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Electroreductive intramolecular coupling of *N*-(oxoalkyl)phthalimides: complementary method to samarium(II) iodide reduction

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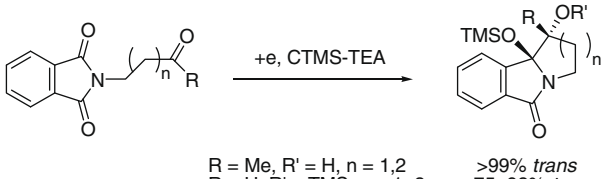
ABSTRACT

The electroreductive intramolecular coupling of phthalimides with ketones in the presence of chlorotrimethylsilane gave five- and six-membered trans-cyclized products stereospecifically (>99%). Similar electroreductive intramolecular coupling of phthalimides with aldehydes afforded five-, six-, and seven-membered trans-cyclized products stereoselectively (75–93%). On the other hand, the reductive coupling of *N*-(oxoalkyl)phthalimides with samarium(II) iodide gave cis-cyclized products stereoselectively (88–>99%).

The reductive cross coupling between different types of carbonyl compounds is a useful method for the synthesis of adjacent difunctional compounds such as 1,2-diols and α-hydroxyketones. Recently, a variety of these types of reactions were realized using samarium(II) iodide as a reducing agent.[1] On the other hand, electroreduction can accomplish the reductive cross coupling of carbonyl compounds without using the expensive rare-earth metal reagent. For instance, we have reported the electroreductive coupling of aromatic ketones with aliphatic ketones and aldehydes,[2] esters,[3] and acylimidazoles[4] in the presence of chlorotrimethylsilane (CTMS). In this context, we report herein that the electroreductive intramolecular coupling of phthalimides with aliphatic ketones and aldehydes in the presence of CTMS produced tricyclic compounds incorporating an isoindolinone ring (Scheme 1). It is noted that trans-dihydroxylated cyclized products were formed stereospecifically (R = Me: >99%) or stereoselectively (R = H: 75–93%) by the electroreduction of *N*-(oxoalkyl)phthalimides. The inter-[5] and intramolecular[6] reductive cross couplings of phthalimides with aldehydes[5,6a] and ketones[5,6b] using samarium(II) iodide have recently been reported. To compare with the electrochemical method, we also tried the reductive coupling of *N*-(oxoalkyl)phthalimides with samarium(II) iodide and found that cis-cyclized products were obtained stereoselectively (R = Me, H: 88–>99%). Therefore, the two reductive methods brought about complemental stereochemical results in the reductive cyclization of *N*-(oxoalkyl)phthalimides.

First, conditions for the electroreductive intramolecular coupling were surveyed with 2-(3-oxobutyl)isoindoline-1,3-dione (**1**) as a substrate and the results are summarized in Table 1. According to the reported procedure,[2–4] the electroreduction of **1** (1 mmol) was carried out in 0.3 M solution of a tetraalkylammonium salt in an aprotic polar solvent such as acetonitrile (15 mL) at a constant current of 100 mA (300 C) employing a divide cell. In the absence of additive, only a complex mixture was obtained (run 1). In the presence of CTMS, no cyclized product was detected and a simply reduced hydroxy lactam **i** and its dehydroxy derivative **ii** were isolated in 42% and 28% yields, respectively (run 2). These results show that the presence of CTMS and TEA is essential for the electroreductive cyclization of **1**. Although the change of supporting electrolyte using acetonitrile as a solvent had a slight effect on the yield of a five-member cyclized product **2** (runs 3–5), the use of DMF and THF in place of acetonitrile decreased the yield of **2** to some extent (runs 6–8). As a cathode material, Pb gave better results than Pt, Ag, Zn, and Sn (runs 3, 9–12). Consequently, the best



Scheme 1.

