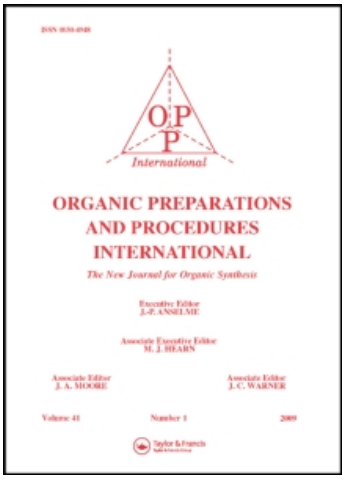

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CONVENIENT PREPARATION OF 14-HYDRO-5*H*-TRIBENZO[*a,d,f*]CYCLOOCTENES

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CONVENIENT PREPARATION OF 14-HYDRO-5*H*-TRIBENZO[*a,d,f*]CYCLOOCTENES

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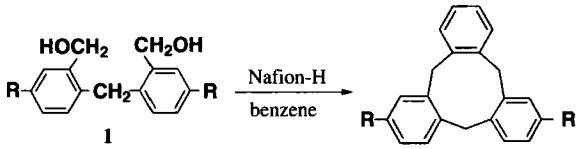
The chemistry of medium and large sized bridged aromatics with one or two aromatic rings has been extensively studied.[1] Recently, many examples of compounds containing three or more aromatic rings have been reported[2] and these are mostly oligomers formed as by-products during the synthesis of lower members.[3] Sato and co-workers have reported[4] the concentrated sulfuric acid catalyzed cyclialkylation of benzene under high dilution conditions to construct a large-membered ring compound, 10,15-dihydro-5*H*-tribenzo[*a,d,g*]cyclononene. We have recently found[5] a convenient and efficient method of cyclization using Lewis acid or Nafion-H (a solid perfluorinated resinsulfonic acid)[6] catalyzed Friedel-Crafts cyclibenzylation of 2,2'-*bis*(hydroxymethyl)diphenylmethanes (**1**) with arenes to afford 10,15-dihydro-5*H*-tribenzo[*a,d,g*]cyclononenes (**2**) under relatively mild reaction conditions, in which an arene molecule is directly incorporated into the cyclic system as a phenylene

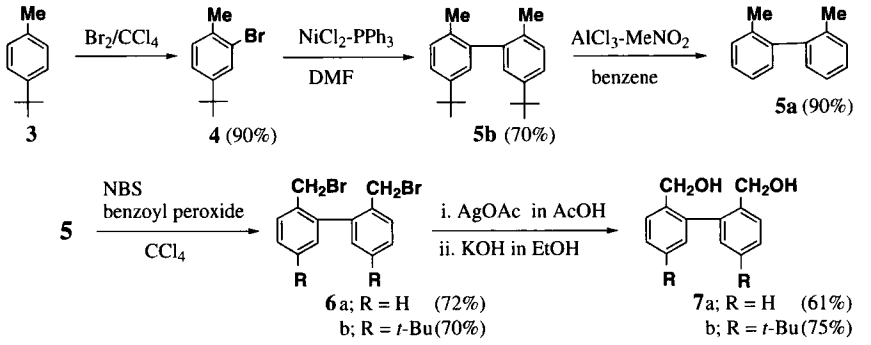


1
a; R = H
b; R = *t*-Bu

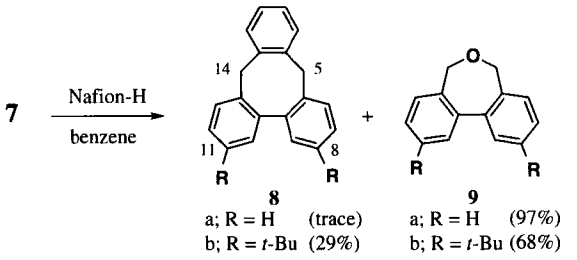
2a; R = H
b; R = *t*-Bu

unit. Cyclibenzylation of 2,2'-*bis*(hydroxymethyl)biphenyls (**7**) with arenes in the presence of Lewis acids or protic acids has not been reported. We report the first successful Lewis acid or Nafion-H catalyzed cyclibenzylation of **7** and the bromomethyl analogues **6** with arenes to give 14-hydro-5*H*-tribenzo[*a,d,f*]cyclooctenes.

