

Metallic Ytterbium-Mediated Reductive Dimerization Cyclization of Arylmethylenecyanoacetates

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The reductive dimerization cyclization of arylmethylenecyanoacetates promoted by metallic ytterbium has been studied. Highly functionalized cyclopentenes were obtained, and the *trans*, *trans*-isomers were the major products.

Carbon–carbon bond formation is the essence of organic synthesis, and the reductive dimerization of carbonyl derivatives by active metal is one of the most valuable methods for establishing carbon-carbon bonds. In general, carbonyl derivatives are aldehydes, ketones, carboxylic esters, acid chlorides, or imines; active metals are alkali or alkaline earth metals; and the reactions carried out in anhydrous solvents.[1] Fujiwara et al. have reported that the cyclodimerization of α, β-unsaturated carbonyl compounds promoted by ytterbium metal alone and trichlorolanthanoid ($LnCl_3$)/Zn can give cyclopentanol derivatives.[2]

During the last decade, metallic ytterbium-mediated organic reactions have received considerable attention.[3] Fujiwara and his group have demonstrated that diaryl (thio)ketones are readily umpoled by ytterbium metal to react with various electrophiles;[4] metallic ytterbium can also promote many reduction or coupling reactions;[5] many reactions catalyzed by metallic ytterbium or organoytterbium reagents have been investigated recently.[6] Herein, we wish to report on the metallic ytterbium-mediated reductive dimerization cyclization of arylmethylenecyanoacetates at room temperature to afford highly functionalized cyclopentenes in both high yields and stereoselectivities (Scheme 1).

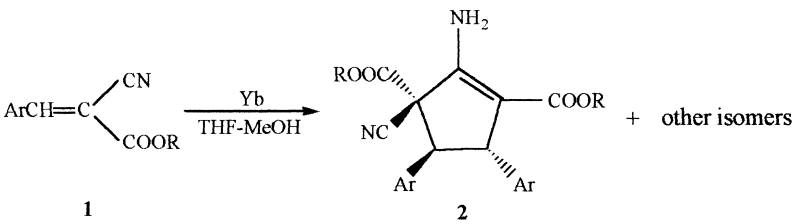
Results and Discussion

Arylmethylenecyanoacetates have sufficient reactivity to complete reductive dimerization cyclization in the presence of metallic ytterbium due to their carbon-carbon double bonds being activated by attached electron-withdrawing cyano and alkoxycarbonyl groups and the stability of five-membered ring products. Various reactions were examined to obtain the optimum condition of the ytterbium metal-mediated reductive dimerization cyclization of arylmethylenecyanoacetates. The results are summarized in Table 1.

We have found that (see Table 1) ethyl benzylidenecyanoacetate can be reduced to the corresponding functionalized cyclopentenes without any additives, but the reactions need a longer time for completion, and the yields are moderate. Because additives are added to the reaction system, they not only accelerate the reaction, but also increase the yields. Although it seemed that HMPA is the most effective among the additives, HMPA is a suspected cancer reagent. We use MeOH as an additive to meet the demand of green chemistry. Also, 1 equivalent of metallic ytterbium is sufficient to complete the reaction as for 2.5 equivalents of the substrate (entry 7); increasing the quantity of metallic ytterbium scarcely improved the yields of products (entries 8, 9, 10).

Our results concerning the metallic ytterbium-mediated reductive dimerization cyclization of arylmethylenecyanoacetates under the optimized condition (MeOH as an additive; Yb/substrate = 2/5) are listed in Table 2.

The four products of stereoisomeric cyclic hydro-dimers could not be separated by TLC. Fortunately, the *trans*, *trans*-isomers could be separated as pure compounds from a mixture of its isomers by the fractional crystallization method. All products could satisfactorily give IR, ^{1}H NMR, MS, and EA. Strong IR absorption at 1660 cm^{-1} is evidence for the CH–C=NH $\rightleftarrows$ C=C–NH_2 entity. Characteristic N–H stretching bands were also present and, in NMR experiments, NH protons were observed in the range of 5.36–6.40 ppm and exchanged in D_2O. Molecular formulate was derived from the MS spectra on molecular ions and CHN elemental analysis.



1 2

Scheme 1.