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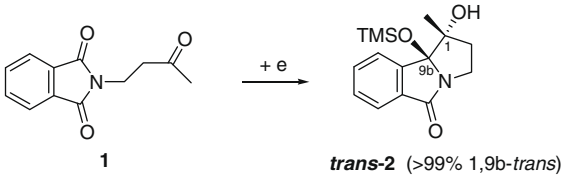
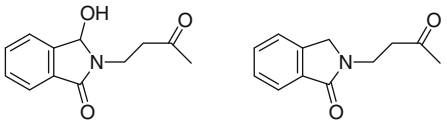
Table 1
Electroreductive intramolecular coupling of **1**



1 **trans-2** (>99% 1,9b-*trans*)

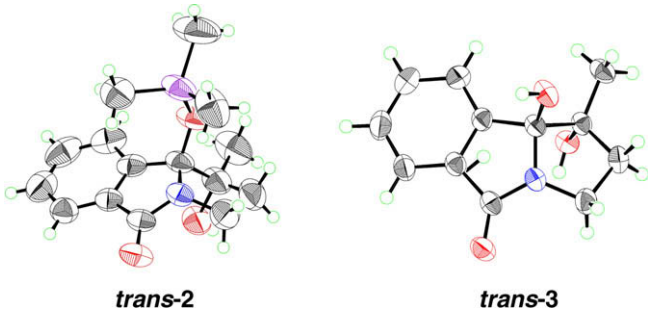
Run	Additive[a]	Catholyte[b]	Cathode material	% Yield of ***trans*-2**[c]
1	None	Et_4NOTs/CH_3CN	Pb	0
2	CTMS	Et_4NOTs/CH_3CN	Pb	0[d]
3	CTMS/TEA	Et_4NOTs/CH_3CN	Pb	71
4	CTMS/TEA	Et_4NBr/CH_3CN	Pb	66
5	CTMS/TEA	Bu_4NClO_4/CH_3CN	Pb	65
6	CTMS/TEA	Et_4NOTs/DMF	Pb	55
7	CTMS/TEA	Bu_4NClO_4/THF	Pb	44
8	CTMS/TEA	Bu_4NPF_6/THF	Pb	45
9	CTMS/TEA	Et_4NOTs/CH_3CN	Pt	52
10	CTMS/TEA	Et_4NOTs/CH_3CN	Ag	52
11	CTMS/TEA	Et_4NOTs/CH_3CN	Zn	62
12	CTMS/TEA	Et_4NOTs/CH_3CN	Sn	40

[a] 5 equiv.
[b] 0.3 M electrolyte in solvent.
[c] Isolated yields.
[d] Simply reduced hydroxy lactam **i** and its dehydroxy variant **ii** were obtained.



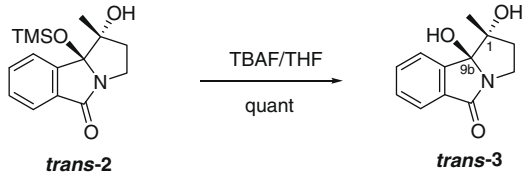
i (42%) **ii** (28%)

yield of **2** (71% yield) was obtained using Et_4NOTs/acetonitrile as a catholyte and a Pb cathode in the presence of CTMS and TEA (run 3).[7] It was found by ^1H NMR analysis that the cyclized product **2** was formed as a mono-trimethylsilyl ether stereospecifically (>99%). The 1,9b-trans stereochemistry and 9b-trimethylsilyloxy group in **2** (designated as ***trans*-2**) were undoubtedly confirmed by X-ray crystallographic analysis (Fig. 1).[8] The mono-silyl ether



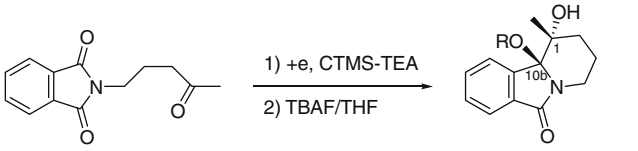
***trans*-2** ***trans*-3**

Figure 1. X-ray crystal structures of ***trans*-2** and ***trans*-3**.



***trans*-2** ***trans*-3**

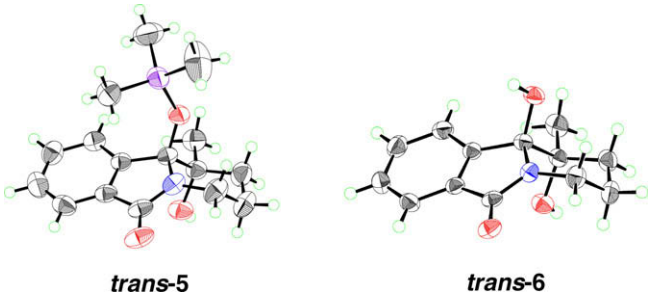
Scheme 2.



