A Diastereoselective Synthesis of (dl)-1,3-Diphenyl-1,3-propanediamines

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Received 9 October 1991

A large-scale and practical synthesis of (dl)-1,3-diphenyl-1,3-propa
diamine (I) has been achieved by a highly diastereoselective phenylcerium dichloride addition to 1-tert-butoxycarbonyl-4,5-di-
hydro-5-phenylpyrazole (3). Alkyllercium addition reaction to the corresponding 5-alkyl substituted 1-Boc-4,5-dihydropyrazoles was
less satisfactory mainly giving ring-cleaved products. Further elabora-
tion of the diamine I to the N-substituted derivatives R"-R bearing
N-methyl, N-ethyl, N-isopropyl, N-neopentyl, N-benzyl, and N-mesy/methyl groups is described.

The employment of scalemic C2-symmetric 1,2-diamines
1 as auxiliaries and controlled groups in asymmetric synthesis has markedly increased in recent years.2 Accordingly a number of new methods for their syntheses have been developed.3 By contrast, the number of 1,3-diamines employed in such efforts is conspicuously small.3 As part of our program on the development of auxiliary-based, chiral, phosphorus-stabilized anions we were interested in studying the alkylations of scalemic 2-alkyl-1,3,2-di-
azaphosphorinane 2-oxides II and thus required a general method for the synthesis of (dl)-1,3-propanediamines, III.

Our initial target, (dl)-1,3-diphenyl-1,3-propanediamine (I) had previously been prepared and resolved.5 The synthesis of I, however, was less than satisfactory requiring 300 g-atoms of sodium metal for the reduction of 80 g of 1,3-diphenyl-1,3-propanedione bisoxime. Moreover, the diastereoselectivity of the reduction was poor (dl/meso, 12:88). We have therefore devised a more efficient, diastereoselective synthesis based on our recently reported method for the synthesis of amines from hydrazones.6

Starting with cinnamaldehyde, dihydropyrazole 2 was easily prepared by treatment with hydrazine hydrate in ethanol (50–66% yield), Scheme 1.7 After protection as the stable Boc-derivative (89–94% yield),8 the dihydropyrazole 3 was ready for the organometallic addition reaction. Our previously described addition of organocerium reagents to achiral and scalemic hydrazones has employed only N,N-dialkylhydrazine derivatives.5 Though formally a hydrazone, we were unsure if 3 would undergo reaction at the azomethine linkage. The organocerium reagent was prepared by a modification of the Imamoto procedure.9 Thus, one equivalent of freshly

titrated phenyllithium was added to a suspension of anhydrous cerium(III) chloride in THF. Reaction of this species with 3 resulted in the formation of 4 in high yield (85–94%) as a single diastereomer. Even though the organocerium addition reaction is reproducible on small scales (1–3 mmol), the efficiency of the reaction is highly dependent on the dryness of cerium(III) chloride, the hydroxide ion content in the phenyllithium, and the amount of phenyllithium. Because of its strong affinity for the cerium cation, the hydroxide ion can compete with phenyllithium for coordination to Ce(III). Incomplete reagent formation and free phenyllithium led to lower yields (45–75%) of 4 along with the formation of nitrile 5 as a side product.10 Other organometallic reagents such as phenyllithium itself and phenylmagnesium bromide are not suitable for this transformation providing the ring-cleaved nitrile 5 as the major product. 10

The stereochemical course and selectivity of the reaction are most likely due to the Boc group on the nitrogen. We assume that the Boc group is coplanar with the dihydropyrazole ring. Thus, the arrangement of C(5)–N(1)–CO–O–r-Bu resembles an allylic system. In conformation a, see the Figure, the pseudoequatorial phenyl group at C(5) experiences a strong A1,3 interaction with tert-butoxy group which is more clearly seen in Newman projection a'. However, the A1,3-strain can be avoided in conformation b (also shown as Newman projection b') by forcing the phenyl group at C(5) to orient in a pseudoaxial position. Therefore, conformation b should be preferred to conformation a. The facial selectivity of the nucleophilic attack on the azomethine linkage of the pyrazoline ring should be, in part, dependent on the shielding effect of the pseudoaxial C(5)-substituent. Therefore, the nucleophile favors anti attack on the side opposite to the phenyl group to avoid the interactions as shown in the Figure.

