Facile Synthesis of Highly Substituted Iminocyclopentenes: A Novel Isocyanide-Based Three-Component Condensation Reaction

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Abstract: The reaction of alkoxymethylenemalononitrile with dialkyl acetylenedicarboxylates (DAAD) and cyclohexylisocyanide results in a three-component condensation reaction in which the alkoxymethylenemalononitrile group acts as a dipolarophile, producing highly functionalized iminocyclopentenes under mild conditions in high yields.

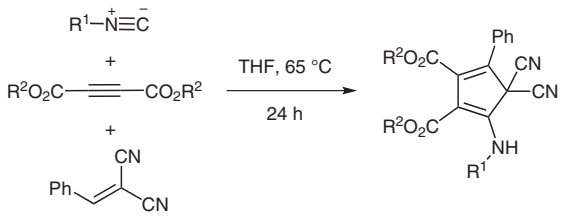
Key words: alkoxymethylenemalononitrile, dialkyl acetylenedicarboxylates, isocyanides, iminocyclopentenes

The development of new multicomponent reactions (MCR) offers efficient and convenient entries into interesting structures.[1] Devising such types of reactions that form several bonds in one-step is one of the main goal in modern organic synthesis.[2] Isocyanide-based MCR (IMCR), leading to novel heterocyclic scaffolds, are particularly useful for the construction of diverse chemical libraries of 'drug-like' molecules.[3]

Owing to their chemical and biological properties, five-membered carbocycles such as cyclopentenes have attracted much attention. They are frequently encountered components in natural products, and they are also valuable intermediates in organic synthesis.[4–6] As examples, cyclopentene-1,2-dicarboxylic acids have been incorporated in ACE inhibitors,[7] and Samuelsson et al. have reported the use of a trisubstituted cyclopentene moiety in VCH NS3 protease inhibitors.[8]

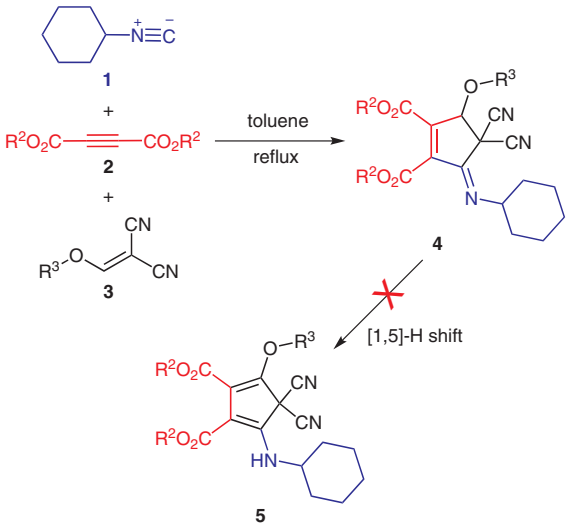
Consequently, a wide range of approaches has been devised for the synthesis of cyclopentene derivatives. One of these employs a classical intramolecular Michael-type conjugate addition to activated alkenes, alkynes,[9] and allenyl solfones.[10] Other approaches to cyclopentene derivatives include Ramberg–Bäcklund rearrangement of thiosugar-drived sulfones[11] and rearrangement of vinyl cyclopropanes.[12] Recent methods for the synthesis of cyclopentenes include the intramolecular Morita–Baylis–Hillman reaction,[13] ring-closing metathesis,[14] intramolecular Wittig reaction,[15,16] and an isocyanide-based three-component reaction.[17]

In 2004, Nair envisaged the possibility of intermolecular trapping of the zwitterionic intermediates generated from isocyanides and DMAD with arylidenemalononitriles as activated alkenes, leading to highly substituted cyclopentadienes (Scheme 1).[18] However, a literature survey revealed that the reaction of isocyanide and dialkyl acetylenedicarboxylate (DAAD) in the presence of alkoxymethylenemalononitrile has not been investigated.



Scheme 1

In the light of our interest in IMCR involving zwitterionic species[19–21] and impressed by the success of the above-mentioned reaction, cyclohexylisocyanide **1** was treated with DAAD and alkoxymethylenemalononitrile **3**. Structural characterization of the isolated product supported that the products obtained here should be **4** rather than **5** (Scheme 2). It should be pointed out that even under prolonged reaction times and high temperatures, product **4** failed to isomerize to **5** via a [1,5]-H shift.