The preparative route for 2,2'-*bis*(bromomethyl)biphenyl (**6a**) and 2,2'-*bis*(bromomethyl)-5,5'-di-*tert*-butylbiphenyl (**6b**) using the *tert*-butyl group as a positional protective group[7] is shown in the following Scheme. An attempted Nafion-H-catalyzed cyclibenzylation of 2,2'-*bis*(hydroxymethyl)biphenyl (**7a**) with benzene under reflux for 2 h failed. Only trace amount of the desired 14-hydro-5*H*-tribenzo[*a,d,f*]cyclooctene (**8a**) was obtained. Instead the intramolecular dehydration product **9a** was obtained in 97% yield. This result indicates that the intramolecular dehydration can



occur much faster than the intermolecular cyclibenzylation in spite of using a large excess of benzene. A higher yield was obtained for **8b** along with **9b** (68%).



The attempted cyclibenzylation reaction of 2,2'-*bis*(bromomethyl)biphenyl (**6a**) with benzene performed in the presence of $TiCl_4$ and $AlCl_3$ $MeNO_2$ at room temperature for 2 h gave the desired cyclibenzylation product 14-hydro-5*H*-tribenzo[*a,d,f*]cyclooctene (**8a**) in 90 and 94% yield, respectively. No *bis*benzylated product was detected under the reaction conditions used. This result indicates that the intramolecular cyclibenzylation can occur much faster than the intermolecular benzylation in spite of using a large excess of benzene. The same results were obtained in the case of

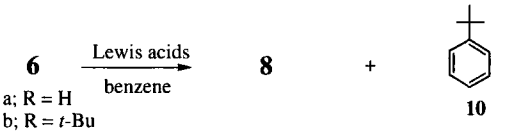


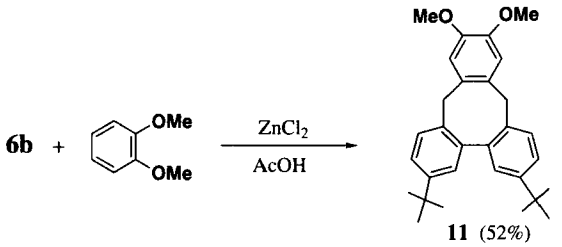
TABLE 1. Lewis Acid-catalyzed Reaction of 2,2'-*bis*-(Bromomethyl)biphenyls (**6**) in Benzene[a]

Run	Substrate	Catalyst	Product Yield (%)[b]
1	**6a**	A	**8a** (90)
2	**6a**	B	**8a** (94)
3	**6b**	A	**8b** (85)
4	**6b**	B	**8a** (75)
5	**8b**	B	**8a** (80)

a) [Benzene]:[Substrate]=30:1; [Catalyst]:[Substrate]= 2.4:1; A: $TiCl_4$; B: $AlCl_3$-$MeNO_2$. b) Isolated yields are shown.

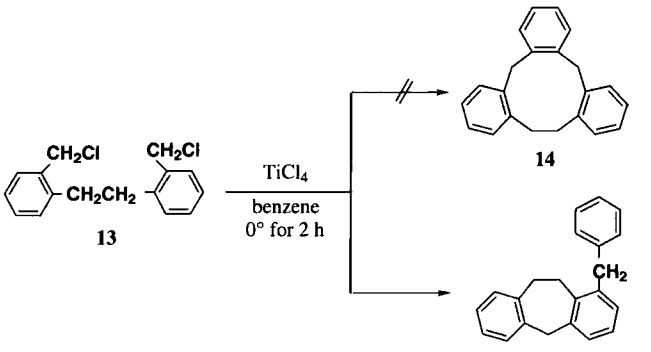
the reaction of 2,2'-*bis*(bromomethyl)-5,5'-di-*tert*-butylbiphenyl (**6b**) in benzene in the presence of $TiCl_4$. Interestingly, using $AlCl_3$-$MeNO_2$[7] as catalyst in benzene afforded de-*tert*-butylated product **8a** in 75% yield along with *tert*-butylbenzene (**10**). Trans-*tert*-butylation of **8b** under the same conditions afforded **8a** in 80% yield. The structures of **8a** and **8b** are elucidated based on their elemental analyses and spectral data. The mass spectral data for **8a** and **8b** (M^+ = 256 and 368) strongly support the cyclibenzylated structures, respectively.