Table 1. Effects of Additives[a] and the Amount of Metallic Ytterbium[b] on the Reductive Dimerization Cyclization Reaction Using Ethyl Benzylidenecyanoacetate as a Model Substrate

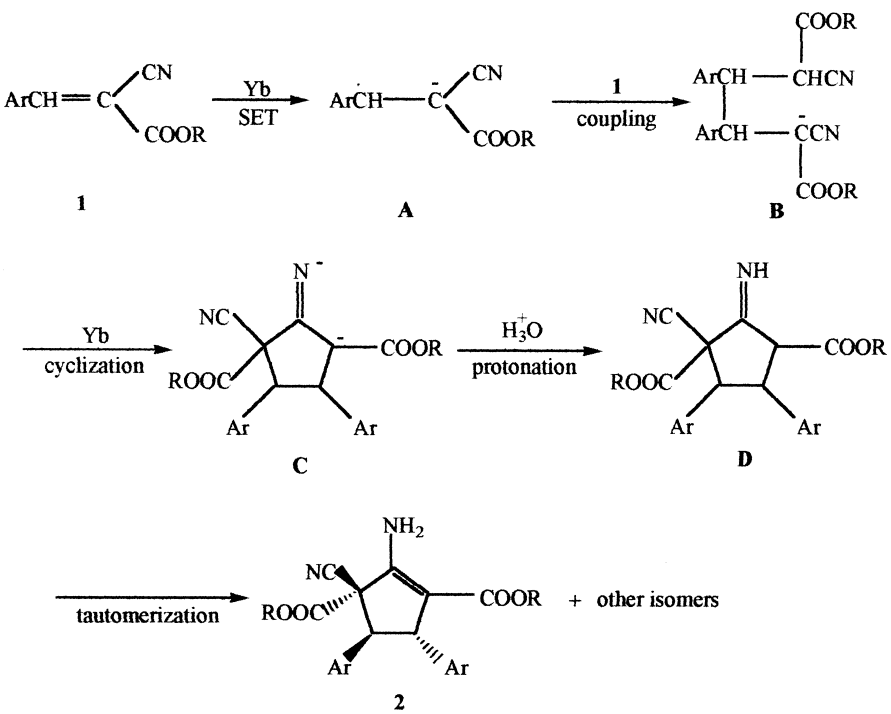
Entry	Additive	Time/h	Yield/%[c]	Entry	Yb/substrate	Yield/%[c]
1	—	6	56	6	1/10	17
2	MeOH	2	80	7	2/5	80
3	*t*-BuOH	3	77	8	3/4	81
4	HMPA	2	83	9	1/1	81
5	TMSCl	3	73	10	3/2	80

a) Carried out in THF-Additive (4:1, 5 mL) using a substrate (2.5 mmol) and ytterbium metal (1 mmol) at r.t. b) Carried out in THF–MeOH (4:1, 3 mL) using a substrate (1 mmol) at r.t. c) Combined isolated yields based on ethyl benzylidenecyanoacetate.

Table 2. Metallic Ytterbium Mediated Reductive Dimerization Cyclization of Arylmethylenecyanoacetates

Entry	Ar	R	Time/h	Yield/%[a]	**2**:other isomers[b]
1	C_6H_5	C_2H_5	2	80	86 (**2a**):14
2	4-ClC_6H_4	CH_3	3	85	88 (**2b**):12
3	4-BrC_6H_4	C_2H_5	2	86	90 (**2c**):10
4	3-BrC_6H_4	*i*-C_3H_7	3	88	86 (**2d**):14
5	4-$CH_3OC_6H_4$	C_2H_5	4	76	85 (**2e**):15
6	3,4-$OCH_2OC_6H_3$	C_2H_5	4	73	88 (**2f**):12
7	2-furyl	C_2H_5	3	83	88 (**2g**):12
8	2-thienyl	C_2H_5	3	88	89 (**2h**):11
9	4-$CH_3C_6H_4$	*i*-C_3H_7	4	78	82 (**2i**):18
10	4-$CH_3OC_6H_4$	CH_3	3	79	89 (**2j**):11

a) Combined isolated yields based on arylmethylenecyanoacetates. b) Ratios determined by ^{1}H NMR.



Scheme 2.

A possible mechanism is shown in Scheme 2. In the initial step, that an electron is transferred from metallic ytterbium to the substrate **1** by a single-electron transfer (SET) process results in the formation of a radical anion **A**; radical anion **A** then attacks another substrate to form a carbon-carbon bond and generates a dianion **B**. The latter reacts intramolecularly to re-

sult in the formation of another carbon-carbon bond and to produce intermediate **C**. Then, form **C** would be transformed to D, and isomerized to product **2** and other isomers.

Unfortunately, when substrates **1** derived from aromatic ketones, aliphatic aldehydes, and ketones were used, no reductive dimerization cyclization product was isolated. Probably, this result can be attributed to the low stability of the radical anion intermediates.

