We also found that this three-component reaction could afford novel 3-hydroxyl or 3-arylamino-2-pyrrolidinones and 2-hydroxytetrahydropyridines under different condition.[22] In order to establish the scope and limitation of this reaction and develop efficient routes for the preparation of biologically active heterocyclic compounds,[23] here we wish to report the results of the one-pot domino reaction of arylamines, acetylenedicarboxylates and ethyl glyoxylate.

2. Results and discussion

Initial studies were carried out by reactions of *p*-methylaniline, dimethyl acetylenedicarboxylate and ethyl glyoxylate in ethanol with *p*-toluenesulfonic acid as catalyst according to our previously established reaction condition.[22] After workup, we were satisfied to find that the expected 3-arylamino-2-pyrrolidinone **1b** (Table 1, entry 2) was obtained in 50% yield. When acetic acid was used as catalyst, the yield of **1b** was increased to 55% yield. But benzoic acid could afford **1b** in 70% yield. If increasing of amount of benzoic acid from 0.5 to 1.5 M, the yields of products were nearly same. Thus benzoic acid proved to be the catalyst of choice and it was adopted for all future studies. With the optimal conditions in hand, we then examined the scope of the one-pot three-component reaction. At first various arylamines were employed in the reaction (Table 1, entries 1–6). It can be seen that all of the reactions proceeded smoothly to afford the corresponding 2-pyrrolidinones **1a**–**f** in good yields. Arylamines with electron-donating methyl, methoxyl groups and weak electron-withdrawing chloro groups reacted efficiently to yield the desired products. Diethyl acetylenedicarboxylate could also be successfully used in the reaction and the expected products **1g**–**j** were obtained in relatively lower yields (Table 1, entries 7–10). The structures of the functionalized 2-pyrrolidinones were fully characterized by ^{1}H and ^{13}C NMR, HRMS, IR spectra, and were further confirmed by single crystal X-

* Corresponding author. E-mail address: cgyan@yzu.edu.cn (C.-G. Yan).

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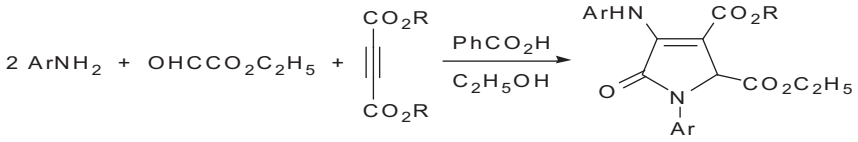
Table 1
Synthesis of the functionalized 2-pyrrolidinones **1a**–**k**[a]



Entry	Compd	Ar	R	Yield (%)[b]
1	**1a**	*p*-$CH_3OC_6H_4$	CH_3	72
2	**1b**	*p*-$CH_3C_6H_4$	CH_3	70
3	**1c**	*m*-$CH_3C_6H_4$	CH_3	50
4	**1d**	C_6H_5	CH_3	64
5	**1e**	*p*-ClC_6H_4	CH_3	55
6	**1f**	*m*-ClC_6H_4	CH_3	58
7	**1g**	*p*-$CH_3OC_6H_4$	C_2H_5	70
8	**1h**	*p*-$CH_3C_6H_4$	C_2H_5	45
9	**1i**	*p*-ClC_6H_4	C_2H_5	70
10	**1j**	*m*-ClC_6H_4	C_2H_5	47

[a] Reaction conditions: amine (4.0 mmol), alkyne (2.0 mmol), ethyl glyoxylate (2.0 mmol) benzoic acid (0.5 mmol) in ethanol (10 mL) at rt for 48 h.
[b] Isolated yield.

ray diffraction studies performed on compounds **1a** and **1g** (Figs. 1 and 2).

To explain the mechanism of this one-pot three-component reaction, a plausible reaction course was proposed based on the previously reported reactions,[21,22] which is illustrated in Scheme 1. The first reaction is the formation of active β-enamino ester (**A**) by the addition of arylamine to acetylenedicarboxylate. The second reaction is the formation of imine (**B**) from the condensation of arylamine with ethyl glyoxylate with the catalysis of *p*-toluenesulfonic acid. Thirdly Michael addition of β-enamino ester (**A**) to imine (**B**) gave the intermediate (**C**). Then the intramolecular nucleophilic addition of amino group to carbonyl group in adduct (**B**) produced a cyclic intermediate (**C**). At last the intramolecular reaction of arylamino group to carbonyl group in ester moiety in intermediate (**C**) with the elimination of methanol gave the final polysubstituted 3-arylamino-2-pyrrolidinones **1**. In this proposed reaction mechanism 2 M anilines reacted with other two substrates to form the β-enamino ester (**A**) and the imine (**B**) intermediates.

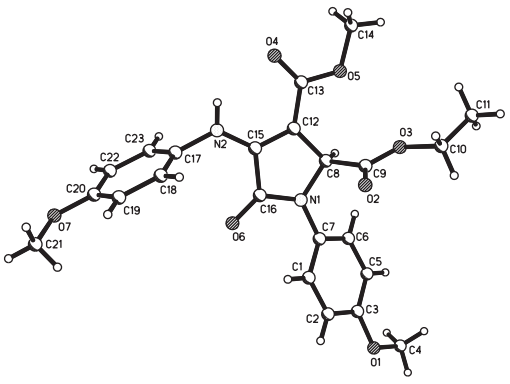


Fig. 1. Molecular structure of 2-pyrrolidinone **1a**.