It was also found that when 2,2'-*bis*(bromomethyl)-5,5'-di-*tert*-butylbiphenyl (**6b**) was treated with 1,2-dimethoxybenzene in acetic acid in the presence of $ZnCl_2$ at 100-105° for 6 h, the desired cyclibenzylation product, **11** was obtained in 52% yield. However, with *p*- or *m*-xylene, only complex mixtures were obtained.



11 (52%)

In contrast, similar treatment of 2,2'-*bis*(chloromethyl)diphenylethane (**13**) with benzene in the presence of $TiCl_4$ gave the intramolecular cyclization to 1-benzyl-10,11-dihydro-5*H*-dibenzo[*a,d*]cycloheptene (**15**) in 95% yield *via* the 1-chloromethyldibenzo[*a,d*]cycloheptene intermediate. No formation of the desired [2.1.1]orthocyclophane (**14**) was observed. Thus, this result is quite different from the results of the cyclibenzylation of the corresponding biphenyl and diphenylmethane analogues with benzene. This finding might be explained by the more favorable formation of the seven-membered 1,4-cycloheptadiene over the corresponding larger ten-membered of **14**.



15 (95%)

Thus, the cyclibenzylation reaction of 2,2'-*bis*(bromomethyl)biphenyls (**6**) with arenes to give cyclibenzylated products **8** appears to be a useful method for the preparation of 14-hydro-5*H*-tribenzo[*a,d,f*]cyclooctenes. Studies of the scope and limitations of the method are in progress.

EXPERIMENTAL SECTION

All melting points are uncorrected. ^{1}NMR spectra were recorded on a Nippon Denshi JEOL FT-270 NMR spectrometer in $CDCl_3$ with TMS as an internal reference. IR spectra were measured as KBr pellets or as liquid films on NaCl plates in a Nippon Denshi JIR-AQ2OM spectrometer. Mass spectra were obtained on a Nippon Denshi JMS-01SA-2 spectrometer at 75 eV using a direct inlet system. GLC analyses were carried out using Shimadzu gas chromatograph (conditions: GLC-14A; Silicone OV-1, 2 m; programmed temperature rise, 12°/min; carrier gas nitrogen, 25 mL/min).

Materials.- 2-Bromo-4-*tert*-butyltoluene (**4**) was prepared from 4-*tert*-butyltoluene (**3**) according to the reported procedure.[8] The preparation of 2,2'-dimethylbiphenyl (**5a**) using $AlCl_3$-$MeNO_2$-catalyzed *trans-tert*-butylation of 5,5'-di-(*tert*-butyl)-2,2'-dimethylbiphenyl (**5b**) has been previously described.[9] 2,2'-*bis*(Hydroxymethyl)diphenylethane (**12**) was prepared according to the reported procedure.[10] Nafion-H catalyst was prepared from commercially available (Du Pont) Nafion-K resin, as previous described.[6]

Coupling Reaction of 2-Bromo-4-*tert*-butyltoluene (4) in the Presence of $NiCl_2$-PPh_3 to Give 5b.- A mixture of $NiCl_2$ (570 mg, 4.40 mmol), PPh_3 (8.80 g, 33.6 mmol), and zinc powder (8.8 g, 0.135 mol) in DMF (66 mL) was heated at 50° for 1 h until the brown solution became blue violet. 2-Bromo-4-*tert*-butyltoluene (**4**) (20.0 g, 88.0 mmol) was added and the mixture was heated at 80° for 16 h. The reaction mixture was cooled to room temperature, poured into ice-water (200 mL), extracted with CH_2Cl_2, washed with water, dried (Na_2SO_4), filtered, and concentrated *in vacuo*. The residue was distilled to give *5,5'-di-tert-butyl-2,2'-dimethylbiphenyl* (**5b**) as a colorless liquid (9.04 g, 70%); b.p.132-134°/3 mm Hg (lit.[9] 132-134°/3 mm Hg).