Scheme 2

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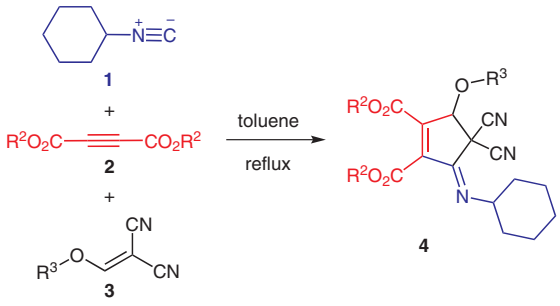
In a pilot experiment, cyclohexylisocyanide, DMAD, and methoxymethylenemalononitrile were refluxed in toluene. After 0.5 hours, the solvent was removed under vacuum. The residue was crystallized from *n*-hexane, to afford the highly substituted iminocyclopentene derivative **4a** in 93% yield. To explore the scope of this reaction further, other acetylenic esters and alkoxymethylenemalononitriles were used in a series of reactions and the outcomes are shown in Table 1. Two other isocyanides, *tert*-butylisocyanide and 1,1,3,3-tetramethylbutylisocyanide were also examined, but no conversion occurred. Furthermore use of unsymmetrical alkynes such as methyl acetylenecarboxylate and ethyl acetylenecarboxylate as reactants afforded a complex mixture of products. ^{1}H NMR analysis indicated the formation of desired product in the crude reaction mixture, but all attempts to purify the product failed.

Table 1 Synthesis of Highly Fuctionalized Iminocyclopentenes

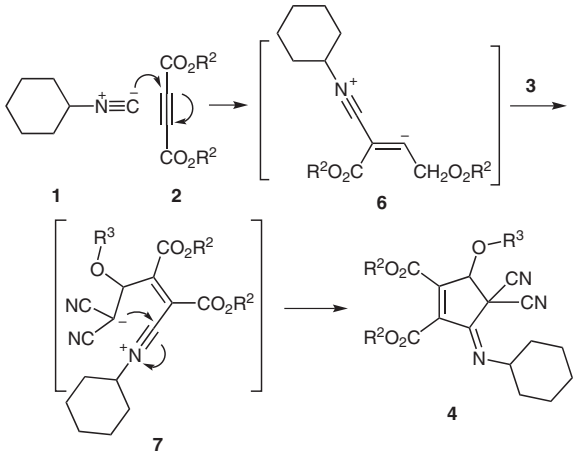


4	R^2	R^3	Time (min)	Yield (%)[a]
4a	Me	Me	30	93
4b	Et	Me	60	82
4c	*i*-Pr	Me	120	84
4d	*N*-P[b]	Me	120	75
4e	Me	Et	25	90
4f	Et	Et	60	86
4j	*i*-Pr	Et	120	60
4h	*N*-P[b]	Et	120	82

[a] Isolated yield.
[b] *N*-P = neopentyl.

The structures of the products were deduced from their IR, ^{1}H NMR, ^{13}C NMR and mass spectral data. 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 a cyclohexyl ring (δ = 1.12–1.85 ppm), a singlet for the methoxy group (δ = 3.73 ppm), two singlets for the two ester methoxy groups (δ = 3.86 and 3.89 ppm), a multiplet for NCH of the cyclohexyl ring (δ = 3.97 ppm), and a singlet for the ring methine proton (δ = 5. 98 ppm). The assignment was supported by IR absorptions at 1741, 1725 (2 C=O), and 1656 (C=N) cm^{-1}. The vibration band for the CN triple-bond stretching did not appear in IR spectrum of **4** owing to being attached to a carbon-bearing electron-accepting cyanide and imine groups.[22] Nevertheless, the appearance of a moderately strong band at 2200 cm^{-1} in the FT-Raman spectrum of **4e**, clearly confirmed the presence of a nitrile group in the products. The proton-decoupled ^{13}C NMR spectrum of **4a** showed 18 distinct resonances. Two characteristic ^{13}C NMR signals due to the cyanide carbons appeared at δ = 108.83 and 111.70 ppm and those of the two ester carbonyls at δ = 161.27 and 161.92 ppm. The ^{1}H NMR and ^{13}C NMR of **4b–h** are comparable to those of **4a** except for the R^1 and R^2 groups, which exhibit characteristic signals with appropriate chemical shifts (see Supplementary Information).