4

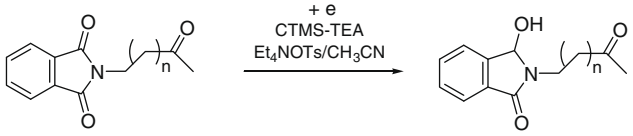
(>99% 1,10b-*trans*)
***trans*-5** (R = TMS) 77%
***trans*-6** (R = H) quant

Scheme 3.



***trans*-5** ***trans*-6**

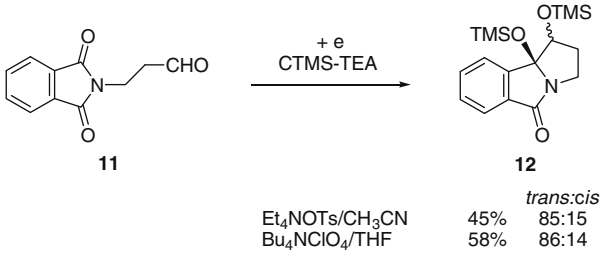
Figure 2. X-ray crystal structures of ***trans*-5** and ***trans*-6**.



7 (n = 0)
9 (n = 3)

8 (n = 0) 53%
10 (n = 3) 46%

Scheme 4.

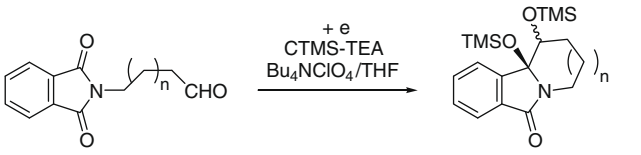


11 **12**

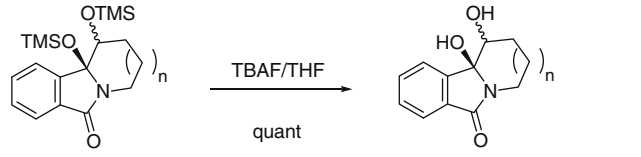
		trans:*cis*
Et_4NOTs/CH_3CN	45%	85:15
Bu_4NClO_4/THF	58%	86:14

Scheme 5.

***trans*-2** was readily desilylated by treatment with TBAF in THF to give the corresponding diol ***trans*-3** quantitatively (Scheme 2). The complete retention of the 1,9b-trans configuration in ***trans*-3** was ascertained by X-ray crystallography (Fig. 1).[8]



Scheme 6.



Scheme 7.

The electroreduction of 2-(4-oxopentyl)isoindoline-1,3-dione (**4**) under the same conditions as run 3 in Table 1 gave a six-membered cyclized product ***trans*-5** in 77% yield stereospecifically (Scheme 3). The stereostructure of ***trans*-5** was determined by X-ray crystallography (Fig. 2);[8] the 1,10b-trans configuration and 10b-trimethylsililoxy substitution were assigned to ***trans*-5**. Desilylation of ***trans*-5** with TBAF gave 1,10b-trans diol ***trans*-6** quantitatively.

Unfortunately, the electroreduction of 2-(2-oxopropyl)isoindoline-1,3-dione (**7**) and 2-(5-oxohexyl)isoindoline-1,3-dione (**9**) afforded simply reduced hydroxy lactams **8** and **10**, respectively; four- and seven-membered cyclized products were not detected at all (Scheme 4). These hydroxy lactams **8** and **10** were obtained by desilylation of initially formed silyl ethers during isolation.

