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One Flask Synthesis of *t*-2,*c*-3-Disubstituted 5-Oxo-*r*-1-cyclopentanecarboxamides by a SmI_2/HMPA-Induced Stereoselective Reductive Coupling/Dieckmann Condensation Sequence of α,β-Unsaturated Amides

Hidetoshi YAMAMOTO,* Shigeru KOBAYASHI, and Shuji KANEMASA

α,β-Unsaturated amides undergo highly *dl*-selective reductive coupling reactions by the action of SmI_2-HMPA. Subsequent Dieckmann condensation is followed in the same flask when the amides bear small *N*-substituents: *N,N*-dibenzylamides afford 3,4-disubstituted *dl*-adipamides as reductive coupling products; *N,N*-dimethylamides give *t*-2,*c*-3-disubstituted 5-oxo-*r*-1-cyclopentanecarboxamides as Dieckmann condensation products, both in exclusively *dl*-selective manners.

As Inanaga and coworkers recently reported, α,β-unsaturated carbonyl compounds, when treated with samarium(II) iodide (SmI_2)/HMPA (hexamethylphosphoric triamide) in the presence of *t*-butyl alcohol in tetrahydrofuran (THF), undergo reductive coupling reactions to give 3,4-disubstituted 1,6-dicarbonyl derivatives.[1,2] Use of HMPA as co-solvent is important in this reaction since conjugate reduction takes place in the absence of HMPA.[3] Although α,β-unsaturated esters are highly reactive, stereoselectivity of the ester coupling reaction is usually poor to give mixtures of *dl*- and *meso*-isomers of adipate derivatives. On the other hand, the reaction using an unsaturated amide substrate, *N,N*-dibenzylcrotonamide, is exclusively stereoselective to give the *dl*-isomer of the corresponding adipamide as a single isomer, though this is the only example reported. Such dramatic change of stereoselectivities observed between α,β-unsaturated esters and amides is interesting from the mechanistic and synthetic viewpoints. However, no satisfactory explanation has been presented so far. Aspects and limitations of this stereoselective process remain unsolved.

During our synthetic work of novel chiral controlling ligands, we tried to apply Inanaga's method to our ligand synthesis. Our synthetic plan stands on the initial *dl*-selective synthesis of 3,4-disubstituted adipic acid derivatives, the Dieckmann type intramolecular condensation to cyclopentane skeletons, and subsequent steps for the adjustment of functional groups. In the present paper, we describe some examples of *dl*-selective SmI_2/HMPA-induced reductive coupling reactions using α,β-unsaturated amides. To be emphasized are the reactions of *N,N*-dimethylamide derivatives which directly lead to a convenient one flask synthesis of *t*-2,*c*-3-disubstituted 5-oxo-*r*-1-cyclopentanecarboxamides as a result of the *dl*- selective reductive coupling reaction followed by Dieckmann condensation.

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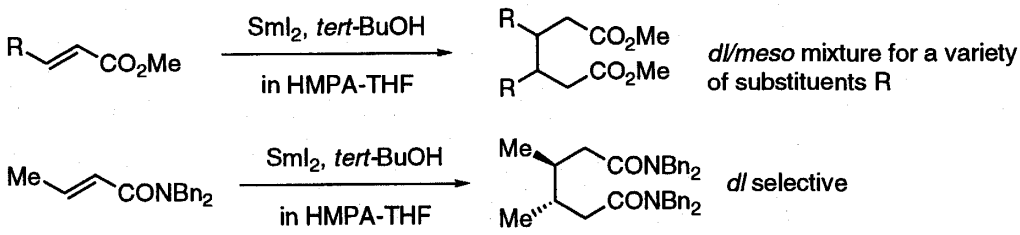
Dedicated to Professor Hiroshi Kobayashi on the occasion of his retirement



Scheme 1

Results and Discussion

Although SmI_2 is commercially available in THF solution, it can be more economically prepared by treatment of samarium metal with a variety of oxidizing agents such as 1,2-diiodoethane,[4] diiodomethane,[5] molecular iodine,[6] and TMSCl-NaI.[7] We adopted the Sm/CH_2I_2 method because of its simple experimental procedure.