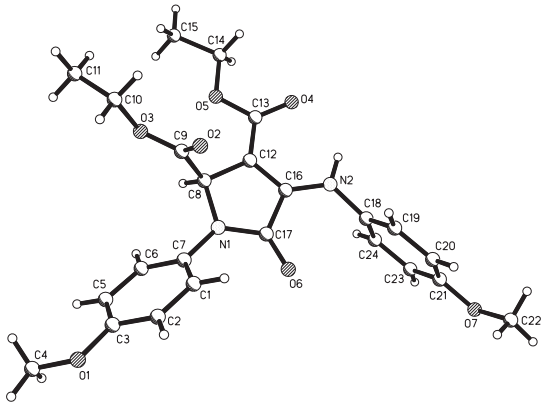
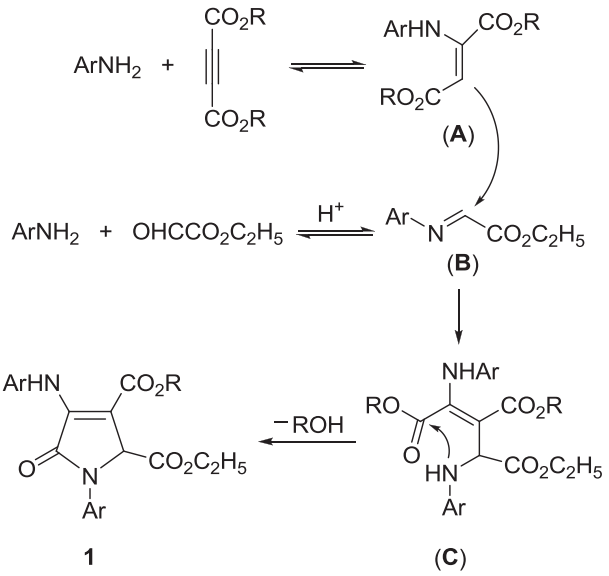


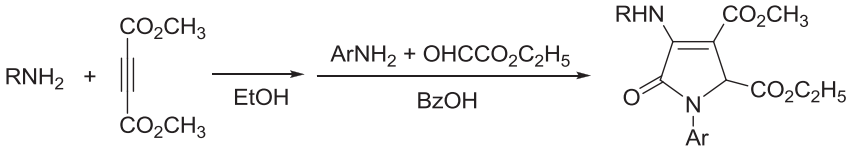
Fig. 2. Molecular structure of 2-pyrrolidinone **1g**.



Scheme 1. Formation mechanism of 2-pyrrolidinone.

Having established an efficient route to the polysubstituted 2-pyrrolidinones, we next turned our attention to examine the reactions containing two different kinds of amines. At first one arylamine and one aliphatic amine, such as benzylamine were introduced in the reaction with a one-pot domino procedure. Thus benzylamine firstly reacted with dimethyl acetylenedicarboxylate in ethanol at room temperature for 10 min for the formation of the active intermediate β-enamino ester. Then one molar arylamine and ethyl glyoxylate as well as benzoic acid was added. The reaction proceeded smoothly at room temperature for 24 h to give the desired 2-pyrrolidinones **2a**–**g** in good yield (Table 2, entries 1–7).

Table 2
Synthesis of the polysubstituted 2-pyrrolidinones **2a**–**o**[a]



Entry	Compd	R	Ar	Yield (%)[b]
1	**2a**	$CH_2C_6H_5$	p-$CH_3C_6H_4$	75
2	**2b**	$CH_2C_6H_5$	p-$CH_3OC_6H_4$	65
3	**2c**	$CH_2C_6H_5$	C_6H_5	50
4	**2d**	$CH_2C_6H_5$	p-ClC_6H_4	45
5	**2e**	$CH_2C_6H_5$	p-BrC_6H_4	40
6	**2f**	$CH_2C_6H_5$	p-$CH_3C_6H_4$	55
7	**2g**	$CH_2C_6H_5$	p-$CH_3OC_6H_4$	70
8	**2h**	$CH_2CH_2C_6H_5$	p-$CH_3C_6H_4$	66
9	**2i**	$CH_2CH_2C_6H_5$	p-$CH_3OC_6H_4$	58
10	**2j**	$CH_2CH_2C_6H_5$	p-ClC_6H_4	43
11	**2k**	$CH(CH_3)C_6H_5$	p-$CH_3C_6H_4$	55
12	**2l**	$CH(CH_3)C_6H_5$	p-$CH_3OC_6H_4$	60
13	**2m**	$CH(CH_3)C_6H_5$	p-ClC_6H_4	35
14	**2n**	p-ClC_6H_4	p-$CH_3C_6H_4$	40
15	**2o**	p-$CH_3OC_6H_4$	p-ClC_6H_4	60

[a] Reaction conditions: amine (2.0 mmol), alkyne (2.0 mmol) in ethanol at rt for 10 min; then ethyl glyoxylate (2.0 mmol), arylamine (2.0 mmol), benzoic acid (0.5 mmol) for 48 h.
[b] Isolated yield.

Then the similar reactions containing 2-phenylethylamine or 1-phenylethylamine also successfully resulted in the expected 2-pyrrolidinones in moderate yields **2h**–**m** (Table 2, entries 8–13). If two arylamines having substituents with larger different electron effects were employed in the reaction by using above sequential procedure, the desired 2-pyrrolidinones **2n** and **2o** were also successfully prepared in lower yields (Table 2, entries 8–13). On other cases we found that a mixture of two kinds of polysubstituted 2-pyrrolidinones would be formed and this mixture is very difficult to separate and gave nearly same analytical data, which was clearly caused by the reversible equilibrium reaction of formations of β-enamino ester and arylimine and their sequential cross reactions.[24,25] Taken together, these one-pot sequential reactions provided fifteen new 2-pyrrolidinone derivatives, of which the compound **2d** was characterized by X-ray crystallography (Fig. 3).