Next, we attempted the electroreduction of 3-(1,3-dioxoisoindolin-2-yl)propanal (**11**). The yield of a five-membered cyclized product **12** was better in Bu_4NClO_4/THF (58% yield, trans:cis = 86:14) than in Et_4NOTs/CH_3CN (45% yield, trans:cis = 85:15) as a catholyte (Scheme 5). Therefore, the electroreduction of 4-(1,3-dioxoisoindolin-2-yl)butanal (**13**) and 5-(1,3-dioxoisoindolin-2-yl)pentanal (**15**) was carried out in Bu_4ClO_4/THF to give six- and seven-membered cyclized products **14** (67% yield, trans:cis = 75:25) and **16** (34% yield, trans:cis = 93:7), respectively (Scheme 6). The ^1H NMR analysis of **12**, **14**, and **16** showed that these cyclized products were obtained as di-trimethylsilyl ethers and mixtures of two diastereomers (trans and cis), in contrast to **2** and **5** described above. After separation of the diastereomers of **12** and **14** by column chromatography, desilylation of each isomer with TBAF/THF gave the corresponding diol **17** or **18** quantitatively (Scheme 7). The stereostructures of both trans and cis isomers of **17** and **18** were confirmed by X-ray crystallography (Fig. 3).[8] Although the diastereomers of **16** could not be separated, the

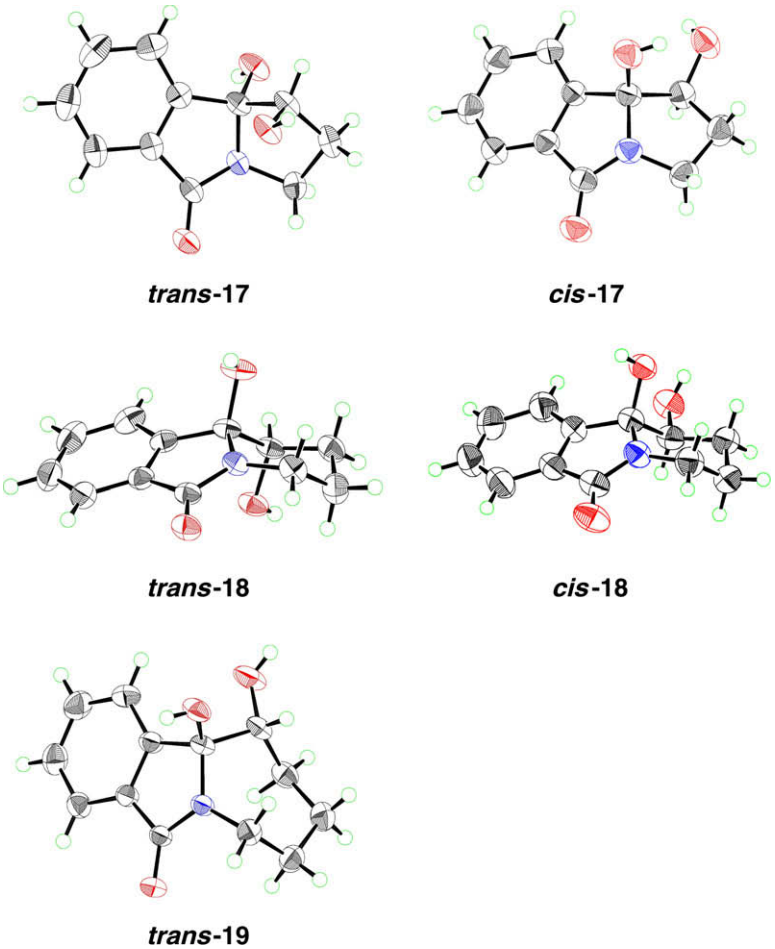
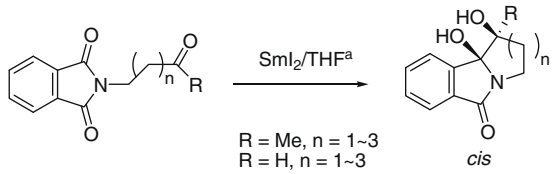


Figure 3. X-ray crystal structures of **17**, **18,** and **19**.