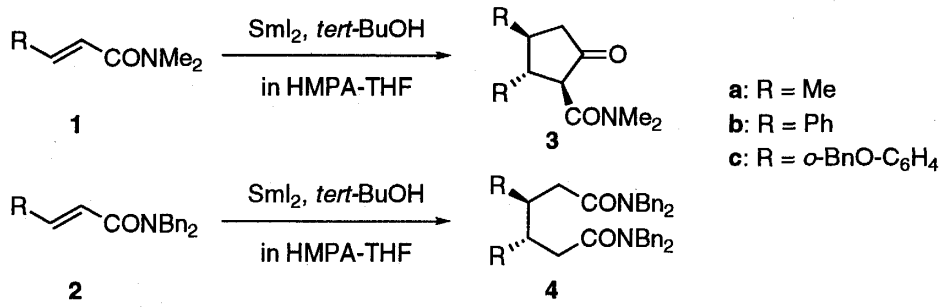
Under the reaction conditions similar to those previously applied by the Inanaga's group,[2] *N,N*-dibenzylcrotonamide (**2a**) was treated with an excess amount (4.3 equiv) of SmI_2 in HMPA-THF in the presence of *t*-butyl alcohol (1 equiv) at room temperature to give the *dl*-isomer of *N,N*-dibenzyl-3,4-dimethyladipamide (**4a**) in 81% yield (Scheme 2 and entry 5 of Table 1). It is now widely accepted that the HMPA enhances the rate of single electron transfer from SmI_2 to unsaturated carbonyl substrates so that, under these conditions, the resulting anion radical intermediates get more chance to undergo coupling reaction rather than either additional electron transfer or hydrogen abstraction leading to conjugate reduction.[3] We applied an experimental procedure in which addition of SmI_2 to the THF/HMPA solution of substrate was complete in a few seconds. Actually, slow addition of SmI_2 led to unsatisfactory results.

When *N,N*-dibenzylcinnamamide (**2b**) as β-aryl-substituted unsaturated amides was employed under equivalent conditions, not only the coupling product **4b** but also the conjugate reduction product, *N,N*-dibenzyl-3-phenylpropionamide, was obtained in 44% yield each (entry 7). Alper and coworkers have reported that the absence of *t*-butyl alcohol, an alcohol additive, favors the formation of conjugate reduction products.[8] For example, reactions of a variety of α,β-unsaturated carbonyl compounds with SmI_2/HMPA in THF led to β-substituted propionic acid derivatives in high yields. To our great surprise, however, the reaction of **2b** in the absence of *t*-butyl alcohol gave the expected coupling product **4b** in 85% yield (entry 6). The anion radical intermediate involved here should be highly stabilized by the phenyl substituent, and as a result, the presence of *t*-butyl alcohol additive disfavors reductive coupling reactions. No clear explanations are in hand so far.

Under the Inanaga's conditions, *N,N*-dimethylcrotonamide (**1a**) produced *t*-2,*c*-3,*N,N*-tetramethyl-5-oxo-*r*-1-cyclopentanecarboxamide (**3a**) in 46% yield (entry 1). This compound **3a** corresponds to a *dl*-selective reductive coupling/Dieckmann condensation product (entry 1). Its stereochemical assignment was based on the vicinal coupling constants, H1/H2 and H2/H3, in the ^1H NMR spectrum (see experimental). Thus, steric bulk of the amide moiety is apparently critical in determining the reaction stage; the amides bearing the sterically bulky *N,N*-dibenzyl amide moiety hinder the intramolecular cyclization step of Dieckmann process. Other *N,N*-dimethylamides **1b,c** underwent similar stereoselective reactions to produce cyclopentanecarboxyamides **3b,c** in the absence of the alcohol additive

(entries 2-4).

In the reaction of crotonamides, *t*-butyl alcohol was employed to avoid the formation of conjugate reduction products[7] and the yields of reductive coupling products were improved. On the contrary, β-aryl substituted α,β-unsaturated amides provided cyclopentanecarboxamides in the absence of *t*-butyl alcohol. As shown in the reaction of dibenzylamides **2**, the single electron transfer from SmI_2 to the amides takes place in a short period and the rapid coupling reaction of the anion radicals is followed. However, in the reactions of *N,N*-dimethylamides **1**, it takes somehow longer reaction times for the second step of Dieckmann condensation reaction.