Preparation of 2,2'-*bis*(Bromomethyl)-5,5'-di-*tert*-butylbiphenyl (6b). – A mixture of **5b** (4 g, 13.6 mmol), N-bromosuccinimide (6.65 g, 37.4 mmol), and benzoyl peroxide (100 mg, 0.376 mmol) in carbon tetrachloride (300 mL) was heated at reflux for 12 h. The reaction mixture was cooled to room temperature, washed with aqueous sodium hydroxide and water, dried (Na_2SO_4), filtered, and evaporated *in vacuo*. The residue was recrystallized from hexane to afford **6b** as colorless prisms (4.3 g, 70 %), mp. 120–122°; NMR ($CDCl_3$): δ 1.35 (18 H, s), 4.16 (2 H, d, *J* 10.0), 4.36 (2 H, d, *J* 10.0), 7.34 (2 H, d, *J* 1.8), 7.43 (2 H, dd, *J* 1.8/8.4), 7.48 (2 H, d, *J* 8.4); mass spectrum: *m/e* 450, 452, 454 (M^+).

Anal. Calcd. for $C_{22}H_{28}Br_2$: C, 58.43; H, 6.24. Found: C, 58.60; H, 6.51

Compound **6a** was prepared in a similar manner in 72% yield.

2,2'-bis(Bromomethyl)biphenyl (**6a**) was obtained as colorless prisms (hexane/benzene, 1:1), mp. 87–89°; NMR ($CDCl_3$): δ 4.19 (2 H, d, *J* 10.0), 4.35 (2 H, d, *J* 10.0), 7.25–7.57 (8 H, m); mass spectrum: *m/e* 338, 340, 342 (M^+).

Anal. Calcd. for $C_{14}H_{12}Br_2$: C, 45.95; H, 3.56. Found: C, 45.66; H, 3.51

Preparation of 5,5'-Di-(*tert*-butyl)-2,2'-*bis*(hydroxymethyl)biphenyl (7b). – A mixture of **6b** (511 mg, 1.13 mmol) and silver acetate (1 g, 6.0 mmol) in acetic acid (20 mL) was heated at 100° for 24 h and the reaction mixture was cooled to room temperature. The reaction mixture was poured into large amount of ice-water, and extracted with CH_2Cl_2. The CH_2Cl_2 solution was washed with aqueous

Na_2CO_3 and water, dried (Na_2SO_4), filtered, and evaporated *in vacuo*. The residue was disolved in EtOH (30 mL). To this solution was added a solution of potassium hydroxide (335 mg, 5.97 mmol) in water (2 mL). The mixture was warmed at 50° for 15 min. The reaction mixture was concentrated under reduced pressure, and extracted with CH_2Cl_2. The CH_2Cl_2 solution was washed with water, dried (Na_2SO_4), filtered, and evaporated in *vacuo*. The residue was recrystallized from hexane to afford the *title compound* **7b** as colorless prisms (277 mg, 75%), mp. 126–129°; IR (KBr): 3275 (OH), 3073, 3028, 2962, 2865, 1605, 1489, 1458, 1438, 1390, 1360, 1257, 1236, 1094, 1050, 1040, 1021, 893, 825, 679, 669, 565 cm^{-1}; NMR ($CDCl_3$): δ 1.32 (18 H, s), 2.91 (2 H, broad s, OH), 4.28 (2 H, d, *J* 11.5), 4.32 (2 H, d, *J* 11.5), 7.19–7.41 (6 H, m); mass spectrum: *m/e* 308 (M^+-H_2O).

Anal. Calcd. for $C_{22}H_{30}O_2$: C, 80.94; H, 9.26. Found: C, 81.06; H, 9.09

Compound **7a** was prepared in a similar manner in 61% yield.

2,2'-*bis(Hydroxymethyl)biphenyl* (**7a**) was obtained as colorless prisms (hexane/benzene, 1:1), mp. 100°; IR (KBr): 3348 (OH), 3265, 1478, 1445, 1424, 1341, 1252, 1194, 1033, 774, 755 cm^{-1}; NMR ($CDCl_3$): δ 2.36 (2 H, broad s), 4.33 (2 H, d, *J* 11.7), 4.38 (2 H, d, *J* 11.7), 7.14–7.51 (8 H, m); mass spectrum: *m/e* 196 (M^+-H_2O).