Table 2
Reductive cyclization of *N*-(oxoalkyl)phthalimides with samarium(II) iodide



Run	R	*n*	Product	% Yield[b]	cis:trans[c]
1	Me	1	**3**	70	88:12
2	Me	2	**6**	70	>99:1
3	Me	3	**20**	67	>99:1
4	H	1	**17**	34	93:7
5	H	2	**18**	28	>99:1
6	H	3	**19**	—[d]	

[a] The reaction was carried out with 2 equiv of SmI_2 at room temperature.
[b] Isolated yields.
[c] Determined by ^{1}N NMR.
[d] Compound **19** could not be obtained.

major isomer of the desilylated diol **19** could be isolated by recrystallization and identified to be trans by X-ray crystallography (Fig. 3).[8]

The reductive cyclization of **11**, **13**, and **15** with samarium(II) iodide has been reported,[6a] but the stereoselectivities of the cyclized products **17**–**19** were not investigated. Therefore, we tried the reduction of *N*-(oxoalkyl)phthalimides **1**, **4**, **9**, **11**, **13**, and **15** with samarium(II) iodide (Table 2). As expected,[9] cis-cyclized products of **3**, **6**, **17**, **18**, and **20** were obtained as major or predominant isomers in all cases (88–>99% stereoselectivities), although **19** could not be obtained from **15**. The stereostructures of cis-isomers of **3**, **6**, and **20** were determined by NMR analysis for ***cis*-3**[10] or by X-ray crystallography for ***cis*-6** and ***cis*-20** (Fig. 4).[8]

The electroreductive intramolecular coupling of *N*-(oxoalkyl)-phthalimides seems to be initiated by the reduction of the phthalimide moiety, according to the results shown in Scheme 4. To ascertain this assumption, we measured the cyclic voltammetry of *N*-methylphthalimide, **1**, and acetone in 0.03 M Bu_4NClO_4/DMF on a Pt cathode: the cyclic voltamograms of *N*-methylphthalimide and **1** (3 mM) showed a reduction peak at −1.53[11] and −1.50 V versus SCE, respectively, whereas that of acetone gave no reduction peak from 0 to −3.0 V versus SCE. These observations suggest that the phthalimide moiety is much more reducible than the keto carbonyl group in **1**. Hence, the reaction mechanism of the electroreductive coupling of **1** can be presumed as illustrated in Scheme 8. Anion A is formed from **1** by a two-electron transfer and following O-silylation. The carbanion in **A** attacks the keto carbonyl group intramolecularly through transition state **TS**. Since ***cis*-TS** is more unfavorable than ***trans*-TS** because of the electronic repulsion between the two oxygen atoms, ***trans*-2** is formed predominantly through subsequent protonation of resulting tertiary O-anion **B**.

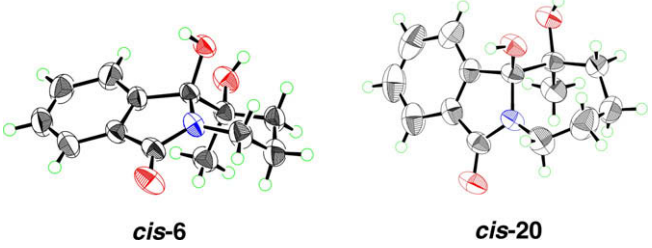
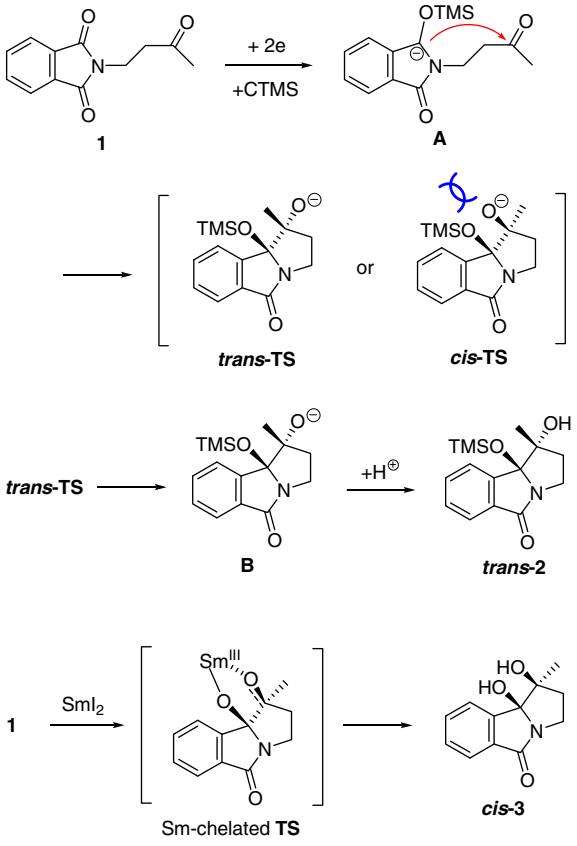


Figure 4. X-ray crystal structures of ***cis*-6** and ***cis*-20**.



