An Efficient Synthesis of Highly Functionalized 4*H*-Pyrano[3,2-*d*]isoxazoles via Isocyanide-Based Three-Component Reaction

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Abstract: We present a novel, convenient, and efficient method for synthesizing polysubstituted 4*H*-pyrano[3,2-*d*]isoxazoles based on a three-component reaction. The zwitterions generated from the reaction of isocyanides and dialkyl acetylenedicarboxylates are reacted with 3-phenylisoxazol-4-(5*H*)-one to produce the title compounds in good yield.

Key words: 4*H*-pyrano[3,2-*d*]isoxazole, isocyanides, 3-phenylisoxazol-4-(5*H*)-one, three-component reactions, acetylenic esters

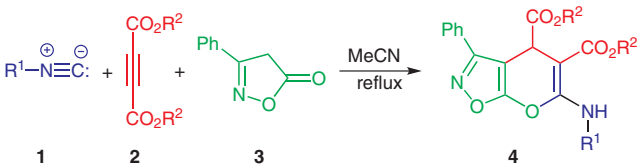
Multicomponent reactions (MCR),[1,2] followed by post-condensation modifications via several ring-forming reactions, are attractive due to their simple experimental procedures, one-pot character, and synthetic efficiency. Devising reactions that form several bonds in one step is an important area in modern organic synthesis.[3] Of pivotal importance in this area are the isocyanide-based MCR such as the versatile Ugi and Passerini reactions.[4,5] Isocyanide-based MCR, leading to interesting heterocyclic scaffolds, are particularly useful for the construction of diverse chemical libraries of druglike molecules.[2a]

One class of hetereocycles, the isoxazoles, has received considerable attention owing to their wide range of applications, including use as organic electrolytes in nonaqueous batteries, photographic emulsions, and fiber dyes.[6,7] Furthermore, they are valuable synthetic intermediates due to the ease of reductive cleavage and ring transformations.[8,9] Interest in pyrano-fused isoxazole derivatives stems from the presence of these heterocyclic systems biologically active compounds.[10] Consequently, there is an ongoing interest in the synthesis of pyrano isoxazoles.

One of the earliest approaches to the synthesis of these compounds was reported by Marcus et al. in 1967, reacting diketene with oximes and 1,4-diazabicyclo[2,2,2]-octane to produce pyrano[3,4-*d*]isoxazol-4-one.[11] In continuation of the work of Marcus, Chantegrel, and coworkers used pyrano[4,3-*b*]pyrans with an excess of hydroxylamine to synthesize pyranoisoxazoles.[12] Pyrano[3,4-*c*]isoxazoles and related compounds have also been prepared by addition of unsaturated alkoxide anions to simple nitroolefines followed by intramolecular nitrile oxide or silyl nitronate cycloadditions.[13–19]

Recent methods for the synthesis of pyranoisoxazoles include cycloaddition between alkynes and nitrile oxides,[20,21] 1,3-dipolar cycloaddition of acetonitrile oxide to an α,β-unsaturated enone,[22] intramolecular 1,3-dipolar cycloaddition of nitrones or nitrile oxides[23] and treatment of 2-aryl-substituted 1-nitro-3-oxa-6-heptynes with BuLi followed by treatment with acetic anhydride.[24] Due to our interest in heterocyclic compounds with potential biological activity,[25,26] we herein describe a novel synthesis of highly substituted 4*H*-pyrano[3,2-*d*]isoxazoles **4**, by the reaction of an isocyanide **1** with a dialkyl acetylenedicarboxylate (DAAD, **2**), and 3-phenylisoxazol-4-(5*H*)-one (**3**)[27] via three-component reaction in good yields (Table 1).

Table 1 Synthesis of 4*H*-Pyrano[3,2-*d*]isoxazole Derivatives



1 2 3 4

Entry	R^1	R^2	Product	Yield (%)[a]
1	*c*-Hex	Me	**4a**	82
2	*c*-Hex	Et	**4b**	79
3	*c*-Hex	*i*-PrOH	**4c**	77
4	*c*-Hex	neopentyl	**4d**	61
5	*c*-Hex	*t*-Bu	**4e**	57
6	*t*-Bu	Me	**4f**	81
7	*t*-Bu	Et	**4g**	80
8	*t*-Bu	neopentyl	**4h**	62

[a] Isolated yield.