Final conversion of 4 to diamine 1 required N–N bond scission of pyrazolidine 4. Although the N–N single bond energy is ca. 37 kcal/mol,12a the N–N bond of 4 could not be cleaved under usual reaction conditions.12 Reduction of the N–N bond was finally accomplished by removal of the Boc group with trifluoroacetic acid followed by high pressure (41 atm of H2) hydrogenation over W-2 Raney nickel. The configuration of diamine 1 was secured by resolution following the literature procedure.5

To extend this protocol for the general synthesis of C2-symmetric 1,3-diamines, a variety of 5-alkyl-substituted (Me, Bu, i-Pr, t-Bu) Boc-dihydropyrazoles was examined. The preparation of the Boc-dihydropyrazoles was accomplished starting from the corresponding α,β-unsaturated aldehydes using the standard procedures described above. The yields were generally higher than for the aryl derivatives. However, the organocerium addition reactions were not as successful, mainly giving the

† Scalemic (from the Greek skalenos, uneven or lopsided as in a scalene triangle) is a term used to describe materials that are enantiomerically enriched.
ring-cleaved nitriles along with small amounts of addition products (less than 20% yield). The basicity of the organocerium reagent may influence the course of the reaction. The more basic nature of the alkylcerium compared to aroylcerium reagents may favor their behavior as bases instead of nucleophiles in this case.

A variety of diamine N-derivatives was prepared from 1 by standard transformations (Scheme 2). Methyl and ethyl derivatives were obtained by LiAlH₄ reduction of the corresponding amides 6a and 6b which were prepared from acetic formic anhydride and acetic anhydride, respectively. The diisopropyl derivative 8c was prepared by in situ catalytic hydrogenation of the acetone Schiff's base in the presence of acetic acid using Adams catalyst. Neopentyl 8d, benzy 8e, and 2,4,6-trimethylbenzyl 8f analogs were formed by in situ reduction of the corresponding bis-imines 7d-7f with sodium borohydride. The experimental data are summarized in the Table. The yields of the diamines are for isolated, purified products which have been fully characterized.

In summary, the highly diastereoselective synthesis of (dl)-1,3-diphenyl-1,3-propanediamine (1) can be accompl-
### Table. Preparation of 1,3-Diamine Derivatives

<table>
<thead>
<tr>
<th>Prod. Method* Yield (%)</th>
<th>mp (°C) (solvent) or bp (°C)/Tor</th>
<th>Molecular Formula</th>
<th>1H NMR (CDCl₃)* δ, J (Hz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6a A 88 228-229 (MeOH)</td>
<td>C₃H₄N₂O₂ (282.34)</td>
<td>8.68 (d, J = 8.2, 2H, NH), 8.08 (s, 2H, HCO), 7.43-7.12 (m, 10H, ArH), 4.95 (q, J = 7.7, 2H, CH(C))</td>
<td>2.02 (t, J = 7.4, 2H, H₂C(2))</td>
</tr>
<tr>
<td>6b A 77 249-250 (MeOH)</td>
<td>C₃H₄N₂O₂ (310.40)</td>
<td>7.36-7.22 (m, 10H, ArH), 5.94 (d, J = 7.5, 2H, NH), 4.90 (q, J = 7.5, 2H, H₂C(1))</td>
<td>2.48 (t, J = 7.5, 2H, H₂C(2)), 1.93 (s, 6H, CH₂C=O)</td>
</tr>
<tr>
<td>8a A 97 150-153/0.4</td>
<td>C₅H₄N₂O₂ (254.37)</td>
<td>7.37-7.21 (m, 10H, ArH), 3.31 (t, J = 6.8, 2H, H₂C(1)), 2.15 (s, 6H, NCH₃)</td>
<td>2.08 (t, J = 6.8, 2H, H₂C(2)), 1.43 (brs, 2H, NH)</td>
</tr>
<tr>
<td>8b A 92 155-156/0.3</td>
<td>C₅H₄N₂O₂ (282.43)</td>
<td>7.36-7.22 (m, 10H, ArH), 3.43 (t, J = 6.8, 2H, H₁(1)), 3.34 (brq, J = 7.1, 4H, 2 × NCH₂CH₃), 2.09 (t, J = 6.8, 2H, H₂C(2)), 1.31 (brs, 2H, NH)</td>
<td>0.96 (t, J = 7.1, 6H, 2 × NCH₂CH₃)</td>
</tr>
<tr>
<td>8c B 91 115/0.05</td>
<td>C₃H₄N₂O₂ (310.48)</td>
<td>7.34-7.20 (m, 10H, ArH), 3.62 (t, J = 6.8, 2H, H₁(1)), 3.50 (t, J = 6.8, 2H, H₂C(2))</td>
<td>1.58 (brs, 2H, NH), 0.90 (d, J = 6.2, 6H, 2 × NCH₂(CH₃)₂)</td>
</tr>
<tr>
<td>8d C 83 192-195/0.4</td>
<td>C₅H₆N₂O₂ (366.59)</td>
<td>7.34-7.20 (m, 10H, ArH), 3.57 (t, J = 6.4, 2H, H₁(1)), 3.07 (Abq, J = 11.4, 4H, 2 × N₄H₂-R-Bu), 1.95 (t, J = 6.4, 2H, H₂C(2))</td>
<td>0.85 (s, 18H, (CH₂)₃)</td>
</tr>
<tr>
<td>8e C 85 68-69 (pentane/Et₂O)</td>
<td>C₅H₆N₂O₂ (406.57)</td>
<td>7.37-7.18 (m, 10H, ArH), 3.70 (t, J = 6.5, 2H, H₁(1)), 3.32 (Abq, J = 12.9, 4H, 2 × NCH₂Ph), 2.08 (t, J = 6.5, 2H, H₂C(2))</td>
<td>1.94 (brs, 2H, NH)</td>
</tr>
<tr>
<td>8f C 87 oil</td>
<td>C₅H₆N₂O₂ (490.73)</td>
<td>7.44-7.31 (m, 10H, ArH), 6.87 (s, 4H, H₁(4)), 3.82 (t, J = 6.6, 2H, H₁(1)), 3.35 (Abq, J = 11.5, 4H, 2 × N₄H₂Ar), 2.31 (s, 6H, 2 × H₂Ar-para), 2.26 (s, 12H, 4 × CH₂Ar-ortho)</td>
<td>2.07 (t, J = 6.6, 2H, H₂C(2)), 1.39 (brs, 2H, NH)</td>
</tr>
</tbody>
</table>