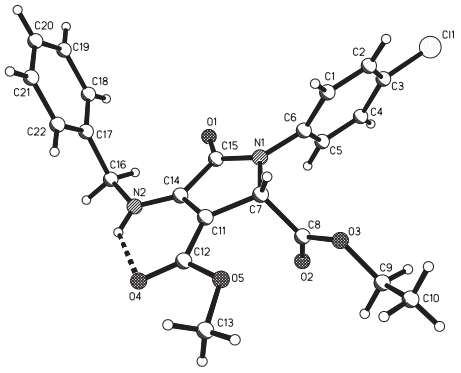


Fig. 3. Molecular structure of 2-pyrrolidinone **2d**.

In summary we were successfully developed a one-pot domino reaction of arylamine, ethyl gloxylate and acetylenedicarboxylate and provided a convenient procedure for the preparation of the functionalized 2-pyrrolidinones in good yields. Furthermore, by using one-pot sequential reaction procedure, two kinds of amines could be employed in the reaction to give more substituted 2-pyrrolidinones. We also proposed rational reaction mechanisms and established the scope and limitation of this reaction, which enabled further modification that led to molecular diversity. The potential uses of the reaction in synthetic and medicinal chemistry might be quite significant.

3. Experimental section

3.1. Typical procedure for the preparation of functionalized 2-pyrrolidinones 1a–j

A mixture of arylamine (4.0 mmol) and acetylenedicarboxylate (2.0 mmol) in 5.0 mL ethanol was stirred at room temperature for 10 min. Then ethyl glyoxylate (2.0 mmol, 50% soln in toluene, 0.45 g) and benzoic acid (0.5 mmol, 0.060 g) were added to it. The solution was stirred at room temperature for two days. The resulting precipitates were collected by filtration and washed with cold alcohol to give the pure product.

Compound **1a**: light yellow solid, 72%, mp 122–123 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 8.27 (s, 1H, NH), 7.44 (d, J=9.0 Hz, 2H, ArH), 7.13 (d, J=9.0 Hz, 2H, ArH), 6.88 (d, J=9.0 Hz, 2H, ArH), 6.85 (d, J=8.4 Hz, 2H, ArH), 5.32 (s, 1H, CH), 4.17–4.12 (m, 1H, CH), 4.10–4.05 (m, 1H, CH), 3.80 (s, 3H, OCH_3), 3.79 (s, 3H, OCH_3), 3.73 (s, 3H, CH_3), 1.11 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.6, 164.6, 163.6, 157.9, 157.5, 145.0, 130.9, 129.4, 125.5, 124.1, 114.2, 113.6, 100.5, 62.1, 61.8, 55.4, 51.3, 14.0; IR (KBr) ν: 3305, 2950, 2836, 2064, 1713, 1632, 1511, 1462, 1382, 1302, 1243, 1186, 1109, 1029, 875, 830, 761 cm^{-1}; MS (m/z): 903.09 ([2M+Na]$^+$) 100%. HRMS (ESI) calcd for $C_{23}H_{23}N_2O_7$ ([M−H]$^+$): 439.1511. Found: 439.1512.

Compound **1b**: light yellow solid, 70%, mp 139–141 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 8.27 (s, 1H, NH), 7.45 (d, J=9.0 Hz, 2H, ArH), 7.16 (d, J=8.4 Hz, 2H, ArH), 7.12 (d, J=8.4 Hz, 2H, ArH), 7.07 (d, J=8.4 Hz, 2H, ArH), 5.38 (s, 1H, CH), 4.17–4.05 (m, 2H, CH_2), 3.73 (s, 3H, CH_3), 2.34 (s, 3H, CH_3), 2.32 (s, 3H, CH_3), 1.11 (t, J=6.9 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.5, 164.6, 163.5, 136.1, 135.2, 135.2, 134.0, 129.7, 129.0, 123.7, 121.9, 101.3, 61.9, 61.8, 51.7, 21.0, 20.9, 14.0; IR (KBr) ν: 3247, 2969, 1744, 1695, 1520, 1445, 1366, 1249, 1199, 1134,

1026, 938, 817 cm^{-1}; MS (*m*/*z*): 839.20 ($[2M+Na]^+$) 100%. HRMS (ESI) calcd for $C_{23}H_{23}N_2O_5$ ($[M-H]^+$): 407.1612. Found: 407.1612.

Compound **1c**: light yellow solid, 50%, mp 106–109 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.24 (s, 1H, NH), 7.46 (s, 1H, ArH), 7.34 (d, *J*=8.4 Hz, 1H, ArH), 7.24–7.19 (m, 2H, ArH), 7.01 (t, *J*=9.6 Hz, 4H, ArH), 5.40 (s, 1H, CH), 4.14–4.10 (m, 2H, CH_2), 3.72 (s, 3H, CH_3), 2.34 (s, 6H, CH_3), 1.11 (t, *J*=8.4 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.5, 164.5, 163.6, 144.5, 139.1, 138.3, 137.8, 136.5, 128.9, 128.2, 127.1, 126.2, 124.2, 122.7, 120.8, 118.9, 102.0, 61.9, 61.8, 51.4, 21.5, 21.4, 14.0; IR (KBr) ν: 3234, 3080, 2968, 1739, 1694, 1651, 1594, 1549, 1492, 1447, 1395, 1357, 1276, 1199, 1024, 896, 786 cm^{-1}; MS (*m*/*z*): 407.29 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{23}H_{23}N_2O_5$ ($[M-H]^+$): 407.1612. Found: 407.1613.