To achieve suitable conditions for the above transformation, in our initial study, the reaction of cyclohexyl isocyanide, dimethyl acetylenedicarboxylate (DMAD), and 3-phenylisoxazol-4-(5*H*)-one was used as a model to optimize the reaction conditions. We examined the reaction in various solvents at different temperatures to increase the product yields and to reduce the reaction time (Table 2). We subsequently used various dialkyl acetylenedicarboxylates and isocyanides with 3-phenylisoxazol-4-(5*H*)-one

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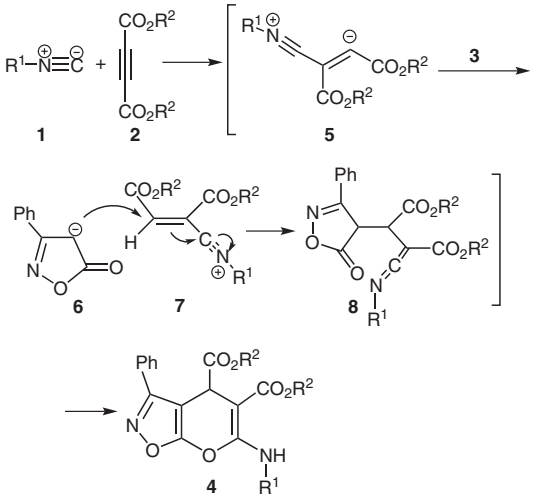
in MeCN under reflux conditions, leading to the formation of the corresponding 4*H*-pyrano[3,2-*d*]isoxazole derivatives **4a**–**h**, in good yields (Table 1). All reactions proceed to completion within three hours. The structures of the products were deduced from their IR, ^{1}H NMR, and ^{13}C NMR spectra. The mass spectra of these compounds displayed molecular ion peaks at the appropriate *m/z* values. For example, the ^{1}H NMR spectrum of **4a** contained multiplet signals for the cyclohexyl ring (δ = 1.33–2.02 ppm), two singlets for two methoxy groups (δ = 3.42 and 3.71 ppm), multiplets for NCH of the cyclohexyl ring (δ = 3.84 ppm), the allylic methine (δ = 4.79 ppm), and five aryl protons (δ = 7.47, 7.78 ppm). An exchangeable doublet for the NH group (δ = 8.94 ppm) was also observed. The chemical shift of NH group indicates that it participates in an intramolecular hydrogen bond with the vicinal carbonyl group. The ^{13}C NMR spectrum of **4a** showed 20 distinct resonances in agreement with the proposed structure, partial assignment of these resonances is given in the experiment section. Characteristic ^{13}C NMR signals were due to two ester carbonyls at δ = 169.45 and 172.59 ppm. The IR spectrum of **4a** showed strong absorptions at 1750 and 1740 cm^{-1} owing to the carbonyls and the amino group at 3250 cm^{-1} as a weak broad band. The ^{1}H decoupled ^{13}C NMR spectra of **4b**–**h** are similar to those of **4a** except for the R^1 or R^2 groups, which exhibit characteristic signals with appropriate chemical shifts.

Table 2 Optimization Conditions for the Synthesis of **4a**

Entry	Solvent	Temp (°C)	Yield (%)
1	MeCN	82	82
2	nitrobenzene	80	71
3	benzene	80	63
4	DMF	80	20
5	THF	66	31
6	DMSO	80	12
7	EtOH	79	40
8	toluene	110	45
9	acetone	56	55
10	CH_2Cl_2	40	50

Although the mechanism of this reaction has not been established experimentally, a possible pathway is proposed in Scheme 1. Mechanistically, it is conceivable that the reaction involves the initial formation of a 1:1 zwitterionic intermediate **5** between the isocyanide and DAAD protonation of **5** by **3** and subsequent attack of the resulting nucleophile generated **6**, to the positively charged ion **7** afforded ketenimine **8** (Scheme 1). Such an addition product may tautomerize and cyclize, under the reaction conditions employed, to produce **4**.