Scheme 8.

In the case of the reductive coupling of an aldehyde variant **11**, silylation of resulting secondary O-anion allows the formation of di-trimethylsilyl ether **12**. On the contrary, the reduction of *N*-(oxoalkyl)phthalimides with samarium(II) iodide prefers cis-cyclization probably owing to the chelation of the two oxygen atoms to the samarium(III) atom in the transition state.[9a,b]

In conclusion, the electroreduction of *N*-(oxoalkyl)phthalimides **1** and **4** in the presence of CTMS and TEA stereospecifically gave five- and six-membered trans-cyclized products ***trans*-2** and ***trans*-5**, respectively. The electroreduction of (1,3-dioxoisoindolin-2-yl)alkanals **11**, **13**, and **15** in the presence of CTMS and TEA afforded five-, six-, and seven-membered cyclized products **12**, **14**, and **16** with trans-stereoselectivity. In contrast to trans-selective cyclization in the electroreduction, the reduction of *N*-(oxoalkyl)phthalimides with samarium(II) iodide resulted in cis-selective cyclization.

References and notes

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7. Typical procedure for electroreduction (Table 1, run 3) is as follows. A 0.3 M solution of Et_4NOTs in CH_3CN (15 mL) was placed in the cathodic chamber of a divided cell (40-mL beaker, 3-cm diameter, 6-cm height) equipped with a lead

cathode (5×5 cm^2), a platinum anode (2×1 cm^2), and a ceramic cylindrical diaphragm (1.5-cm diameter). A 0.3 M solution of Et_4NOTs in DMF (4 mL) was placed in the anodic chamber (inside the diaphragm). 2-(3-Oxobutyl)isoindoline-1,3-dione (**1**) (217 mg, 1 mmol), CTMS (0.64 mL, 5 mmol), and triethylamine (0.70 mL, 5 mmol) were added to the cathodic chamber. After 300 C of electricity was passed at a constant current of 100 mA at room temperature, the catholyte was evaporated in vacuo. The residue was dissolved in ethyl acetate (20 mL) and insoluble Et_4NOTs was filtered off. After the solvent was removed, the crude mixture was purified by column chromatography on silica gel (hexanes–ethyl acetate) to give ***trans*-2** in 71% yield. ***trans*-2**: White solid. R_f 0.55 (hexanes–ethyl acetate, 1:1). Mp 116–117 °C. ^{1}H NMR ($CDCl_3$) δ −0.12 (q, 9 H), 0.92 (br s, 1H), 1.56 (s, 3H), 2.16–2.22 (m, 1H), 2.48–2.56 (m, 1H), 3.33–3.39 (m, 1H), 3.73–3.86 (m, 1H), 7.48–7.52 (m, 2H), 7.55–7.59 (m, 1H), 7.73–7.76 (m, 1H). ^{13}C NMR ($CDCl_3$) δ 0.5 (q), 21.0 (q), 39.5 (t), 40.6 (t), 79.0 (s), 100.0 (s), 122.3 (d), 123.6 (d), 129.8 (d), 132.2 (d), 134.3 (s), 144.0 (s), 171.5 (s).

8. All measurements of X-ray crystallographic analysis were made on a Rigaku RAXIS imaging plate area detector with graphite-monochromated Mo Kα radiation. The structure was solved by direct methods with SIR-97 and refined with SHELXL-97. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. All calculations were performed using the YADOKARI-XG software package. Crystal data are as follows: CCDC 746359–746367 and 748636–748637 contain the supplementary crystallographic data. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

   ***trans*-2** (CCDC 746359): $C_{15}H_{21}NO_3Si$, FW = 291.42, mp 116–117 °C, triclinic, $P\bar{1}$ (no. 2), colorless block, a = 8.487(12) Å, b = 9.557(10) Å, c = 20.91(2) Å, α = 90.26 (5), β = 92.72(7), γ = 94.92 (7), V = 1688(3) Å^3, T = 298 K, Z = 4, D_{calcd} = 1.147 g/cm^3, μ = 1.45 cm^{-1}, GOF = 1.035.