Although the mechanism of the reaction between the isocyanide and dialkyl acetylenedicarboxylate in the presence of arylidenemalononitrile has not yet been established experimentally, a possible explanation is proposed in Scheme 3. The initial event is the formation of the zwitterion **6** from the isocyanide **1** and DAAD **2**. The zwitterion can add to the electrophilic carbon–carbon double bond of **3** resulting in the formation of **7**, which then undergoes cyclization to product **4**.



Scheme 3

In conclusion, we have devised a novel three-component, one-pot reaction for the synthesis of highly substituted iminocyclopentene derivatives. The simple and readily available starting materials, lack of need for catalysts, neutral reaction conditions, and good to high yields of the products are the main advantages of our method.

General Procedure for the Synthesis of Dimethyl-4,4-dicyano-3-(cyclohexylimino)-5-methoxycyclopent-1-ene-1,2-dicarboxylate (4a)

To a stirred solution of methoxymethylenemalononitrile **3a** (1.0 mmol) and dimethyl acetylenedicarboxylate (1.0 mmol) in toluene (5 mL) was added dropwise a solution of cyclohexyl isocyanide (1.0 mmol) over 10 min by syringe, and the reaction mixture was stirred and refluxed for about 0.5 h. On completion of the reaction, as monitored by TLC, solvent was removed under vacuum. The residue

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was then crystallized from *n*-hexane to yield the fully substituted cyclopentene **4a** in 93% yield.

Compound 4a
White solid; yield 93%; mp 112–114 °C. IR (KBr): 2916 and 2843 (CH), 1741 and 1725 (2 C=O), 1664 (C=N), 1436, 1270 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.27–1.85 (m, 10 H), 3.73 (s, 3 H), 3.86 (s, 3 H), 3.89 (s, 3 H), 3.97 (m, 1 H), 5.98 (s, 1 H). ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 23.62, 23.69, 25.25, 32.10, 32.13 (5 CH_2), 37.41 (C), 53.16, 53.21 (2 OMe), 61.50 (CHN), 64.78 (OMe), 86.96 (CHOMe), 108.83, 111.70 (2 CN), 140.80, 143.61 (2 C), 150.12 (C=N), 161.27, 161.92 (2 C=O). MS: *m/z* (%) = 359 (4) [M^+], 327 (17) [M^+ – H – OMe], 300 (8) [M^+ – CO_2Me], 245 (33) [M^+ – C_6H_{11} – OMe], 83 (100) [C_6H_{11}].

Diethyl-4,4-dicyano-3-(cyclohexylimino)-5-methoxycyclopent-1-ene-1,2-dicarboxylate (4b)
White solid; yield 82%; mp 104–106 °C. IR (KBr): 2923 (CH), 1739 and 1720 (2 C=O), 1656 (C=N), 1629, 1332, 1253, 11.96 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.28–1.39 (m, 9 H), 1.43–1.64 (m, 3 H), 1.78–1.84 (m, 3 H), 3.73 (s, 3 H), 3.97 (m, 1 H), 4.32 (m, 4 H), 5.09 (s, 1H). ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 13.88, 13.98 (2 CH_3), 23.54, 23.61, 25.27, 32.10, 32.13 (5 CH_2), 37.37 (C), 61.47 (CHN), 62.36 (OCH_2Me), 64.50 (OMe), 87.01 (CHOMe), 108.92, 111.77 (2 CN), 140.77, 143.49 (2 C), 150.15 (C=N), 160.81, 161.48 (2 C=O). MS: *m/z* (%) = 387 (7) [M^+], 355 (8) [M^+ – H – OMe], 341 (17) [M^+ – H – OEt], 314 (15) [M^+ – CO_2Et], 259 (51) [M^+ – C_6H_{11} – OEt], 231 (50) [M^+ – CO_2Et – C_6H_{13}], 82 (100) [C_6H_{10}].