Scheme 2

Table 1 Reaction of α,β-Unsaturated Amides with SmI_2[a)]

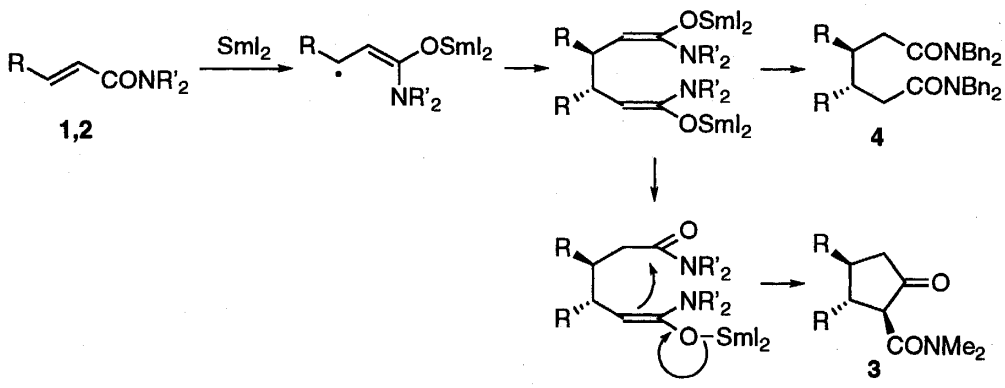
Entry	Amide	SmI_2 equiv.	*tert*-BuOH equiv.	Time min	Product	Yield %
1	**1a**	3.0	1.0	150	**3a**	46
2	**1b**	2.5	-	40	**3b**	39
3	**1c**	2.0	-	30	**3c**	43
4	**1c**	2.0	-	120	**3c**	52
5	**2a**	4.3	1.0	5	**4a**	81
6	**2b**	2.5	-	50	**4b**	85
7	**2b**	2.0	1.0	5	**4b**[b)]	44

a) Reaction conditions: see experimental section.
b) *N,N*-Dibenzyl-3-phenylpropionamide was also obtained in 44% yield.

To the best of our knowledge, the SmI_2-induced sequence of reductive coupling/Dieckmann condensation reaction of α,β-unsaturated carbonyl compounds is unknown. The reaction mechanism remains unclarified. Two examples are known for the reductive coupling/Dieckmann condensation reaction of α,β-unsaturated carbonyl compounds: One is the reductive coupling reaction of α,β-unsaturated esters with ytterbium metal[9] and the other is the electroreductive coupling reaction of α,β-unsaturated esters and amides.[10] The present reactions should require one electron transfer from SmI_2 to α,β-unsaturated amides to form the corresponding anion radical species, which then would undergo homocoupling to give the samarium bisenolates of α,ω-diamides. A part of the bisenolate is quenched and then reacts as electrophile with the internal enolate to produce the cyclopentanecarboxamide.

Among these three types of reductive coupling/Dieckmann condensation reactions, there are differences with respect to the reaction modes and stereoselectivities: 1) Although in both the Yb-induced and electrochemical reactions, α,β-unsaturated esters produce cyclopentanecarboxylates, the SmI_2-induced reactions of α,β-unsaturated esters lead to diesters, 2) diastereoselectivities are poor in the electroreductive coupling of β-substituted

α,β-unsaturated carbonyls, except a cinnamate, while SmI_2-induced α,β-unsaturated amides are highly *dl*-selective, 3) the bulkiness of substituents on the amide nitrogen determines the mode of SmI_2-induced reactions. Since mechanistic study of SmI_2-induced reductive coupling reaction is now in progress, the details will be reported elsewhere.



Scheme 3

Experimental

The IR spectra were taken with a JASCO A-702 spectrometer. The ^1H and ^{13}C NMR spectra were recorded on a JEOL EX-90 (90 MHz for ^1H NMR) and a GSX-270 (270 MHz for ^1H NMR and 67.94 MHz for ^{13}C NMR) instruments. Chemical shifts are expressed in ppm downfield from tetramethylsilane as an internal standard.