Compound **1d**: light yellow solid, 64%, mp 109–111 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.28 (s, 1H, NH), 7.59 (d, *J*=2.7 Hz, 2H, ArH), 7.35 (d, *J*=15.0 Hz, 4H, ArH), 7.19 (d, *J*=2.4 Hz, 4H, ArH), 5.42 (s, 1H, CH), 4.14–4.09 (m, 2H, CH_2), 3.71 (s, 3H, CH_3), 1.10 (s, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.4, 164.5, 163.7, 144.3, 138.0, 136.6, 129.2, 128.4, 126.3, 125.4, 123.5, 121.9, 102.2, 62.0, 61.8, 58.4, 51.4, 18.4, 14.0, 13.9; IR (KBr) ν: 3289, 3063, 2986, 1741, 1717, 1672, 1639, 1594, 1498, 1470, 1452, 1370, 1300, 1251, 1230, 1201, 1119, 1019, 919, 817, 763 cm^{-1}; MS (*m*/*z*): 379.31 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{21}H_{19}N_2O_5$ ($[M-H]^+$): 379.1299. Found: 379.1301.

Compound **1e**: white solid, 55%, mp 164–165 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.30 (s, 1H, NH), 7.55 (d, *J*=8.4 Hz, 2H, ArH), 7.35 (d, *J*=8.4 Hz, 2H, ArH), 7.29 (d, *J*=9.0 Hz, 2H, ArH), 7.11 (d, *J*=9.0 Hz, 2H, ArH), 5.38 (s, 1H, CH), 4.19–4.16 (m, 1H, CH), 4.12–4.09 (m, 1H, CH), 3.76 (s, 3H, CH_3), 1.13 (t, *J*=6.9 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.0, 164.3, 163.4, 144.1, 136.3, 135.1, 131.7, 130.8, 129.3, 128.6, 124.9, 122.8, 102.9, 62.2, 61.5, 51.6, 14.0; IR (KBr) ν: 3243, 3121, 3070, 2992, 1743, 1698, 1652, 1592, 1542, 1452, 1363, 1291, 1211, 1095, 1019, 939, 882, 831 cm^{-1}; MS (*m*/*z*): 447.30 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{21}H_{17}Cl_2N_2O_5$ ($[M-H]^+$): 447.0520. Found: 447.0522.

Compound **1f**: white solid, 58%, mp 110–112 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.27 (s, 1H, NH), 7.72 (s, 1H, ArH), 7.50 (d, *J*=7.8 Hz, 1H, ArH), 7.31 (t, *J*=8.1 Hz, 1H, ArH), 7.24 (s, 1H, ArH), 7.20–7.17 (m, 3H, ArH), 7.07 (d, *J*=7.8 Hz, 1H, ArH), 5.40 (s, 1H, CH), 4.21–4.13 (m, 2H, CH_2), 3.77 (s, 3H, CH_3), 1.15 (t, *J*=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 167.9, 164.2, 163.4, 143.7, 139.0, 137.7, 135.0, 134.0, 130.2, 129.4, 126.3, 125.5, 123.6, 121.7, 121.6, 119.2, 103.7, 62.3, 61.5, 51.7, 14.0; IR (KBr) ν: 3390, 3242, 3120, 3073, 2977, 2388, 1961, 1735, 1699, 1652, 1590, 1541, 1480, 1441, 1393, 1355, 1266, 1210, 1021, 939, 889, 850, 789 cm^{-1}; MS (*m*/*z*): 447.25 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{21}H_{17}Cl_2N_2O_5$ ($[M-H]^+$): 447.0520. Found: 447.0517.

Compound **1g**: light yellow solid, 70%, mp 119–121 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.27 (s, 1H, NH), 7.44 (d, *J*=9.0 Hz, 2H, ArH), 7.13 (d, *J*=8.4 Hz, 2H, ArH), 6.88 (d, *J*=9.0 Hz, 2H, ArH), 6.85 (d, *J*=8.4 Hz, 2H, ArH), 5.33 (s, 1H, CH), 4.25–4.16 (m, 2H, CH_2), 4.12–4.08 (m, 2H, CH_2), 3.80 (s, 3H, OCH_3), 3.79 (s, 3H, OCH_3), 1.23 (t, *J*=6.9 Hz, 3H, CH_3), 1.12 (t, *J*=6.9 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.7, 164.4, 163.6, 157.9, 157.4, 144.9, 130.9, 129.4, 125.4, 124.2, 114.3, 113.6, 101.0, 62.2, 61.8, 60.3, 55.4, 14.2, 14.0; IR (KBr) ν: 3464, 3405, 3287, 2985, 2923, 2836, 2550, 2057, 1893, 1742, 1713, 1633, 1511, 1459, 1377, 1303, 1243, 1183, 1109, 1030, 946, 879, 832, 761 cm^{-1}; MS (*m*/*z*): 931.02 ($[2M+Na]^+$) 100%. HRMS (ESI) calcd for $C_{24}H_{25}N_2O_7$ ($[M-H]^+$): 453.1667. Found: 453.1663.