Scheme 1 Proposed mechanism

In summary, we have developed a new efficient one-pot synthesis of 4*H*-pyrano[3,2-*d*]isoxazoles **4** from 3-phenylisoxazol-4-(5*H*)-one (**3**), isocyanides **1**, and dialkyl acetylenedicarboxylates **2**, via a three-component, one-pot reaction without any specific modifications. Good yields of the products, the readily availability of the starting materials, and experimental simplicity are the main advantages of this method. To the best of our knowledge, this method represents the first example of an efficient, three-component method for the synthesis of 4*H*-pyrano[3,2-*d*]isoxazole derivatives **4a**–**h**. We believe this approach will be of value to those requiring access to novel synthetic of pyrano[3,2-*d*]isoxazoles.

Dimethyl 6-(Cyclohexylamino)-3-phenyl-4*H*-pyrano[3,2-*d*]isoxazole-4,5-dicarboxylate (4a); Typical Procedure

To a magnetically stirred solution of 3-phenylisoxazol-4-(5*H*)-one (0.16 g, 1 mmol) and dimethyl acetylenedicarboxylate (0.14 g, 1 mmol) in MeCN (5 mL) was added dropwise a solution of cyclohexyl isocyanide (0.11 g, 1 mmol) in MeCN (1 mL) at r.t. over 10 min. The reaction was then refluxed for 3 h. The solvent was removed under reduced pressure, and the residue was purified by silica gel (Merck 230–240 mesh) column chromatography using a short column eluting with *n*-hexane–EtOAc–Et_3N (75:5:1) to yield **4a** as a white solid (0.34 g, 82%); mp 122–124 °C. IR (KBr): ν_{max} = 3250, 1750, 1740 cm^{-1}. MS: *m/z* (%) = 412 (5) [M$^+$], 353 (98), 271 (60), 239 (100), 105 (60), 77 (30), 55(45). ^{1}H NMR (300.13 MHz, $CDCl_3$): δ = 1.25–2.02 (10 H, m, 5 CH_2 of cyclohexyl), 3.42 (3 H, s, OCH_3), 3.71 (3 H, s, OCH_3), 3.84 (1 H, m, C*H*NH), 4.79 (1 H, s, C*H*CO_2CH_3), 7.47 (3 H, m, arom.), 7.78 (2 H, m, arom.), 8.94 (1 H, d, $^3J_{HH}$ = 7.8 Hz, CHN*H*) ppm. ^{13}C NMR (75.47 MHz, $CDCl_3$): δ = 24.3, 24.34, 25.28, 33.42, 33.69 (5 CH_2 of cyclohexyl), 37.4 (*C*HCO_2CH_3), 50.82, 51.19 (2 OCH_3), 52.08 (CHNH), 71.69, 87.32, 127.72, 128.21, 128.50, 130.19, 159.03, 161.29, 164.64 (C alkene, C Ar), 169.45, 172.59 (2 C=O) ppm. Anal. Calcd (%) for $C_{22}H_{24}N_2O_6$: C, 64.07; H, 5.87; N, 6.79. Found: C, 63.67; H, 5.91; N, 6.75.

Diisopropyl-6-(cyclohexylamino)-3-phenyl-4*H*-pyrano[3,2-*d*]isoxazole-4,5-dicarboxylate (4c)

White powder (0.36 g, 77%), mp 102–104 °C. IR (KBr): ν_{max} = 3250, 1750, 1740 cm^{-1}. MS: *m/z* (%) = 468 (5) [M$^+$], 381 (100), 339