***N,N,t*-2,*c*-3-Tetramethyl-5-oxo-*r*-1-cyclopentanecarboxamide (3a).** To a bluish-green solution of samarium (II) iodide (0.15 M in THF, 8 ml, 1.2 mmol) were added HMPA (0.8 ml) and *t*-BuOH (40 μl, 0.4 mmol) at room temperature under nitrogen atmosphere. *N,N*-Dimethylcrotonamide (46 mg, 0.4 mmol) was added all at once to the resulting deep purple solution, and stirred for 2.5 h. After added silica gel and stirred for overnight, insoluble materials were filtered off and washed with diethyl ether. The filtrate and washings were evaporated under reduced pressure, and the residual yellow oil (0.491 g) was chromatographed on silica gel with hexane-diethyl ether (1/2 v/v) as eluent to yield *N,N,t*-2,*c*-3- tetramethyl-5-oxo-*r*-1-cyclopentanecarboxamide (17 mg, 46 %). Pale yellow solid; mp <35 °C; IR (neat) 2896, 1728 (C=O), 1621 (C=O), 1450, 1400, 1291, 1256, 1216, 1150, 1112, 1056, 984, 923, 859, 784, and 696 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.12 and 1.17 (each 3H, each d, J=6.2 Hz, Me-2 and Me-3), 1.79 (1H, m, H-2), 2.01 (1H, dd, J_{gem}=18.3 and J_{4cis-3}=11.7 Hz, H_{cis}-4), 2.43 (1H, m, H-3), 2.51 (1H, ddd, J_{gem}=18.3 Hz, $J_{4trans-3}$=6.6, and $J_{4trans-1}$=0.9 Hz, H_{trans}-4), 3.01 and 3.11 (each 3H, each s, NMe_2), and 3.16 (1H, dd, J_{1-2}=11.5 and $J_{1-4trans}$= 0.9 Hz, H-1); ^{13}C NMR ($CDCl_3$) δ=17.00 and 17.48 (Me-2 and Me-3), 35.88, 36.69, and 37.59 (C-3 and NMe_2), 42.62 (C-2), 47.50 (C-4), 61.55 (C-1), 167.91 (CO-1), and 212.61 (C-5); Mass *m/z* (rel. intensity, %) 184 (10), 183 (M^+, 68), 182 (20), 168 (11), 140 (12), 139 (15), 115 (20), 113 (base peak), 112 (26), 111 (M^+-$CONMe_2$, 96), 110 (70), 98 (48), 97 (42), 96 (10), 95 (13), 87 (12), 83 (13), 71 (14), 68 (25), 67 (14), 55 (33), and 53 (14).

***N,N*-Dimethyl-5-oxo-*t*-2,*c*-3-diphenyl-*r*-1-cyclopentanecarboxamide (3b).** To a solution of *N,N*-dimethylcinnamamide (0.105 g, 0.6 mmol) in dry THF (1.5 ml) were added HMPA (1.5 ml) and samarium (II) iodide (0.15 M in THF, 10 ml, 1.5 mmol) at room temperature under nitrogen atmosphere. After stirred for 40 minutes, silica gel was added to the mixture and stirred for 10 minutes. Insoluble materials were removed off and washed with diethyl ether, and the filtrate and washings were evaporated under reduced pressure to give a yellow oil (1.336 g). The residual oil was chromatographed on silica gel with hexane-

diethyl ether (1/1 v/v) as eluent to yield *N,N*-dimethyl-5-oxo-*t*-2,*c*-3-diphenyl-*r*-1-cyclopentanecarboxamide (36.3 mg, 39%). Colorless needles (from EtOH); mp 183.2-183.7 °C; IR (KBr) 3030, 2909, 1738 (C=O), 1634 (C=O), 1496, 1458, 1400, 1269, 1226, 1144, 1120, 1075, 1037, 917, 757, 700, 645, 616, and 509 cm^{-1}; ^1H NMR ($CDCl_3$) δ=2.74 (1H, dd, J_{gem}=18.3 and J_{4cis-3}=12.5 Hz, H_{cis}-4), 2.93 (1H, ddd, J_{gem}=18.3, $J_{4trans-3}$=7.3, and $J_{4trans-1}$=0.7 Hz, H_{trans}-4), 2.95 and 2.97 (each 3H, each s, NMe_2), 3.57 (1H, ddd, J_{3-4cis}=12.5, J_{3-2}=11.4, and $J_{3-4trans}$=7.3 Hz, H-3), 3.85 (1H, dd, J_{1-2}=11.4 and $J_{1-4trans}$=0.7 Hz, H-1), 4.21 (1H, t, J_{2-1}=J_{2-3}=11.4 Hz, H-2), and 7.13-7.26 (10H, m, Ph); ^{13}C NMR ($CDCl_3$) δ=35.95 and 37.57 (NMe_2), 47.23 and 47.66 (C-2 and C-3), 53.09 (C-4), 61.29 (C-1), 126.99, 127.05, 127.41, 127.54, 128.58, 128.66, 139.94, 140.05 (each Ph), 167.50 (C=O), and 210.60 (C-5); Mass *m/z* (rel. intensity, %) 308 (20), 307 (M^+, 39), 236 (35), 235 (M^+-$CONMe_2$, base peak), 176 (39), 174 (14), 131 (59), 115 (11), 114 (11), 104 (47), 103 (75), 102 (23), 98 (13), 91 (24), 78 (19), 77 (45), 72 (67), and 69 (11); Anal. Found: C, 77.88; H, 6.89; N, 4.82%. Calcd. for $C_{20}H_{21}NO_2$: C, 78.15; H, 6.89; N, 4.56%.