Compound **1h**: white solid, 45%, mp 144–146 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.25 (s, 1H, NH), 7.46 (d, *J*=8.4 Hz, 2H, ArH), 7.16 (d, *J*=7.8 Hz, 2H, ArH), 7.11 (d, *J*=7.8 Hz, 2H, ArH), 7.07 (d, *J*=8.4 Hz, 2H, ArH), 5.38 (s, 1H, CH), 4.24–4.15 (m, 2H, CH_2), 4.12–4.09 (m, 2H, CH_2), 2.33 (s, 3H, CH_3), 2.32 (s, 3H, CH_3), 1.22 (t, *J*=7.2 Hz, 3H, CH_3), 1.12 (t, *J*=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.6, 164.2, 163.7, 144.5, 136.0, 135.4, 135.0, 134.1, 129.6, 129.0, 123.5, 121.9, 101.8, 61.9, 60.4, 21.0, 20.9, 14.1, 14.0; IR (KBr) ν: 3464, 3414, 3337, 3286, 3045, 2987, 2920, 2867, 2591, 1905, 1718, 1641, 1514, 1462, 1402, 1363, 1290, 1245, 1183, 1111, 1027, 946, 888, 814, 761 cm^{-1}; MS (*m*/*z*): 867.06 ($[2M+Na]^+$) 100%. HRMS (ESI) calcd for $C_{24}H_{25}N_2O_5$ ($[M-H]^+$): 421.1769. Found: 421.1765.

Compound **1i**: light yellow solid, 70%, mp 119–121 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.30 (s, 1H, NH), 7.56 (d, *J*=8.4 Hz, 2H, ArH), 7.34 (d, *J*=8.4 Hz, 2H, ArH), 7.28 (d, *J*=9.0 Hz, 2H, ArH), 7.11 (d, *J*=8.4 Hz, 2H, ArH), 5.39 (s, 1H, CH), 4.26–4.18 (m, 2H, CH_2), 4.15–4.11 (m, 2H, CH_2), 1.25 (t, *J*=6.9 Hz, 3H, CH_3), 1.15 (t, *J*=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.1, 164.0, 163.5, 143.9, 136.4, 135.1, 131.6, 130.7, 129.3, 128.6, 124.7, 122.8, 103.5, 62.2, 61.6, 60.7, 14.1, 14.0; IR (KBr) ν: 3470, 3229, 3121, 3063, 2983, 1904, 1743, 1697, 1637, 1595, 1542, 1493, 1379, 1334, 1286, 1216, 1099, 1033, 889, 827, 772 cm^{-1}; MS (*m*/*z*): 461.27 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{22}H_{19}Cl_2N_2O_5$ ($[M-H]^+$): 461.0677. Found: 461.0672.

Compound **1j**: white solid, 47%, mp 99–101 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.28 (s, 1H, NH), 7.74 (s, 1H, ArH), 7.54 (t, *J*=8.1 Hz, 1H, ArH), 7.33 (t, *J*=8.1 Hz, 1H, ArH), 7.26 (d, *J*=7.8 Hz, 1H, ArH), 7.21 (t, *J*=3.9 Hz, 1H, ArH), 7.20–7.17 (m, 2H, ArH), 7.10–7.08 (m, 1H, ArH), 5.42 (s, 1H, CH), 4.28–4.15 (m, 4H, CH_2), 1.26 (t, *J*=6.9 Hz, 3H, CH_3), 1.18 (t, *J*=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.0, 163.9, 143.4, 139.1, 137.7, 135.0, 134.1, 130.2, 129.4, 126.3, 123.4, 121.6, 121.5, 119.3, 104.3, 62.3, 61.6, 60.8, 14.1, 14.0; IR (KBr) ν: 3464, 3238, 3121, 3070, 2982, 2913, 1743, 1697, 1636, 1590, 1541, 1480, 1393, 1333, 1296, 1212, 1120, 1034, 929, 872, 781 cm^{-1}; MS (*m*/*z*): 461.25 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{22}H_{19}Cl_2N_2O_5$ ($[M-H]^+$): 461.0677. Found: 461.0676.

3.2. Typical procedure for the preparation of functionalized 2-pyrrolidinones 2a–o

In a round bottom flask a mixture of aliphatic amine or arylamine (2.0 mmol) and acetylenedicarboxylate (2.0 mmol) in 5.0 mL ethanol was stirred at room temperature for 10 min. Then arylamine (2.0 mmol), ethyl glyoxylate (2.0 mmol, 50% soln in toluene, 0.45 g) and benzoic acid (0.5 mmol, 0.060 g) were added to it. The solution was stirred at room temperature for two days. The resulting precipitates were collected by filtration and washed with cold alcohol to give the pure product.

Compound **2a**: white solid, 75%, mp 100–102 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.43 (d, *J*=8.4 Hz, 2H, ArH), 7.34 (d, *J*=4.8 Hz, 4H, ArH), 7.29–7.26 (m, 1H, ArH), 7.18 (d, *J*=8.4 Hz, 2H, ArH), 5.29 (s, 1H, CH), 5.21 (s, 1H, CH), 5.05 (d, *J*=15.0 Hz, 1H, CH), 4.14–4.09 (m, 1H, CH), 4.07–4.01 (m, 1H, CH), 3.74 (s, 3H, CH_3), 2.33 (s, 3H, CH_3), 1.09 (t, *J*=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.9, 136.3, 134.0, 129.7, 128.7, 127.6, 127.5, 122.3, 61.7, 51.1, 21.0, 14.0; IR (KBr) ν: 3476, 3327, 3060, 2995, 2922, 2322, 1942, 1752, 1704, 1643, 1515, 1449, 1371, 1303, 1239, 1109, 1025, 970, 922, 816, 766 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{23}N_2O_5$ ($[M-H]^+$): 407.1612. Found: 407.1610.