* A: Amide formation followed by LiAlH₄ reduction. B: In situ catalytic hydrogenation with PdO₂C. C: Schiff base formation followed by reduction with NaBH₄.

* Yield of isolated and purified product.

* Satisfactory microanalyses obtained: C, H, N ± 0.3.

* Obtained in DMSO-d₆ for 6a.

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llished by employing the "phenylcerium dichloride" addition reaction to the Boc-protected dihydroropyrazole 3, which is readily available from cinnamaldehyde. An alkyl-substituted variant of this protocol was not successful mainly due to the more basic nature of the alkylcerium reagent affording a ring-cleaved nitriles 5 as the major products. A variety of N-alkyl derivatives of the diamine 1 were prepared through a number of reductive alkylation procedures. Further studies employing these diamines as chiral auxiliaries for phosphorys-stabilized anions will be reported elsewhere.

1H NMR spectra were recorded at 300 MHz with TMS (δ = 0.00 ppm) or CDCl₃ (δ = 7.26) as an internal reference in CDCl₃ solutions unless otherwise specified. ¹³C NMR-spectra were recorded at 75.5 MHz with CDCl₃ (δ = 77.00) as an internal reference in CDCl₃ solutions unless otherwise specified. Assignment of individual ¹³C resonances are supported by APT in most cases. IR spectra were obtained on an IBM-32 FT infrared spectrophotometer in CCl₄ solutions unless otherwise specified with the following relative intensities: s (strong), 67–100%, m (medium), 34–66%, and w (weak), 0–33%. Mass spectra (EI) were obtained with a ionization voltage of 70 eV. Elemental analysis were performed by the University of Illinois Microanalytical Service Laboratory. Bulb-to-bulb distillations were done on a Büchi GKR-50 Kugelrohr apparatus; boiling points (bp) refer to air bath temperatures and are uncorrected. Melting points (mp) were obtained on a Thomas Hoover capillary melting point apparatus. Analytical TLC was performed by using 0.25 mm silica gel plates (Merck) with F-254 indicator. Visualization was accomplished by UV light, iodine, and 5% phosphomolybdic acid solution in EtOH. Flash chromatography was performed using 30-60 μm silica gel (Woelm) with technical grade hexane (distilled from anhydrous CaCl₂), EtOAc (distilled from K₂CO₃), and reagent grade i-PrOH. All solvents used in reactions were distilled from appropriate drying agents before use. THF (sodium benzophenone ketyl), DME (sodium benzophenone ketyl), hexane (CaH₄), CH₂Cl₂ (CaH₂). All reactions were performed in an atmosphere of dry nitrogen. PhLi was titrated by the double titration method and the use of phenylacetic acid. CeCl₃ was dried at 140°C under vacuum (0.01–0.02 Torr) overnight just before use.