***t*-2,*c*-3-Bis(2-benzyloxyphenyl)-*N,N*-dimethyl-5-oxo-*r*-1-cyclopentanecarboxamide (3c).** To a solution of *N,N*-dimethyl-(2-benzyloxy)cinnamamide (0.422 g, 1.5 mmol) in dry THF (3 ml) were added HMPA (1.5 ml) and samarium (II) iodide (0.1 M in THF, 30 ml, 3 mmol) at room temperature under nitrogen atmosphere. After stirred for 2 h, silica gel was added to the mixture and stirred for 10 minutes. Insoluble materials were removed off and washed with diethyl ether, and the filtrate and washings were evaporated under reduced pressure. The residual oil was chromatographed on silica gel with hexane-ethyl acetate (2/1 v/v) containing triethylamine (0.5 vol.%) as eluent to yield *t*-2,*c*-3-bis(2-benzyloxyphenyl)-*N,N*-dimethyl-5-oxo-*r*-1-cyclopentanecarboxamide (0.204 g, 52%). Colorless solid; IR (neat) 3011, 2832, 1736 (C=O), 1632 (C=O), 1488, 1448, 1387, 1286, 1227, 1136, 1104, 1013, and 744 cm^{-1}; ^1H NMR ($CDCl_3$) δ=2.06 (1H, dd, J_{gem}=18.5 and J_{4-3}=11.5 Hz, one of H-4), 2.78-2.92 (1H, m, the other of H-4), 2.80 and 2.88 (each 3H, each s, NMe_2), 4.35-4.56 (3H, m, H-1, H-2, and H-3), 4.74 (1H, d, J_{gem}=11.7 Hz, OCH_2), 4.87 (2H, s, OCH_2), 4.96 (1H, d, J_{gem}=11.7 Hz, OCH_2), 6.77 (3H, m, Ar), 6.89 (1H, m, Ar), 7.05-7.35 (10H, m, Ar), and 7.46 (1H, m, Ar); ^{13}C NMR ($CDCl_3$) δ=35.78, 37.28, and 37.36 (C-3 and NMe_2), 46.52 (C-2), 50.19 (C-4), 57.80 (C-1), 69.95 and 70.05 (OCH_2), 111.83, 112.09, 120.93, 121.12, 127.09, 127.25, 127.44, 127.74, 127.94, 128.52, 128.62, 130.08, 131.21, 136.94, 137.22, 156.71, and 157.04 (each Ar), 168.46 (CO-1), and 212.28 (C-5); Mass *m/z* (rel. intensity, %) 521 (79), 520 (94), 519 (M^+, base peak), 518 (31), 430 (39), 429 (82), 428 (73), 427 (17), 411 (18), 410 (24), 384 (53), 383 (72), 365 (23), 283 (31), 282 (60), 173 (26), 147 (52), 136 (27), 135 (23), 134 (10), 118 (30), 115 (18), 114 (59), 103 (42), 93 (16), and 89 (61).