(28), 257 (40), 239 (50), 105 (40). ^{1}H NMR (300.13 MHz, $CDCl_3$): δ = 0.58, 1.03. 1.19, 1.28 [12 H, 4 d, $^3J_{HH}$ = 6.2 Hz OCH(CH_3)$_2$,], 1.29–2.02 (10 H, m, 5 CH_2 of cyclohexyl), 3.78 (1 H, br s, C*H*NH), 4.70 [1 H, sept, $^3J_{HH}$ = 6.2 Hz OC*H*(CH_3)$_2$], 4.72 [1 H, s, C*H*CO_2CH(CH_3)$_2$], 5.05 [sept, $^3J_{HH}$ = 6.2 Hz, OC*H*(CH_3)$_2$], 7.47 (3 H, m, arom.), 7.78 (2 H, m, arom.), 8.96 (1 H, d, $^3J_{HH}$ = 7.8 Hz, CHN*H*) ppm. ^{13}C NMR (75.47 MHz, $CDCl_3$): δ = 20.58, 21.55, 21.90, 22.24 (4 CH_3), 24.43, 25.37, 33.53, 33.81, 37.67 (5 CH_2 of cyclohexyl), 37.7 [*C*HCO$_2$CH(CH_3)$_2$], 50.22 (*C*HNH), 67.17, 68.56 [2 OCH(CH_3)$_2$], 72.17, 87.16, 128.16, 128.49, 128.67, 130.14, 158.97, 161.39, 164.79 (C alkene, C Ar), 168.8, 171.9 (2 C=O) ppm. Anal. Calcd (%) for $C_{26}H_{32}N_2O_6$: C, 66.65; H, 6.88; N, 5.98. Found: C, 66.56; H, 6.93; N, 5.96.

Di-*tert*-butyl-6-(cyclohexylamino)-3-phenyl-4*H*-pyrano[3,2-*d*]isoxazole-4,5-dicarboxylate (4e)

White powder (0.28 g, 57%), mp 134–135 °C. IR (KBr): ν_{max} = 3250, 1750, 1740 cm^{-1}. MS: *m/z* (%) = 496 (5) [M$^+$], 409 (100), 257 (24), 105 (25), 77 (5) cm^{-1}. ^{1}H NMR (300.13 MHz, $CDCl_3$): δ = 1.10, 1.50 [18 H, 2 s, 2 OC(CH_3)$_3$], 1.26–2.00 (10 H, m, 5 CH_2 of cyclohexyl), 3.76 (1 H, br s, C*H*NH), 4.63 [1 H, s, C*H*CO$_2$C(CH_3)$_3$], 7.48 (3 H, t, m, arom.), 7.81 (2 H, m, arom.), 8.87 (1 H, d, CHN*H*, $^3J_{HH}$ = 7.8 Hz) ppm. ^{13}C NMR (75.47 MHz, $CDCl_3$): δ = 24.65, 25.42 (2 CH_2 of cyclohexyl), 27.48, 28.51 [2 OC(CH_3)$_3$], 33.73, 33.97, 38.74 (3 CH_2 of cyclohexyl), 38.79 [*C*HCO$_2$C(CH_3)$_3$], 50.33 (*C*HNH), 73.39 (C alkene), 80.08, 81.03 [2 OC(CH_3)$_3$], 87.58, 128.01, 128.63, 128.96, 130.09, 158.85, 161.28, 164.99 (C alkene, C Ar), 168.92, 171.89 (2 C=O) ppm. Anal. Calcd (%) for $C_{28}H_{36}N_2O_6$: C, 67.72; H, 7.31; N, 5.64. Found: C, 67.35; H, 7.37; N, 5.59.

Dimethyl-6-(*tert*-butylamino)-3-phenyl-4*H*-pyrano[3,2-*d*]isoxazole-4,5-dicarboxylate (4f)

White powder (0.31 g, 81%), mp 124–126 °C. IR (KBr): ν_{max} = 3250, 1750, 1740 cm^{-1}. MS: *m/z* (%) = 386 (5) [M$^+$], 328 (20), 327 (72), 271 (83), 239 (100), 105 (25), 77 (18). ^{1}H NMR (300.13 MHz, $CDCl_3$): δ = 1.48 [9 H, s, C(CH_3)$_3$], 3.42, 3.70 (6 H, 2 s, 2 OCH_3) 4.79 (1 H, s, C*H*CO$_2$CH$_3$), 7.46 (3 H, m, arom.), 7.77 (2 H, m, arom.), 9.09 (1 H, s, CHN*H*) ppm. ^{13}C NMR (75.47 MHz, $CDCl_3$): δ = 30.44 [C(*C*H$_3$)$_3$], 37.44 (*C*HCO$_2$CH$_3$), 51.38 [*C*(CH$_3$)$_3$], 52.15, 53.57 (2 OCH_3), 72.56, 87.34, 127.78, 128.29, 128.58, 130.26, 160.39, 161.38, 164.41 (C alkene, C Ar), 169.61, 172.58 (2 C=O). Anal. Calcd (%) for $C_{20}H_{22}N_2O_6$: C, 62.17; H, 5.74; N, 7.25. Found: C, 61.66; H, 5.75; N, 7.19.