   ***trans*-3** (CCDC 746360): $C_{12}H_{13}NO_3$, FW = 219.23, mp 177–179 °C, monoclinic, $C2/c$ (no. 15), colorless block, a = 31.481(4) Å, b = 11.6684(12) Å, c = 11.6388(8) Å, β = 92.307(7), V = 4271.9(7) Å^3, T = 298 K, Z = 16, D_{calcd} = 1.364 g/cm^3, μ = 0.99 cm^{-1}, GOF = 1.048.

   ***trans*-5** (CCDC 746361): $C_{16}H_{23}NO_3Si$, FW = 293.43, mp 125–127 °C, monoclinic, $P2_{1/a}$ (no. 14), colorless block, a = 18.892(4) Å, b = 7.974(3) Å, c = 18.548(4) Å, β = 36.492(9), V = 1661.7(8) Å^3, T = 298 K, Z = 4, D_{calcd} = 1.173 g/cm^3, μ = 1.48 cm^{-1}, GOF = 1.026.

   ***trans*-6** (CCDC 746362): $C_{13}H_{15}NO_3$, FW = 233.26, mp 250–252 °C, monoclinic, $P2_{1/c}$ (no. 14), colorless block, a = 8.3771(17) Å, b = 8.1937(19) Å, c = 16.750(3) Å, β = 94.114(10), V = 1146.7(4) Å^3, T = 298 K, Z = 4, D_{calcd} = 1.351 g/cm^3, μ = 0.96 cm^{-1}, GOF = 1.010.

   ***trans*-17** (CCDC 746363): $C_{11}H_{11}NO_3$, FW = 205.21, mp 170–172 °C, monoclinic, $P2_{1/n}$ (no. 14), colorless block, a = 9.456(2) Å, b = 10.608(2) Å, c = 9.8385(19) Å, β = 104.617(11), V = 955.0(4) Å^3, T = 298 K, Z = 4, D_{calcd} = 1.427 g/cm^3, μ = 1.05 cm^{-1}, GOF = 1.030.

   ***cis*-17** (CCDC 746364): $C_{11}H_{11}NO_3$, FW = 205.21, mp 175–177 °C, monoclinic, $P2_{1/n}$ (no. 14), colorless block, a = 7.0959(15) Å, b = 15.838(4) Å, c = 8.495(3) Å, β = 97.035(13), V = 947.5(4) Å^3, T = 298 K, Z = 4, D_{calcd} = 1.439 g/cm^3, μ = 1.06 cm^{-1}, GOF = 1.083.

   ***trans*-18** (CCDC 746365): $C_{12}H_{13}NO_3$, FW = 219.23, mp 180–182 °C, monoclinic, $P2_{1/n}$ (no. 14), colorless block, a = 9.5284(16) Å, b = 10.1851(13) Å, c = 11.093(2) Å, β = 102.153(7), V = 1052.4(3) Å^3, T = 298 K, Z = 4, D_{calcd} = 1.384 g/cm^3, μ = 1.00 cm^{-1}, GOF = 1.028.

   ***cis*-18** (CCDC 746366): $C_{12}H_{13}NO_3$, FW = 219.23, mp 182–184 °C, triclinic, $P\bar{1}$ (no. 2), colorless block, a = 8.681(3) Å, b = 14.366(5) Å, c = 17.580(5) Å, α = 90.68(3), β = 90.01(2), γ = 98.45(3), V = 2168.4(3) Å^3, T = 298 K, Z = 8, D_{calcd} = 1.343 g/cm^3, μ = 0.97 cm^{-1}, GOF = 1.043.