Anal. Calcd. for $C_{14}H_{14}O_2$: C, 78.48; H, 6.59. Found: C, 78.65; H, 6.52

Reaction of 5,5'-Di-(*tert*-butyl)-2,2'-*bis*(hydroxymethyl)biphenyl (7b) with Benzene in the Presence of Nafion-H.- A mixture of **7b** (300 mg, 0.919 mmol) and Nafion-H (300mg) in benzene (30 mL) was heated at reflux for 2 h. The solid resinsulfonic acid was then filtered off and the filtrate was analyzed by GLC (OV-1, 2 m). The filtrate was concentrated under the reduced pressure to a residue, which was column chromatographed on silica gel with hexane and a mixture of hexane and benzene (1:1 v/v) as eluent to give **8b** (98 mg, 29%) and **9b** (193 mg , 68%), respectively.

Recrystallization of crude **8b** from methanol afforded *8,11-di-(tert-butyl)-14-hydro-5H-tribenzo[a,d,f]cyclooctene* (**8b**) as colorless prisms; mp. 118–120°; IR (KBr): 2963, 2950, 2905, 2867, 1495, 1435, 1362, 1262, 838, 818, 745, 690 cm^{-1}; NMR ($CDCl_3$): δ 1.36 (18 H, s), 3.66 (4 H, s), 7.10–7.42 (10 H, m); mass spectrum: *m/e* 368 (M^+).

Anal. Calcd. for $C_{28}H_{32}$: C, 91.25; H, 8.75. Found: C, 90.93; H, 8.95

Recrystallization of crude **9b** from methanol afforded *2'',3'-di-(tert-butyl)-2,7-dihydro-3,4,5,6-dibenzoxepin* (**9b**) as colorless prisms; mp. 141–144°; IR (KBr): 3027, 2962, 2904, 2859, 1463, 1363, 1210, 1076, 903, 813 cm^{-1}; NMR ($CDCl_3$): δ 1.41 (18 H, s), 4.34 (4 H, s), 7.35 (2 H, d, *J* 7.8), 7.44 (2 H, dd, *J* 2.1/7.8), 7.56 (2 H, d, *J* 2.1); mass spectrum: *m/e* 308 (M^+).

Anal. Calcd. for $C_{22}H_{28}O$: C, 85.66; H, 9.15. Found: C, 85.40; H, 9.07

Cyclibenzylation of 2,2'-*bis*(Bromomethyl)biphenyls (6) with Benzene in the Presence of Lewis Acid. Typical Procedure.- To a solution of **6b** (452 mg, 1 mmol) in benzene (20 mL) was added $TiCl_4$ (0.26 mL, 2.4 mmol) at room temperature. After stirring the reaction mixture at room temperature for 2 h, it was poured into ice-water (30 mL) and extracted with benzene (2 x 30 mL). The combined extracts were washed with water (2 x 30 mL), dried (Na_2SO_4), filtered, and the solvent was evaporated *in vacuo*. The residue was chromatographed on silica gel using hexane as eluent to give

crude **8b** as a colorless solid. Recrystallization from methanol afforded *8,11-di-tert-butyl-14-hydro-5H-tribenzo[a,d,f]cyclooctene* (**8b**) (332 mg, 90%) as colorless prisms.

Compound **8a** was prepared, similarly. The reaction conditions and yields are compiled in Table 1.

14-Hydro-5H-tribenzo[a,d,f]cyclooctene (**8a**) was obtained as colorless prisms (MeOH); mp. 138–140°; IR (KBr): 3058, 3015, 1492, 1478, 1445, 1437, 1429, 776, 758, 747, 735, 629 cm^{-1}; NMR ($CDCl_3$): δ (27°) 3.68 (4 H, s), 7.15–7.40 (12 H, m); ($CDCl_3/CS_2$, 1:3) (-80°) 3.54 (2 H, d, *J* 13.5), 3.65 (2 H, d, *J* 13.5), 7.15–7.40 (12 H, m); mass spectrum: *m/e* 256 (M^+).