Compound **2b**: yellow solid, 65%, mp 138–141 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.29 (s, 1H, NH), 7.56 (d, *J*=9.0 Hz, 2H, ArH), 7.32 (d, *J*=9.0 Hz, 2H, ArH), 7.13 (d, *J*=9.0 Hz, 2H, ArH), 6.86 (d, *J*=9.0 Hz, 2H, ArH), 5.36 (s, 1H, CH), 4.18–4.15 (m, 1H, CH), 4.11–4.08 (m, 1H, CH), 3.81 (s, 3H, OCH_3), 3.75 (s, 3H, CH_3), 1.13 (t, *J*=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.3, 164.7, 163.5, 157.7, 145.0, 135.3, 131.4, 130.6, 129.2, 125.8, 125.6, 124.2, 122.8, 114.3, 113.7, 113.6, 100.7, 62.1, 61.4, 58.5, 55.4, 51.5, 14.0; IR (KBr) ν: 3251, 3056, 2987, 2839, 1699, 1640, 1503, 1449, 1376, 1297, 1220, 1114, 1032, 937, 830, 779 cm^{-1}; MS (*m*/*z*): 443.36 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{22}H_{20}ClN_2O_6$ ($[M-H]^+$): 443.1015. Found: 443.1015.

Compound **2c**: white solid, 50%, mp 77–79 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.57 (d, *J*=7.8 Hz, 2H, ArH), 7.39 (t, *J*=8.4 Hz, 2H, ArH), 7.35 (d, *J*=4.8 Hz, 4H, ArH), 7.30–7.28 (m, 1H, ArH), 7.23 (t, *J*=7.8 Hz, 1H, ArH), 5.34 (s, 1H, CH), 5.24 (s, 1H, CH), 5.07–5.03 (m, 1H, CH), 4.15–4.10 (m, 1H, CH), 4.07–4.02 (m, 1H, CH), 3.75 (s, 3H,

CH_3), 1.08 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.7, 136.5, 129.1, 128.7, 127.6, 127.5, 126.3, 122.1, 61.8, 51.1, 14.0; IR (KBr) ν: 3465, 3356, 3074, 3026, 2941, 2046, 1956, 1743, 1680, 1618, 1497, 1448, 1371, 1291, 1231, 1122, 1021, 972, 899, 812, 757 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{21}N_2O_5$ ($[M-H]^+$): 393.1456. Found: 393.1456.

Compound **2d**: white solid, 45%, mp 111–113 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.55 (d, J=8.4 Hz, 2H, ArH), 7.35 (d, J=12.0 Hz, 6H, ArH), 7.29 (d, J=5.4 Hz, 1H, ArH), 5.30 (s, 1H, CH), 5.20 (d, J=2.4 Hz, 1H, CH), 5.06–5.03 (m, 1H, CH), 4.17–4.11 (m, 1H, CH), 4.09–4.04 (m, 1H, CH), 3.75 (s, 3H, CH_3), 1.11 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.6, 135.2, 131.6, 129.3, 128.8, 127.5, 123.1, 61.9, 51.2, 14.0; IR (KBr) ν: 3361, 3034, 2984, 2948, 1739, 1681, 1620, 1490, 1446, 1378, 1314, 1204, 1098, 1023, 970, 913, 849, 809 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{20}ClN_2O_5$ ($[M-H]^+$): 427.1066. Found: 427.1066.

Compound **2e**: white solid, 40%, mp 127–128 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.50 (s, 4H, ArH), 7.35 (d, J=6.6 Hz, 4H, ArH), 7.29 (d, J=6.0 Hz, 1H, ArH), 5.30 (s, 1H, CH), 5.19 (s, 1H, CH), 5.06–5.02 (m, 1H, CH), 4.16–4.12 (m, 1H, CH), 4.09–4.05 (m, 1H, CH), 3.75 (s, 3H, CH_3), 1.11 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.5, 135.7, 132.2, 128.8, 127.5, 123.3, 119.4, 62.0, 51.2, 14.0; IR (KBr) ν: 3458, 3360, 3032, 2982, 2948, 1927, 1739, 1681, 1619, 1487, 1446, 1378, 1315, 1203, 1116, 1061, 1020, 912, 845, 808 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{20}BrN_2O_5$ ($[M-H]^+$): 471.0197. Found: 471.0230.

Compound **2f**: white solid, 55%, mp 98–100 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.43 (d, J=8.4 Hz, 2H, ArH), 7.35–7.32 (m, 4H, ArH), 7.29–7.26 (m, 1H, ArH), 7.18 (d, J=8.4 Hz, 2H, ArH), 5.30 (s, 1H, CH), 5.21 (s, 1H, CH), 5.05 (t, J=10.2 Hz, 1H, CH), 4.24–4.17 (m, 2H, CH_2), 4.09–4.05 (m, 2H, CH_2), 2.33 (s, 3H, CH_3), 1.27 (t, J=7.2 Hz, 3H, CH_3), 1.10 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.9, 136.2, 134.0, 129.7, 128.7, 127.6, 127.5, 122.3, 61.7, 60.0, 21.0, 14.4, 14.0; IR (KBr) ν: 3473, 3312, 3053, 2983, 2917, 2322, 2087, 1753, 1704, 1645, 1513, 1450, 1368, 1302, 1237, 1108, 1028, 983, 921, 875, 813, 766 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{25}N_2O_5$ ($[M-H]^+$): 421.1769. Found: 421.1765.