1-tert-Butoxy carbonyl-4,5-dihydro-5-phenylpyrazoles (3): 4,5-Dihydro-5-phenylpyrazole (2):
A 250-mL, three-necked round-bottom flask equipped with a pressure-equalizing addition funnel, a reflux condensor, and a magnetic stirring bar was charged with hydrazine hydrate (100 mL). The addition funnel was charged with a solution of cinnamaldehyde (105 g, 0.80 mol) in EtOH (50 mL). The cinnamaldehyde solution was added dropwise to the refluxing hydrazine over 30 min. After complete addition the mixture was further refluxed for 3 h. After allowing the mixture to cool to r.t., the organic layer was separated and the aqueous hydrazine layer was extracted with EtOAc (50 mL). The combined organic layers were dried (Na₂SO₄), filtered, and concentrated in vacuo to give a yellow oil. Fractional distillation of this oil under reduced pressure (110–112°C/0.3 Torr) gave 76.9 g (66%) of dihydropyrazole 2 as a colorless oil which solidified at −20°C.

1-tert-Butoxy carbonyl-4,5-dihydro-5-phenylpyrazole (3):
The dihydropyrazole 2 (30.3 g, 20.4 mol) was dissolved in dry THF (100 mL) in a 250-mL, three-necked, round-bottom flask equipped with a magnetic stirring bar, an addition funnel and a N₂ inlet tube. A solution of di(tert-butyl) dicarbonate (50 g, 22.8 mmol) in THF (50 mL) was slowly added through the addition funnel over 10 min. The mixture was stirred at r.t. overnight (~12 h). After evaporation of the solvent, the residue was purified by preparative LC (silica gel, Waters Prep 500) using hexane/EtOAc (4:1–2:1) to give 44.5 g (89%) of Boc-dihydropyrazole 3 as a pale yellow solid. An analytical sample was obtained by recrystallization from hexane/CHCl₃, mp 63–64°C; Rₚ 0.22 (hexane/EtOAc, 2:1).

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(3R,5R*,S*)-1-tet-Butyloxy carbonyl-3,5-diphenyl pyrazolidine (4): A 1-L, three-necked, round-bottom flask equipped with a glass stopper, a rubber septum, a magnetic stirring bar and a gas-inlet tube was charged with CeCl₃·H₂O (27.0 g, 72.5 mmol). The gas-inlet tube was connected to a trap cooled in liquid N₂. The flask was heated to 140 °C under vacuum (0.1–0.2 Torr) overnight. After cooling to r.t., the stopper was replaced with thermometer and the water trap was removed. Dry THF (250 mL) was added to the flask to give a white suspension which was stirred for 2 h. The suspension was cooled to −78 °C and PhLi (1.6 M in cyclohexane/Et₂O, 70:30, 46 mL, 72.5 mmol) was slowly added over 30 min while maintaining a temperature of −75 to −78 °C to give a lemon-colored solution. After an additional 1 h stirring at −78 °C, the solution of Boc-dicyclopropylzolidine 3 (17.8 g, 72.5 mmol) in THF (50 mL) was added via syringe over 20 min. The mixture was quenched with MeOH after 1 h at −78 °C and allowed to warm to r.t. The mixture was poured into water (300 mL) and extracted with EtOAc (3 × 300 mL). The combined organic layers were washed with brine (300 mL), dried (Na₂SO₄), and filtered through a pad of Celite. The filtrate was concentrated to give a pale yellow oil which was directly purified by preparative LC (silica gel) eluting with hexane/EtOAc (4:1–2:1) to give 22.1 g (94% yield) of pyrazolinedione 4 as a white powder. Yield is variable from 75–94% depending upon dryness of CeCl₃ and hydroxide content of PhLi. An analytical sample was obtained by recrystallization from pentane/Et₂O, mp 112–113°C; Rf 0.42 (hexane/EtOAc, 2:1).