In consulusion, ytterbium metal can be used for the reductive dimerization cyclization of arylmethylenecyanoacetates to afford functionalized cyclopentenes in both high yields and stereoselectivities. Further studies on the applications of ytterbium metal in organic synthesis are now in progress.

Experimental

Metallic ytterbium, aldehydes, alkyl cyanoacetates, and all solvents, except for tetrahydrofuran, were purchased from commercial sources and used without purification. Arylmethylenecyanoacetates were synthesized by the reaction of carbonyl compounds and alkyl cyanoacetates following the usual procedure.[7] Tetrahydrofuran (THF) was distilled from sodium/benzophenone immediately prior to use. All reactions were carried on under a dry-nitrogen atmosphere. The melting points were uncorrected. Infrared spectra were recorded on a Perkin-Elmer 683 spectrometer in KBr with absorptions in cm^{-1}. ^1H NMR spectra were determined on a Bruker AC-400 instrument with $CDCl_3$ used as the solvent. The chemical shifts are expressed in ppm downfield from internal tetramethylsilane. Mass spectra were recorded on a HP 5989B MS spectrometer. A microanalysis was carried out on a Carlo-Erba 1106 instrument.

General Procedure: In a 25 mL flask were placed Yb metal powder (40 mesh, 1 mmol) and a magnetic stirring bar. The flask was sealed with a serum cap. Pure, dry nitrogen was then passed through, and the metal was dried under a stream of nitrogen. Methyl iodide (2 μL) was added by a microsyringe, and the metal was slightly heated by a heat gun to activate the Yb metal. The addition of THF (4 mL) gave a pale-yellow slurry, to which MeOH (1 mL) was introduced. Arylmethylenecyanoacetates (2.5 mmol) in THF (3 mL) were added. The mixture was then stirred at room temperature for a given time (see Table 2). The products were treated with 0.1 M HCl (5 mL), and then extracted with diethyl ether (3 × 30 mL). The combined organic extracts were washed with brine and dried over anhydrous $MgSO_4$. After the solvent was evaporated under reduced pressure, the crude products were purified by preparative TLC on silica gel using ethyl acetate–cyclohexane (1:4) as an eluent. Pure compounds **2** were obtained after fractional crystallization of the mixture with suitable solvents (the mixture of ether, chloroform and petroleum ether).

Diethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-diphenyl-1-cyclopentene-1, 3-dicarboxylate (2a). Mp 173–174 °C; IR (KBr) 3424, 3332, 3200, 3026, 2986, 2934, 2900, 2250, 1736, 1677, 1642, 1578, and 1455 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.86 (3H, t, J = 6.6 Hz, CH_3), 1.28 (3H, t, J = 7.0 Hz, CH_3), 3.70–4.21 (3H, m, CH_2 and CH), 4.26–4.66 (3H, m, CH_2 and CH), 5.98 (2H, br s, NH_2), and 7.10–7.80 (10H, m, ArH); MS m/z (%) 404 (M^+, 36), 403 (100), 374 (37), 332 (26), 331 (85), 312 (28), 311 (39), 286 (36), 285 (84), 257 (55), 181 (13), 131 (27), and 130 (30); Found: C, 71.22; H, 6.10; N, 6.06%. Calcd for $C_{24}H_{24}N_2O_4$: C, 71.27; H, 5.98; N, 6.93%.

Dimethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-bis(4-chlorophenyl)-1-cyclopentene-1,3-dicarboxylate (2b). Mp 175–177 °C; IR (KBr) 3450, 3350, 2250, 1766, 1685, 1645, 1575, 1500, 1450, 1300, 1220, 1090, 1015, and 820 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.39 (3 H, s, OCH_3), 3.72 (1H, d, J = 8.0 Hz, CH), 3.81 (3H, s, OCH_3), 4.30 (1H, d, J = 8.0 Hz, CH), 5.94 (2H, br s, NH_2), and 6.94–7.30 (8H, m, ArH); MS m/z (%) 444 (M^+, 100), 443 (29), 413 (43), 388 (12), 386 (62), 384 (94), 352 (72), 324 (58), 288 (37), 255 (32), 215 (27), 189 (37), 164 (61), and 155 (37); Found: C, 59.39; H, 4.12; N, 6.22%. Calcd for $C_{22}H_{18}Cl_2N_2O_4$: C, 59.34; H, 4.07; N, 6.29%.

Diethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-bis(4-bromophenyl)-1-cyclopentene-1,3-dicarboxylate (2c). Mp 190–193 °C; IR (KBr) 3424, 3328, 3201, 3045, 2990, 2944, 2908, 2250, 1737, 1674, 1636, 1582, and 1490 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.93 (3H, t, J = 6.8 Hz, CH_3), 1.33 (3 H, t, J = 7.0 Hz, CH_3), 3.66–4.11 (3H, m, CH_2 and CH), 4.16–4.63 (3H, m, CH_2 and CH), 6.06 (2H, br s, NH_2), and 6.77–7.80 (8H, m, ArH) MS m/z (%) 560 (M^+, 50), 533 (39), 491 (47), 489 (86), 487 (45), 445 (38), 443 (70), 435 (39), 334 (29), 255 (35), 210 (44), and 208 (47); Found: C, 51.34; H, 3.80; N, 5.06%. Calcd for $C_{24}H_{22}Br_2N_2O_4$: C, 51.27; H, 3.94; N, 4.98%.

Diisopropyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-bis(3-bromophenyl)-1-cyclopentene-1,3-dicarboxylate (2d). Mp 130–132 °C; IR (KBr) 3450, 3360, 3280, 3000, 2250, 1750, 1685, 1650, 1580, 1480, 1380, 1275, and 1100 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.73 (3H, d, J = 6.0 Hz, CH_3), 1.10 (3H, d, J = 6.0 Hz, CH_3), 1.30 (3H, d, J = 6.0 Hz, CH_3), 1.36 (3H, d, J = 6.0 Hz, CH_3), 3.93 (1H, d, J = 8.0 Hz, CH), 4.36 (1H, d, J = 8.0 Hz, CH), 4.90 (1H, m, J = 6.0 Hz, OCH), 5.20 (1H, m, J = 6.0 Hz, OCH), 6.07 (2H, br s, NH_2), and 7.03–7.80 (8H, m, ArH); MS m/z (%) 588 (M^+, 2), 547 (8), 505 (10), 503 (16), 461 (16), 208 (15), 171 (23), 169 (26), and 43 (100); Found: C, 52.80; H, 4.60; N, 4.95%. Calcd for $C_{26}H_{26}Br_2N_2O_4$: C, 52.71; H, 4.63; N, 4.99%.

Diethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-bis(4-methoxyphenyl)-1-cyclopentene-1,3-dicarboxylate (2e). Mp 160–163 °C; IR (KBr) 3400, 3326, 3242, 3190, 3025, 2837, 2250, 1737, 1644, 1638, 1567, and 1460 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.93 (3H, t, J = 6.9 Hz, CH_3), 1.30 (3H, t, J = 7.0 Hz, CH_3), 3.77–4.32 (9H, m, CH_2 , CH and 2 × OCH_3), 4.27–4.55 (3 H, m, CH_2 and CH), 6.08 (2H, br s, NH_2), and 6.73–7.79 (8H, m, ArH) MS m/z (%) 464 (M^+, 47), 418 (16), 390 (28), 373 (20), 372 (35), 344 (28), 317 (19), 237 (10), 233 (14), 232 (53), 186 (12), 161 (33), 160 (79), and 159 (15); Found: C, 67.28; H, 6.18; N, 6.05%. Calcd for $C_{26}H_{28}N_2O_6$: C, 67.23; H, 6.08; N, 6.03%.

Diethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-di(3,4-methylenedioxyphenyl)-1-cyclopentene-1,3-dicarboxylate (2f). Mp 188–189 °C; IR (KBr) 3422, 3322, 3185, 3070, 2987, 2918, 2882, 2248, 1740, 1660, 1630, 1562, and 1490 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.94 (3H, t, J = 7.0 Hz, CH_3), 1.30 (3H, t, J = 7.2Hz, CH_3), 3.90–4.42 (3H, m, CH_2 and CH), 4.52–4.90 (3H, m, CH_2 and CH), 6.25 (2H, s, OCH_2O), 6.35 (2H, s, OCH_2O), 6.40 (2H, br s, NH_2), and 6.93–7.70 (6H, m, ArH); MS m/z (%) 492 (M^+, 87), 463 (100), 419 (34), 399 (44), 372 (41), 344 (23), 297 (11), 268 (16), 246 (53), 202 (23), 174 (97), and 144 (32); Found: C, 63.50; H, 4.88; N, 5.60%. Calcd for $C_{26}H_{24}N_2O_8$: C, 63.41; H, 4.91; N, 5.69%.

Diethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-di(2-furyl)-1-cyclopentene-1,3-dicarboxylate (2g). Mp 130–133 °C; IR (KBr): 3412, 3327, 3204, 2983, 2938, 2875, 2246, 1743, 1677, 1630, 1582, and 1465 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.80 (3H, t, J = 6.8 Hz, CH_3), 1.28 (3H, t, J = 7.0 Hz, CH_3), 3.80–4.12 (3H, m, CH_2 and CH), 4.26–4.50 (3H, m, CH_2 and CH), 5.93 (2H, br s, NH_2), 6.12–6.40 (4H, m, ArH), and 7.10–7.50 (2H, m, ArH);

MS *m*/*z* (%) 384 (M^+, 37), 383 (100), 354 (24), 337 (30), 310 (90), 292 (23), 266 (21), 265 (91), 243 (23), 238 (20), 237 (97), and 164 (21); Found: C, 62.40; H, 5.38; N, 7.40%. Calcd for $C_{29}H_{20}N_2O_6$: C, 62.49; H, 5.24; N, 7.29%.

Diethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-di(2-thienyl)-1-cyclopentene-1,3-dicarboxylate (2h). Mp 138–139 °C; IR (KBr): 3420, 3322, 3205, 2980, 2938, 2900, 2876, 2248, 1746, 1677, 1630, 1582, and 1469 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.95 (3 H, t, J = 7.0 Hz, CH_3), 1.25 (3 H, t, J = 7.0 Hz, CH_3), 3.86–4.12 (3 H, m, CH_2 and CH), 4.27–4.70 (3 H, m, CH_2 and CH), 5.36 (2 H, br s, NH_2), and 6.93–7.53 (6 H, m, ArH); MS *m*/*z* (%) 417 (M + 1, 11), 416 (M^+, 37), 387 (6), 370 (17), 343 (31), 324 (15), 298 (18), 297 (39), 269 (26), 187 (14), 149 (63), 142 (27), 141 (32), 137 (39), 136 (69), 97 (82), 86 (59), and 84 (100); Found: C, 57.77; H, 4.88; N, 5.75%. Calcd for $C_{20}H_{20}N_2O_4S_2$: C, 57.69; H, 4.81; N, 5.77%.

Diisopropyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-bis(4-methylphenyl)-1-cyclopentene-1,3-dicarboxylate (2i). Mp 152–155 °C; IR (KBr) 3460, 3350, 3270, 3210, 3000, 2250, 1750, 1645, 1580, 1520, 1480, 1380, 1100, and 830 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.70 (3 H, d, J = 6.0 Hz, CH_3), 1.10 (3 H, t, J = 6.0 Hz, CH_3), 1.30 (3 H, d, J = 6.0 Hz, CH_3), 1.33 (3 H, d, J = 6.0 Hz, CH_3), 2.20 (3 H, s, CH_3), 2.30 (3 H, s, CH_3), 3.96 (1 H, d, J = 8.0 Hz, CH), 4.46 (1 H, d, J = 8.0 Hz, CH), 4.87 (1 H, m, J = 6.0 Hz, OCH), 5.20 (1 H, m, J = 6.0 Hz, OCH), 5.90 (2 H, br s, NH_2), and 7.00–7.65 (8 H, m, ArH); MS *m*/*z* (%) 461 (M + 1, 30), 460 (M^+, 33), 417 (55), 372 (42), 330 (41), 312 (48), 285 (22), 221 (12), 195 (14), 144 (29), 115 (17), 91 (8), and 43 (100); Found: C, 73.08; H, 6.89; N, 6.13%. Calcd for $C_{28}H_{32}N_2O_4$: C, 73.02; H, 7.00; N, 6.08%.

Dimethyl 3,4-*trans*-4,5-*trans*-2-Amino-3-cyano-4,5-bis(4-methoxyphenyl)-1-cyclopentene-1,3-dicarboxylate (2j). Mp 170–172 °C; IR (KBr) 3440, 3345, 3280, 3220, 2970, 2950, 2250, 1760, 1690, 1645, 1585, 1520, 1440, 1180, and 1030 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.40 (3 H, s, OCH_3), 3.60–3.80 (10 H, m, 3 × OCH_3 and CH), 4.31 (1 H, d, J = 8.0 Hz, CH), 5.85 (2 H, br s, NH_2), and 6.90–7.30 (8 H, m, ArH); MS *m*/*z* (%) 437 (M + 1, 34), 436 (M^+, 100), 403 (34), 376 (60), 371 (65), 344 (52), 328 (48), 316 (33), 218 (38), 174 (25), 160 (52), 121 (55), and 59 (35); Found: C, 66.08; H, 5.59; N, 6.50%. Calcd for $C_{24}H_{24}N_2O_6$: C, 66.04; H, 5.54; N, 6.42%.

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