Compound **2g**: light orange solid, 70%, mp 89–91 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.43 (d, J=8.4 Hz, 2H, ArH), 7.34 (t, J=6.6 Hz, 4H, ArH), 7.29–7.27 (m, 1H, ArH), 6.91 (d, J=8.4 Hz, 2H, ArH), 5.24 (d, J=19.2 Hz, 2H, CH_2), 5.06–5.02 (m, 1H, CH), 4.24–4.17 (m, 2H, CH_2), 4.09–4.06 (m, 2H, CH_2), 3.80 (s, 3H, CH_3O), 1.27 (t, J=7.2 Hz, 3H, CH_3), 1.11 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.9, 158.0, 129.3, 128.7, 127.6, 127.5, 124.5, 114.4, 61.7, 59.9, 55.4, 14.3, 14.0; IR (KBr) ν: 3452, 3340, 2981, 2934, 2839, 2048, 1736, 1704, 1684, 1620, 1515, 1450, 1382, 1301, 1246, 1196, 1117, 1085, 1030, 970, 812 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{25}N_2O_6$ ($[M-H]^+$): 437.1718. Found: 437.1715.

Compound **2h**: light yellow solid, 66%, mp 126–127 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.41 (d, J=7.8 Hz, 2H, ArH), 7.31 (t, J=7.8 Hz, 2H, ArH), 7.25 (d, J=4.2 Hz, 1H, ArH), 7.22 (t, J=7.2 Hz, 2H, ArH), 7.18 (d, J=8.4 Hz, 2H, ArH), 5.26 (s, 1H, CH), 4.16–4.09 (m, 2H, CH_2), 4.07–4.01 (m, 2H, CH_2), 3.73 (s, 3H, CH_3), 2.93 (t, J=7.8 Hz, 2H, CH_2), 2.33 (s, 3H, CH_3), 1.09 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.9, 138.5, 136.2, 134.0, 129.7, 129.0, 128.6, 126.5, 122.4, 61.7, 51.0, 21.0, 14.0; IR (KBr) ν: 3458, 3331, 3028, 2953, 1953, 1887, 1741, 1679, 1618, 1513, 1480, 1382, 1305, 1224, 1114, 1021, 918, 812, 755 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{25}N_2O_5$ ($[M-H]^+$): 421.1769. Found: 421.1765.

Compound **2i**: white solid, 58%, mp 100–101 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.40 (d, J=9.0 Hz, 2H, ArH), 7.31 (t, J=7.8 Hz, 2H, ArH), 7.24 (d, J=9.0 Hz, 2H, ArH), 7.22 (d, J=7.2 Hz, 1H, ArH), 6.91 (d, J=9.0 Hz, 2H, ArH), 5.21 (s, 1H, CH), 4.21–4.09 (m, 2H, CH_2), 4.07–4.01 (m, 2H, CH_2), 3.80 (s, 3H, CH_3), 3.73 (s, 3H, CH_3), 2.93 (t, J=7.2 Hz, 2H, CH_2), 1.09 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.9, 158.0, 138.5, 129.3, 128.9, 128.6, 126.5, 124.5, 114.4, 61.6, 55.4, 51.0, 14.0; IR (KBr) ν: 3454, 3330, 2949, 2838, 2050, 1740, 1676, 1617, 1513, 1477, 1385, 1307, 1222, 1116, 1026, 915, 829, 757 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{25}N_2O_6$ ($[M-H]^+$): 437.1718. Found: 437.1715.

Compound **2j**: white solid, 43%, mp 119–120 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.53 (d, J=8.4 Hz, 2H, ArH), 7.35 (d, J=8.4 Hz, 2H, ArH), 7.31 (t, J=7.8 Hz, 2H, ArH), 7.23 (t, J=9.0 Hz, 3H, ArH), 5.26 (s, 1H, CH), 4.17–4.10 (m, 2H, CH_2), 4.09–4.02 (m, 2H, CH_2), 3.74 (s, 3H, CH_3), 2.92 (t, J=7.2 Hz, 2H, CH_2), 1.10 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.6, 138.4, 135.2, 131.5, 129.2, 128.9, 128.6, 126.5, 123.0, 61.8, 51.1, 14.0; IR (KBr) ν: 3452, 3331, 3025, 2950, 2401, 2317, 1883, 1740, 1676, 1619, 1487, 1380, 1306, 1223, 1089, 1018, 915, 826, 753 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{22}ClN_2O_5$ ($[M-H]^+$): 441.1223. Found: 441.1221.

Compound **2k**: light orange solid, 55%, mp 148–150 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.38 (d, J=6.6 Hz, 2H, ArH), 7.34–7.29 (m, 4H, ArH), 7.22 (d, J=6.6 Hz, 1H, ArH), 7.16 (d, J=7.2 Hz, 2H, ArH), 6.14 (s, 1H, CH), 5.25 (d, J=21.6 Hz, 1H, CH), 4.10–4.02 (m, 2H, CH_2), 3.76 (s, 3H, CH_3), 2.31 (s, 3H, CH_3), 1.59 (d, J=6.6 Hz, 3H, CH_3), 1.07 (s, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.7, 136.2, 133.9, 129.7, 128.7, 128.6, 127.1, 125.9, 122.4, 61.7, 51.1, 24.3, 20.9, 14.0; IR (KBr) ν: 3457, 3327, 3022, 2974, 2927, 1931, 1740, 1699, 1623, 1515, 1454, 1381, 1311, 1202, 1116, 1021, 906, 807, 760 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{25}N_2O_5$ ($[M-H]^+$): 421.1769. Found: 421.1768.