C₂₃H₂₇N₄O₂ calc. C 68.27 H 7.37 N 11.37.
(246.31) found 68.37 7.69 11.38.
MS (70 eV); m/z (%) = 246 (M⁺), 145 (21), 104 (14), 78 (10), 68 (16), 57 (100), 41 (25).
IR: ν = 2980 m, 1732 s, 1659 w, 1597 m, 1495 m, 1478 m, 1456 m, 1401 s, 1368 s, 1248 m, 1134 s, 885 cm⁻¹.
1H NMR (300 MHz): δ = 7.35–7.17 (m, 5H, ArH), 6.89 (br s, 1H, H(C3)), 3.56 (dd, J = 12.1, 5.4 Hz, 1H, H(C5)), 3.42 (dd, J = 18.5, 12.1, 1.2 Hz, 1H, H(C4)), 2.8 (dd, J = 18.5, 5.4, 1.1 Hz, 1H, H(C4)), 1.34 (br s, 9H, (CH₃)₂C).
13C NMR (75.5 MHz): δ = 151.54 (C-O), 143.92 (C3), 142.60 (Ar-iso), 128.43, 127.21, 125.17, 80.80 ((CH₃)₂C), 39.23 (C5), 43.83 (C4), 27.84 ((CH₃)₂C).

(3R)-1,3-Diphenyl-1,3-propanediamine (1): In a 250 mL, three-necked, round-bottom flask equipped with a magnetic stirring bar, a N₂ inlet and a gas outlet tube was placed a solution of pyrazolinedione 4 (21.68 g, 66.8 mmol) in CH₂Cl₂ (45 mL). The mixture was cooled at ice-bath temperature and TFA (45 mL, 0.58 mmol) was slowly added over 30 min. Gas evolved immediately. After 2 h stirring at r.t., excess TFA and CH₂Cl₂ were removed under reduced pressure. The residue was placed into a ceramic-coated cylindrical vessel as a solution in CH₂Cl₂ (250 mL). Raney nickel (W-2) (33 g, damp weight) and HOOAc (10 mL) were added. The vessel was placed into an autoclave and the mixture was hydrogenated under 41 atm pressure of H₂ with vigorous stirring. After 3 days, the mixture was filtered through a pad of Celite with the aid of MeOH. The filtrate was concentrated in vacuo and the residue was dissolved in water (500 mL). The aqueous solution was basified with 20% NaOH solution to pH ~10 and extracted with CHCl₃ (3 × 500 mL). During the extraction, a second filtration might need depending on the reaction scale. The combined extracts were dried (Na₂SO₄, K₂CO₃) and concentrated to give a light orange oil which was purified by Kugelrohr distillation to give 12.37 g (82% yield) of diamine 1 as a colorless oil. The yield was variable from 75–90% depending on the reaction scale, b.p. 145–150°C/0.1 Torr; Rf 0.12 (0.5% NH₄OH in 10% MeOH/CH₂Cl₂).
C₁₃H₁₉N₂ calc. C 79.61 H 8.02 N 12.38.
(226.32) found 79.65 8.03 12.28.
MS (70 eV); m/z (%) = 210 (M⁺ + 1–NH₂, 20), 209 (M⁺ – NH₂, 100), 208 (67), 13 (12), 132 (18), 20 (78), 107 (75), 106 (100), 105 (36), 104 (100), 103 (25), 91 (19), 80 (10), 79 (100), 78 (27), 77 (85), 51 (27), 43 (19), 42 (26), 39 (12), 32 (15), 31 (22).
IR: ν = 3382 br, 3301 br, 3085 m, 3063 s, 3029 s, 2938 m, 1945 w, 1877 w, 1808 w, 1603 s, 1493 s, 1453 s, 1352 m, 1312 m, 1196 m, 1067 m, 1028 w, 911 s, 831 s cm⁻¹.
1H NMR (300 MHz): δ = 7.36–7.24 (m, 10 H, ArH), 3.93 (t, J = 7.0 Hz, 2H, H(C1)), 2.03 (t, J = 7.0 Hz, 2H, H(C2)), 1.6 (br 4H, NH₂).
13C NMR (75.5 MHz): δ = 146.05 (Ar-iso), 127.99, 126.44, 125.79, 52.88 (C1), C(3), 48.11 (C2).