Diisopropyl-4,4-dicyano-3-(cyclohexylimino)-5-methoxycyclopent-1-ene-1,2-dicarboxylate (4c)
White solid; yield 84%; mp 82–83 °C. IR (KBr): 2926 (CH), 1730, 1725 (2 C=O), 1659 (C=N), 1642, 1448, 1442, 1367, 1285 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.25–1.41 (m, 16 H), 1.45–1.63 (m, 4 H), 1.79–1.85 (m, 4 H), 3.75 (s, 3 H), 3.98 (m, 1 H), 5.11 (s, 1 H), 5.13–5.32 (m, 2 H). ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 21.58 21.65 (2 CH_3), 23.46, 23.52, 25.34, 32.18 (5 CH_2), 37.31 (C), 61.41 (CHN), 64.23 (OMe), 70.47, 70.56 [2 OCH $(Me)_2$], 87.24 (CHOMe), 108.98, 111.89 (2 CN), 140.61, 143.55 (2 C), 150.12 (C=N), 160.38, 161.17 (2 C=O). MS: *m/z* (%) = 415 (5) [MH^+], 383 (5) [M^+ – CO_2Me], 356 (4) [M^+ – $OCH(CH_3)_2$], 328 (10) [M^+ + 1 – $CO_2CH(CH_3)_2$], 314 (15) [M^+ – CN – OMe – $CH(CH_3)_2$], 83 (89) [C_6H_{13}].

Dineopentyl-4,4-dicyano-3-(cyclohexylimino)-5-methoxycyclopent-1-ene-1,2-dicarboxylate (4d)
White solid; yield 75%; mp 131–133 °C. IR (KBr): 2918 (CH), 1740 and 1720 (C=O), 1642 (C=N), 1638, 1365, 1362, 1278 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ = 0.96 (s, 18 H), 1.26–1.57 (m, 5 H), 1.57–1.60 (m, 2 H), 1.61–1.9 (m, 4 H), 3.76 (s, 3 H), 3.90–4.1 (m, 5 H), 5.11 (s, 1 H), 5.13–5.32 (m, 2 H). ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 23.67, 23.75, 25.23 (3 CH_2), 26.31 (CH_3), 31.41, 31.69 (2 C), 32.24 (2 CH_2), 37.21 (C), 61.39 (CHN), 64.78 (OMe), 75.55, 75.76 [2 $OCH_2C(Me)_3$], 87.31 (CHOMe), 108.96, 111.82 (2 CN), 140.66, 143.40 (2 C), 150.39 (C=N), 161.09, 161.66 (2 C=O). MS: *m/z* (%) = 472 (47) [MH^+], 456 (7) [M^+ – Me], 439 (6) [M^+ – OMe], 356 (7) [M^+ – $CH_2C(CH_3)_3$], 83 (27) [C_6H_{11}], 71 (100) [$CH_2C(CH_3)_3$].

Dimethyl-4,4-dicyano-3-(cyclohexylimino)-5-ethoxycyclopent-1-ene-1,2-dicarboxylate (4e)
White solid; yield 90%; mp 92–94 °C. IR (KBr): 2928 and 2849 (CH), 1747 and 1727 (C=O), 1660 (C=N), 1637, 1270, 1428, 1272 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.22–1.77 (m, 13 H), 3.78–4.10 (m, 9 H), 5.12 (s, 1 H), ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 14.77 (CH_3), 23.65, 23.72, 25.25, 32.10, 32.13 (5 CH_2), 37.77 (C), 53.19 (OCH_3), 64.81 (CHN), 70.06 (OCH_2), 85.34 (HCOEt), 109.15, 111.76 (2 CN), 141.00, 143.48 (2 C), 150.21 (C=N), 161.29, 162.07 (2 C=O). MS: *m/z* (%) = 374 (93) [MH^+], 341 (19) [M^+ – OMe – H], 327 (19) [M^+ – OEt – H], 314 (18) [M^+ – CO_2Me], 291 (20) [M^+ –C_6H_{11}], 83 (100) [C_6H_{11}].