Compound **2l**: light yellow solid, 60%, mp 125–127 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.38 (d, J=8.4 Hz, 2H, ArH), 7.34 (t, J=6.6 Hz, 2H, ArH), 7.32–7.27 (m, 2H, ArH), 7.22 (t, J=7.2 Hz, 1H, ArH), 6.90–6.87 (m, 2H, ArH), 6.14 (s, 1H, CH), 5.21 (d, J=22.2 Hz, 1H, CH), 4.13–4.06 (m, 1H, CH), 4.04–3.99 (m, 1H, CH), 3.79 (d, J=4.8 Hz, 3H, CH_3O), 3.76 (s, 3H, CH_3), 1.59–1.56 (m, 3H, CH_3), 1.08 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.7, 158.0, 129.3, 128.7, 128.6, 127.1, 125.9, 125.8, 124.5, 114.3, 61.6, 55.4, 51.1, 24.3, 14.0; IR (KBr) ν: 3456, 3325, 3070, 2973, 2930, 2840, 2045, 1741, 1698, 1622, 1514, 1455, 1380, 1305, 1244, 1200, 1116, 1026, 906, 846, 808, 762 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{25}N_2O_6$ ($[M-H]^+$): 437.1718. Found: 437.1718.

Compound **2m**: white solid, 35%, mp 156–158 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.50 (d, J=8.4 Hz, 2H, ArH), 7.33 (t, J=6.6 Hz, 6H, ArH), 7.23 (d, J=6.0 Hz, 1H, ArH), 6.09 (s, 1H, CH), 5.24 (s, 1H, CH), 4.12–4.10 (m, 1H, CH), 4.07–4.02 (m, 1H, CH), 3.77 (s, 3H, CH_3), 1.59 (d, J=6.6 Hz, 3H, CH_3), 1.09 (t, J=4.8 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.5, 135.2, 131.6, 129.2, 128.7, 127.2, 125.8, 123.2, 61.9, 51.2, 24.4, 14.0; IR (KBr) ν: 3456, 3331, 3088, 2975, 2928, 1785, 1711, 1681, 1624, 1489, 1379, 1309, 1203, 1116, 1021, 907, 848, 808, 761 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{22}ClN_2O_5$ ($[M-H]^+$): 441.1223. Found: 441.1223.

Compound **2n**: white solid, 40%, mp 139–141 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.24 (s, 1H, NH), 7.44 (d, J=8.4 Hz, 2H, ArH), 7.24 (t, J=8.1 Hz, 1H, ArH), 7.19–7.18 (m, 3H, ArH), 7.14 (d, J=8.4 Hz, 1H, ArH), 7.07 (d, J=7.8 Hz, 1H, ArH), 5.39 (s, 1H, CH), 4.16–4.08 (m, 2H, CH_2), 3.74 (s, 3H, CH_3), 2.33 (s, 3H, CH_3), 1.12 (t, J=6.9 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.2, 164.3, 163.4, 143.9, 139.2, 136.4, 134.0, 133.8, 129.8, 129.7, 129.3, 129.0, 125.2, 123.7, 123.4, 122.1, 121.9, 121.5, 103.5, 62.1, 62.0, 51.6, 21.0, 14.0; IR (KBr) ν: 3468, 3297, 3059, 2969, 1797, 1748, 1714, 1645, 1587, 1516, 1468, 1370, 1296, 1243, 1106, 1030, 932, 859, 811, 761 cm^{-1}; MS (m/z): 427.26 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{22}H_{20}ClN_2O_5$ ($[M-H]^+$): 427.1066. Found: 427.1068.

Compound **2o**: yellow solid, 60%, mp 138–141 °C; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.29 (s, 1H, NH), 7.56 (d, J=9.0 Hz, 2H, ArH), 7.32 (d, J=9.0 Hz, 2H, ArH), 7.13 (d, J=9.0 Hz, 2H, ArH), 6.86 (d, J=9.0 Hz, 2H, ArH), 5.36 (s, 1H, CH), 4.18–4.15 (m, 1H, CH), 4.11–4.08 (m, 1H, CH), 3.81 (s, 3H, OCH_3), 3.75 (s, 3H, CH_3), 1.13 (t, J=7.2 Hz, 3H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 168.3, 164.7, 163.5, 157.7, 145.0, 135.3, 131.4, 130.6, 129.2, 125.8, 125.6, 124.2, 122.8, 114.3, 113.7, 113.6, 100.7, 62.1, 61.4, 58.5, 55.4, 51.5, 14.0; IR (KBr) ν: 3251,

3056, 2987, 2839, 1699, 1640, 1503, 1449, 1376, 1297, 1220, 1114, 1032, 937, 830, 779 cm^{-1}; MS (*m*/*z*): 443.36 ($[M-1]^+$) 100%. HRMS (ESI) calcd for $C_{22}H_{20}ClN_2O_6$ ($[M-H]^+$): 443.1015. Found: 443.1015.

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Supplementary data

Crystallographic data **1a** (CCDC 875483), **1g** (CCDC 875484) and **2d** (CCDC 875485) been deposited at the Cambridge Crystallographic Database Centre. These data can be obtained free of charge via www.ccdc.ac.ck./data_request/cif. Supplementary data related to this article can be found online at http://dx.doi.org/10.1016/j.tet.2012.11.018.

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