(1R*,3R*)-N,N'-Bisformyl-1,3-diphenyl-1,3-propanediamine (6a): Typical Procedure: To a cold (ice-bath) solution of diamine 1 (510 mg, 2.22 mmol) in CH₂Cl₂ (5 mL) was added aconitic anhydride (94% purity, 527 mg, 5.63 mmol, 2.5 equiv) in CH₂Cl₂ (5 mL) over 5 min. White solid immediately precipitated and the mixture turned to a thick cake. At this moment, the cold bath was removed and the mixture was stirred at r.t. for 30 min. The white solid was filtered and the filter cake was washed with Et₂O (10 mL) and pentane (10 mL) and then dried under vacuum to give 507 mg (80%) of bisformamide 6a as a white powder. The washes were concentrated and dried under vacuum to remove HOOAc and the residue was triturated with Et₂O. G1 filtration gave an additional 52 mg (8%) of bisformamide 6a as a white powder. An analytical sample was obtained by recrystallization from MeOH, mp 228–229°C; Rf 0.96 (0.5% NH₄OH in 10% MeOH/CH₂Cl₂).
MS (70 eV): m/z (%) = 264 (M+ - H2O, 18), 237 (2), 208 (8), 192 (8), 149 (8), 148 (66), 146 (12), 135 (42), 134 (46), 120 (15), 107 (8), 106 (47), 105 (14), 104 (100), 103 (19), 91 (10), 79 (33), 78 (16), 77 (36), 51 (11).
IR (KBr): v = 3251 br, 2934 m, 2868 w, 1690 s, 1649 w, 1584 w, 1543 s, 1495 m, 1449 m, 1435 m, 1385 s, 1354 m, 1256 m, 1213 m, 1105 w, 1057 w, 1026 w, 808 w cm⁻¹.
13C NMR (DMSO-d6, 75.5 MHz): δ = 164.36, 160.57, and 160.43 (major rotamer) (C=O), 143.55, 143.20 (major), and 143.00 (14-isoform), 128.46, 128.34, 126.28 (major), 126.19, 128.14, 127.04, 126.79 (major), 126.16 (major), 126.08, 54.85, 52.69, 48.27 (major), and 47.95 (C1 (C3)), 43.26 and 42.94 (major) (C2 (C4)).

(1R*,3R*)-N,N-Bis-[2,2-dimethylpropyl]-1,3-diphenyl-1,3-propanediol (8a); Typical Procedure: To a solution of diamine 1 (950 mg, 4.20 mmol) in dry MeOH (10 mL) was added pivaldehyde (1.14 mL, 10.5 mmol, 2.5 equiv). The mixture was heated to reflux for 1 h. The oil bath was removed and the mixture was cooled to rt. Toluene (10 mL) was added to the mixture followed by addition of NaBH4 in small portions over 20 min. After the complete addition of NaBH4 (770 mg, 20.8 mmol, 5 mol equiv), the mixture was stirred for 3 h and water (20 mL) was slowly added. The mixture was extracted with EtOAc (3 x 30 mL) washed with brine (20 mL), dried (Na2SO4), and concentrated to give a colorless oil which was purified by kugelrohr distillation to give 1.8 g (83%) of diamine 8d as a colorless oil, bp 192–195°C/0.4 Torr. Rf 0.50 (hexane/ EtOAc, 9:1).

MS (70 eV): m/z (%) = 366 (M+), 293 (25), 280 (20), 279 (18), 278 (17), 277 (16), 276 (15), 275 (14), 274 (13), 273 (12), 272 (11), 271 (10), 270 (9), 269 (8), 268 (6), 267 (5), 266 (4), 265 (3), 264 (2), 263 (1). C27H32O3 (430.55) calc’d: 37.77; found: 37.76.

IR: v = 3320 br, 2963 w, 2743 m, 2603 m, 2543 m, 2473 m, 2273 m, 1701 s, 1611 s, 1571 w, 1411 s, 1371 w, 1201 s, 1161 s, 1061 s, 1001 s, 941 s, 801 s, 701 s, 601 s cm⁻¹.
13C NMR (75.5 MHz): δ = 143.15 (Ar-ipso), 128.11, 127.06, 126.80, 62.20 (C1 (C3)), 45.19 (C2 (C4)), 39.35 (NCH3).
13C NMR (75.5 MHz): $\delta = 144.48$ (Ar-ipsa), 136.88 (Ar-ortho), 136.17 (Ar-ipsa), 133.82 (Ar-para), 128.77, 128.29, 127.10, 126.93, 61.00 (C1, C3), 46.62 (C2), 45.69 (NCH$_2$Ar), 20.82 (CH$_3$Ar-para), 19.29 (CH$_3$Ar-ortho).