Diethyl-4,4-dicyano-3-(cyclohexylimino)-5-ethoxycyclopent-1-ene-1,2-dicarboxylate (4f)
White solid; yield 86%; mp 115–116 °C. IR (KBr): 2972 and 2919 (CH), 1737 and 1718 (C=O), 1658 (C=N), 1633, 1328, 1252 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.24–1.35 (m, 12 H), 1.39–1.50 (m, 2 H), 1.54–1.63 (m, 2 H), 1.65–1.70 (m, 1 H), 1.78–1.89 (m, 4 H), 3.84–4.04 (m, 3 H), 4.21–4.39 (m, 4 H), 5.18 (s, 1 H). ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 13.88, 13.99, 14.74 (3 CH_3), 23.55, 23.61, 25.29, 32.13 (5 CH_2), 37.75 (C), 62.28, 62.33 (2 OCH_2), 64.48 (CHN), 70.00 (OCH_2), 85.48 (HCOEt), 109.22, 111.86 (2 CN), 140.97, 143.41 (2 =C), 150.29 (C=N), 160.82, 161.58 (2 C=O). MS: *m/z* (%) = 402 (51) [MH^+], 401 (6) [M^+], 355 (19) [M^+ – OEt – H], 328 (12) [M^+ – CO_2Me], 273 (15) [M^+ – OEt – C_6H_{11}], 83 (97) [C_6H_{11}].

Diisopropyl-4,4-dicyano-3-(cyclohexylimino)-5-ethoxycyclopent-1-ene-1,2-dicarboxylate (4g)
White solid; yield 60%; mp 77–79 °C. IR (KBr): 2937, 2924 and 2848 (CH), 1733 and 1716 (C=O), 1655 (C=N), 1628, 1446, 1365, 1257 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.31 (m, 18 H), 1.45–1.63 (m, 4 H), 1.78–1.84 (m, 4 H), 3.84–4.01 (m, 3 H), 5.12–5.28 (m, 3 H). ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 14.78, 21.55, 21.63 (3 CH_3), 23.44, 23.51, 25.34, 32.16 (5 CH_2), 37.68 (C), 64.14 (CHN), 69.97 [$OCH(Me)_2$], 70.38 (OCH_2), 85.65 (HCOEt), 109.27, 111.96 (2 CN), 140.86, 143.46 (2 C), 150.30 (C=N), 160.36, 161.23 (2 C=O). MS: *m/z* (%) = 430 (24) [MH^+], 429 (4) [M^+], 383 (5) [M^+ – OEt – H], 370 (4) [M^+ – OCH $(CH_3)_2$], 342 (13) [M^+ – $O_2CH(CH_3)_2$], 83 (97) [C_6H_{11}].

Dineopentyl-4,4-dicyano-3-(cyclohexylimino)-5-ethoxycyclopent-1-ene-1,2-dicarboxylate (4h)
White solid; yield (82%); mp 122–124 °C. IR (KBr): 2919, 2844 (CH), 1738 and 1720 (C=O), 1657 (C=N), 1637, 1473, 1366, 1271 cm^{-1}. ^1H NMR (300 MHz,$CDCl_3$): δ = 0.96 (s, 18 H), 1.26–1.60 (m, 8 H), 1.68–1.72 (m, 1 H), 1.80–1.90 (m, 4 H), 3.88–4.11 (m, 4 H), 5.22 (s, 1 H). ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 14.74 (CH_3), 23.68, 23.76, 25.24 (3 CH_2), 26.3 (CH_3), 31.43, 31.70 (2 C), 32.24 (2 CH_2), 37.56 (C), 64.76 (CHN), 70.03, 75.54, 75.54 (3 CH_2), 85.75 (HCOEt), 109.26, 111.91 (2 CN), 140.90, 143.20 (2 C), 150.55 (C=N), 161.13, 161.77 (C=O). MS: *m/z* (%) = 486 (30) [MH^+], 485 (3) [M^+], 470 (6) [M^+ – Me], 439 (5) [M^+ – OEt – H], 370 (6) [M^+ – $O_2CH_2C(CH_3)_3$], 83 (25) [C_6H_{11}], 71 (100) [M^+ – $CH_2C(CH_3)_3$].

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synlett. Copies of the IR, mass, ^1H NMR, and ^{13}C NMR spectra for products are available.

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