***N,N,N',N'*-Tetrabenzyl-3,4-dimethyladipamide (4a).** To a solution of *N,N*-dibenzylcrotonamide (0.265 g, 1 mmol) in dry THF (1 ml) were added HMPA (2.4 ml), *t*-butyl alcohol (94 μl, 1 mmol), and samarium (II) iodide (0.072 M in THF, 60 ml, 4.32 mmol) at room temperature under nitrogen atmosphere. After stirred for 5 minutes, the mixture was quenched with silica gel and stirred for 10 minutes. Insoluble materials were removed off and the filtrate was evaporated under reduced pressure. The residual yellow oil (1.358 g) was chromatographed on silica gel with hexane-ethyl acetate (1/1 v/v) as eluent to yield *N,N,N',N'*-tetrabenzyl-3,4-dimethyladipamide (0.216 g, 81%). Pale yellow oil; IR (neat) 3024, 2947, 1632 (C=O), 1448, 1416, 1354, 1200, 1078, 1029, 950, 906, 728, and 696 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.86 (6H, d, J=5.9 Hz, Me-3 and Me-4), 2.12-2.28 (4H, m, H-2, H-3, H-4, and H-5), 2.32-2.47 (2H, m, H-2 and H-5), 4.43 (4H, s, NCH_2), 4.53 and 4.65 (each 2H, each d, J_{gem}=14.8 Hz, NCH_2), 7.07-7.38 (20H, m, Ph); ^{13}C NMR ($CDCl_3$) δ=15.16 (Me-3 and Me-4), 34.51 (C-3 and C-4), 38.42 (C-2 and C-5), 48.26 and 49.98 (NCH_2), 126.39, 127.34, 127.58, 128.30, 128.59, 128.94, 136.67, and 137.59 (each Ph), and 172.85 (C-1 and C-6); Mass *m/z* (rel.

intensity, %) 533 (27), 532 (M^+, 50), 442 (16), 441 (15), 414 (17), 413 (20), 338 (83), 308 (15), 295 (37), 293 (24), 267 (69), 266 (79), 265 (27), 199 (12), 1989 (72), 195 (22), 182 (34), 181 (base peak), 176 (54), 175 (63), 168 (11), 167 (22), 148 (16), 136 (13), and 69 (66).

***N,N,N',N'*-Tetrabenzyl-3,4-diphenyladipamide (4b).** To a solution of *N,N*-dibenzylcinnamamide (0.196 g, 0.6 mmol) in dry THF (1.5 ml) were added HMPA (1.5 ml) and samarium (II) iodide (0.15 M in THF, 10 ml, 1.5 mmol) at room temperature under nitrogen atmosphere. After stirred for 50 minutes, silica gel was added to the mixture and stirred for 10 minutes. Insoluble materials were removed off and washed with diethyl ether, and the filtrate and washings were evaporated under reduced pressure to give a yellow oil (1.298 g). The residual oil was chromatographed on silica gel with hexane-diethyl ether (1/1 v/v) as eluent to yield *N,N,N',N'*-tetrabenzyl-3,4-diphenyladipamide (0.168 g, 85%). Colorless needles (from hexane-diethyl ether); mp 141.5-142.5 °C; IR (KBr) 3000, 2890, 1622 (C=O), 1491, 1442, 1413, 1350, 1288, 1230, 1195, 1160, 1078, 1029, 1000, 954, 731, 696, 576, and 504 cm^{-1}; ^1H NMR ($CDCl_3$) δ=2.63-2.95 (4H, m, H-2 and H-5), 3.72 (2H, m, H-3 and H-4), 4.15 (2H, d, J_{gem}=17.2, NCH_2), 4.28 (2H, d, J_{gem}=15.0 Hz, NCH_2), 4.34 (2H, d, J_{gem}=17.2 Hz, NCH_2), 4.67 (2H, d, J_{gem}=15.0 Hz, NCH_2), 6.82-7.34 (30H, m, Ph); ^{13}C NMR ($CDCl_3$) δ=37.55 (C-2 and C-5), 47.33, 48.38, and 50.02 (NCH_2, C-3, and C-4), 126.27, 126.43, 127.12, 127.56, 127.90, 128.45, 128.88, 128.91, 136.53, 137.09, and 141.88 (each Ph), and 172.15 (C-1 and C-6); Anal. Found: C, 83.61; H, 6.76; N, 4.34%. Calcd. for $C_{46}H_{44}N_2O_2$: C, 84.11; H, 6.75; N, 4.26%.

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