Anal. Calcd. for $C_{20}H_{16}$: C, 93.71; H, 6.29. Found: C, 93.50; H, 6.19

$AlCl_3$-$MeNO_2$ Catalyzed *trans-tert*-Butylation of 8,11-Di-*tert*-butyl-14-hydro-5*H*-tribenzo[*a,d,f*]cyclooctene (8b) in Benzene.- To a solution of **8b** (369 mg, 1 mmol) in benzene (20 mL) was added a solution of $AlCl_3$ (320 mg, 2.4 mmol) in $MeNO_2$ (0.6 mL) at room temperature. After stirring of the reaction mixture at room temperature for 2 h, it was poured into ice/ water (30 mL) and extracted with benzene (2 x 30 mL). The combined extracts were washed with water (2 x 30 mL), dried (Na_2SO_4), filtered, and the solvent was evaporated *in vacuo*. The residue was chromatographed on silica gel using hexane as eluent to give crude **8a** as a colorless solid. Recrystallization from methanol afforded **8a** (205 mg, 80%) as colorless prisms. The formation of *tert*-butylbenzene (**10**) was confirmed by GLC.

Cyclibenzylation of 6b with 1,2-Dimethoxybenzene in the Presence of $ZnCl_2$.- To a solution of **6b** (452 mg, 1 mmol) and 1,2-dimethoxybenzene (166 mg, 1.2 mmol) in acetic acid (10 mL) was added $ZnCl_2$ (100 mg, 0.73 mmol) at room temperature. After stirring of the reaction mixture at 100-105° for 6 h, it was poured into ice-water (30 mL) and extracted with benzene (2 x 30 mL). The combined extracts were washed with water (2 x 30 mL), dried (Na_2SO_4), filtered, and the solvent was evaporated *in vacuo*. The residue was chromatographed on silica gel using benzene as eluent to give crude **11** as a colorless solid. Recrystallization from methanol afforded *8,11-di-tert-butyl-2,3-dimethoxy-14-hydro-5H-tribenzo[a,d,f]cyclooctene* (**11**) (223 mg, 52%) as colorless prisms, mp. 127–129°; NMR ($CDCl_3$): δ 1.36 (18 H, s), 3.60 (4 H, broad s), 3.87 (6 H, s), 6.82 (2 H, s), 7.25–7.42 (6 H, m); mass spectrum: *m/e* 428 (M^+).

Anal. Calcd. for $C_{30}H_{36}O_2$: C, 84.07; H, 8.47. Found: C, 84.35; H, 8.65.

Preparation of 2,2'-*bis*(Chloromethyl)diphenylethane (**13**).- To a solution of 2,2'-*bis*(hydroxylmethyl)diphenylethane **12**[10] (310 mg, 1.27 mmol) and a few drops of pyridine in benzene (50 mL) was added gradually a solution of thionyl chloride (2 mL) in benzene (5 mL) at room temperature. After the reaction mixture had been heated at reflux for 4 h, it was cooled to room temperature, washed with water, dried (Na_2SO_4), filtered, and evaporated *in vacuo*. The residue was recrystallized from hexane to afford **13** (324 mg, 91%) as colorless prisms, mp. 103–104°; IR (KBr): 3026, 1601, 1492, 1453, 1429, 1259, 841, 791, 768, 706, 659 cm^{-1}; NMR ($CDCl_3$): δ 3.08 (4 H, s), 4.58 (4 H, s), 7.20–7.36 (8 H, m); mass spectrum: *m/e* 278, 280, 282 (M^+).

Anal. Calcd. for $C_{16}H_{16}Cl_2$: C, 68.83; H, 5.78. Found: C, 68.77; H, 5.85

Reaction of 2,2'-*bis*(Chloromethyl)diphenylethane (13) with Benzene in the Presence of $TiCl_4$. – To a solution of **13** (200 mg, 0.511 mmol) in benzene (30 mL) was gradually added $TiCl_4$ (0.18 mL, 1.64 mmol) at 0°. The reaction mixture was allowed to stand at room temperature for 1 h before adding cold water (20 mL). The organic layer was washed with water, dried (Na_2SO_4), and concentrated under reduced pressure. The residue was taken up in CH_2Cl_2 and chromatographed over silica gel with a mixture of hexane and benzene (1:1) as eluent to give *1-benzyl-10,11-dihydro-5H-dibenzo[a,d]cycloheptene* (**15**) (193 mg, 95%) as a colorless oil; NMR ($CDCl_3$): δ 2.90–3.12 (4 H, m), 4.00 (2 H, s), 4.11 (2 H, s), 6.95–7.30 (12 H, m); mass spectrum: *m/e* 284 (M^+).

Anal. Calcd. for $C_{22}H_{20}$: C, 92.70; H, 7.10. Found: C, 92.91; H, 7.09

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