Diethyl-6-(*tert*-butylamino)-3-phenyl-4*H*-pyrano[3,2-*d*]isoxazole-4,5-dicarboxylate (4g)

White powder (0.33 g, 80%), mp 109–111 °C. IR (KBr): ν_{max} = 3250, 1750, 1740 cm^{-1}. MS: *m/z* (%) = 414 (5) [M$^+$], 342 (28), 341 (100), 285 (92), 239 (73), 105 (30), 77 (15). ^{1}H NMR (300.13 MHz, $CDCl_3$): δ = 0.86 (3 H, t, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 1.24 (3 H, t, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 1.48 (9 H, s, NC(CH_3)$_3$], 3.85 (m, 2 H, OCH_2CH_3), 4.15 (m, 2 H, OCH_2CH_3), 4.76 (1 H, s, C*H*CO$_2$CH$_2$CH$_3$), 7.47 (3 H, m, arom.), 7.77 (2 H, m, arom.), 9.10 (1 H, s, CHN*H*) ppm. ^{13}C NMR (75.47 MHz, $CDCl_3$): δ = 13.56, 14.39 (2 OCH_2CH_3), 30.45 [C(*C*H$_3$)$_3$], 37.54 [*C*HCO$_2$C(CH$_3$)$_3$], 53.45 [*C*(CH$_3$)$_3$], 59.95, 60.99 (2 $CO_2CH_2CH_3$), 72.71, 87.22, 127.98, 128.47, 128.53, 130.19, 160.27, 161.47, 164.41 (C alkene, C Ar), 169.26, 172.42 (2 C=O) ppm. Anal. Calcd (%) for $C_{22}H_{26}N_2O_6$: C, 63.76; H, 6.32; N, 6.76. Found: C, 63.24; H, 6.41; N, 7.23.

Dineopentyl-6-(*tert*-butylamino)-3-phenyl-4*H*-pyrano[3,2-*d*]isoxazole-4,5-dicarboxylate (4h)

White powder (0.31 g, 62%), mp 138–139 °C. IR (KBr): ν_{max} = 3250, 1750, 1740 cm^{-1}. MS: *m/z* (%) = 498 (5) [M$^+$], 382 (30), 383 (100), 327 (48), 257 (47), 239 (34), 105 (28). ^{1}H NMR (300.13 MHz, $CDCl_3$): δ = 0.66. 0.95 [18 H, 2s, 2 $CH_2C(CH_3)_3$], 1.48 [9 H, s, NC(CH_3)$_3$], 3.61 [2 H, AB system, $CH_AH_BC(CH_3)_3$, $^2J_{HH}$ = 10.5 Hz], 3.80 [2 H, AB system, $CH_AH_BC(CH_3)_3$, $^2J_{HH}$ = 10.5 Hz], 4.85 [1 H, s, C*H*CO$_2$CH$_2$C(CH$_3$)$_3$], 7.45–7.48 (3 H, m, arom.), 7.85–7.89 (2 H, m, arom.), 9.12 (1 H, s, CHN*H*) ppm. ^{13}C NMR (75.47 MHz, $CDCl_3$): δ = 26.04, 26.54 [2 $CH_2C(CH_3)_3$], 30.44 [NC(*C*H$_3$)$_3$], 31.04, 31.42 [2 $CH_2C(CH_3)_3$], 37.74 [*C*HCO$_2$CH$_2$C(CH$_3$)$_3$], 73.58, 74.59 [2 O*C*H$_2$C(CH$_3$)$_3$], 73.41, 87.71, 127.62, 128.54, 128.88, 130.36, 160.7, 161.02, 164.79 (C alkene, C Ar), 169.25, 171.79 (2 C=O) ppm. Anal. Calcd (%) for $C_{28}H_{38}N_2O_6$: C, 67.45; H, 7.68; N, 5.62. Found: C, 66.79; H, 7.95; N, 5.47.

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