   ***trans*-19** (CCDC 746367): $C_{13}H_{15}NO_3$, FW = 233.26, mp 178–180 °C, triclinic, $P\bar{1}$ (no. 2), colorless block, a = 7.699(3) Å, b = 8.626(3) Å, c = 9.554(4) Å, α = 69.18(3), β = 74.31(4), γ = 75.44(2), V = 562.4(4) Å^3, T = 298 K, Z = 2, D_{calcd} = 1.377 g/cm^3, μ = 0.98 cm^{-1}, GOF = 1.037.

   ***cis*-3** (CCDC 748636): $C_{13}H_{15}NO_3$, FW = 233.26, mp 179–180 °C, monoclinic, $P2_{1/c}$ (no. 14), colorless block, a = 17.5138(16) Å, b = 9.3613(7) Å, c = 28.653(2) Å, β = 88.801(3), V = 4696.6(7) Å^3, T = 298 K, Z = 16, D_{calcd} = 1.320 g/cm^3, μ = 0.94 cm^{-1}, GOF = 1.025.

   ***cis*-20** (CCDC 748637): $C_{14}H_{17}NO_3$, FW = 247.29, mp 168–169 °C, orthorhombic, P_{bca} (no. 61), colorless block, a = 19.3585(13) Å, b = 8.1074(8) Å, c = 35.917(2) Å, V = 5637.1(8) Å^3, T = 298 K, Z = 16, D_{calcd} = 1.165 g/cm^3, μ = 0.82 cm^{-1}, GOF = 1.046.

9. cis-Selective intramolecular pinacol coupling of 1,ω-dicarbonyl compounds with samarium(II) iodide has been reported: (a) Molander, G. A.; Kenny, C. *J. Org. Chem.* **1988**, *53*, 2134–2138; (b) Chiara, J. L.; Cabri, W.; Hanessian, S. *Tetrahedron Lett.* **1991**, *32*, 1125–1128; (c) Hoffmann, H. M. R.; Münnich, I.; Nowitzki, O.; Stucke, H.; Williams, D. J. *Tetrahedron* **1996**, *52*, 11783–11798; (d) Nowitzki, O.; Münnich, I.; Stucke, H.; Hoffmann, H. M. R. *Tetrahedron* **1996**, *52*, 11799–11810.

10. ***trans*-3**: White solid. R_f 0.25 (hexanes–ethyl acetate, 1:2). Mp 177–179 °C. ^{1}H NMR ($CDCl_3$) δ 1.02 (br s, 1H), 1.60 (s, 3H), 2.21–2.28 (m, 1H), 2.60–2.69 (m, 1H), 2.91 (br s, 1H), 3.35–3.42 (m, 1H), 3.70–3.79 (m, 1H), 7.49–7.53 (m, 1H), 7.56–7.61 (m, 2H), 7.69–7.72 (m, 1H). ^{13}C NMR ($CDCl_3$) δ 21.2 (q), 40.0 (t), 40.2 (t), 78.4 (s), 99.1 (s), 122.2 (d), 123.8 (d), 130.2 (d), 132.5 (d), 134.5 (s), 143.4 (s), 171.0 (s). ***cis*-3**: White solid. R_f 0.25 (hexanes–ethyl acetate, 1:2). Mp 179–180 °C. ^{1}H NMR ($CDCl_3$) δ 0.80 (s, 3H), 2.22–2.28 (m, 1H), 2.61–2.70 (m, 1H), 3.05 (br s, 1H), 3.40–3.54 (m, 2H), 3.63 (br s, 1H), 7.47–7.51 (m, 1H), 7.55–7.61 (m, 2H), 7.64–7.66 (m, 1H). ^{13}C NMR ($CDCl_3$) δ 22.0 (q), 39.4 (t), 40.9 (t), 77.1 (s), 95.1 (s), 122.8 (d), 123.5 (d), 129.7 (d), 132.5 (d), 132.8 (s), 144.5 (s), 170.1 (s).

11. The reduction peak of *N*-methylphthalimide was reported to be −1.47 V versus SCE in Bu_4NPF_6/acetonitrile on a glassy carbon cathode: Warzecha, K.-D.; Gorner, H.; Griesbeck, A. G. *J. Phys. Chem. A* **2006**, *110*, 3356–3363.