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Sodium Diarylbismuthide as a Reagent for the Bismuthanation of Reactive Arenes. Application to the Synthesis of Mixed Triarylbismuthanes Bearing a Substituent Group Incompatible with Grignard and Organolithium Reagents

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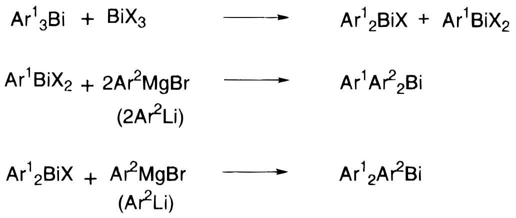
Summary: Several mixed triarylbismuthanes bearing an electron-withdrawing functionality, such as the nitro, carbonyl, or ester group, which are inaccessible by the conventional methods based on organometallic reagents such as Grignard and organolithium compounds, are synthesized in low to acceptable yields by the reaction of in situ generated sodium diarylbismuthide with iodoarenes or arenediazonium tetrafluoroborates.

Introduction

In connection with the ongoing program in our laboratory, we needed some unsymmetrical triarylbismuthanes bearing an electron-withdrawing functionality, such as the nitro, carbonyl, or ester group. The most general method for the synthesis of unsymmetrical triarylbismuthanes is the reaction of an aryl Grignard or an aryllithium reagent with an arylbismuth dihalide or a diarylbismuth halide.[1] These arylbismuth halides are in turn obtained by the ligand exchange reaction between a symmetrical triarylbismuthane and a bismuth(III) halide, choice of the major product depending on the ratio of the respective reagents (Scheme 1).[1,2] In any case, however, these organometallic approaches are not applicable for our present purpose because of their incompatibility with the unsaturated polar functions, such as the nitro, carbonyl, and ester group.

An alternative possible choice may be the bismuthanation of haloarenes with sodium diarylbismuthide, which has long been known and can be generated by the reaction between diarylbismuth halides and sodium metal in liquid ammonia.[3] To our knowledge, however, this type of reaction has not been previously examined as a means for the preparation of unsymmetrical triarylbismuthanes. In order to circumvent the use of sodium metal and liquid ammonia, we sought to find an alternative method for the generation of the diarylbismuthide anion and found that the combination of the diarylbismuth trifluoromethanesulfonate−hexamethylphosphoric triamide (HMPA) complex[4] and sodium naphthalenide produced, smoothly, the corresponding bismuthide anion as a deep green tetrahydrofuran (THF) solution at low temperature. Herein, we report a new and reliable method for the preparation of the title triarylbismuthanes.

Scheme 1



$$Ar^1{}_3Bi + BiX_3 \longrightarrow Ar^1{}_2BiX + Ar^1BiX_2$$

$$Ar^1BiX_2 + \underset{(2Ar^2Li)}{2Ar^2MgBr} \longrightarrow Ar^1Ar^2{}_2Bi$$

$$Ar^1{}_2BiX + \underset{(Ar^2Li)}{Ar^2MgBr} \longrightarrow Ar^1{}_2Ar^2Bi$$

Results and Discussion

Bis(4-methylphenyl)bismuth trifluoromethanesulfonate−HMPA complex (**2**), prepared from tris(4-methylphenyl)bismuthane (**1**) according to the reported procedure,[4] was treated with sodium naphthalenide in THF at −78 °C to generate sodium bis(4-methylphenyl)bismuthide (**3**), which was subsequently reacted with 1-iodonaphthalene (**4a**) or 9-iodophenanthrene (**4b**) to give the corresponding mixed triarylbismuthanes (**6a** and **b**) in 46% and 52% yields, respectively (Scheme 2). However, hindered 9-iodoanthracene (**4c**) failed to react in the expected manner. Mixed triarylbismuthanes **6a,b** were also obtainable by the reaction of a diarylbismuth halide and an aryllithium. Thus, when treated with 1-naphthyllithium (**9a**) and 9-phenanthryllithium (**9b**), bis(4-methylphenyl)bismuth bromide (**8**) gave the corresponding tertiary bismuthanes **6a** and **6b** in 82% and 52% yields, respectively. By this procedure, 9-anthrylbis(4-methylphenyl)bismuthane (**6c**) was also accessible in 51% yield.

When the above methodology based on sodium diarylbismuthide was extended to the reaction with halonitroarenes, to our disappointment a complicated mixture of products resulted and no tertiary bismuthanes containing the nitrophenyl moiety could be isolated in our hands.

Next, we turned out attention to the arenediazonium salt, a more reactive aryl donor as compared with iodoarene. Barton and co-workers have previously reported that the copper-induced decomposition of 4-nitrobenzenediazonium tetrafluoroborate (**5d**) in dimeth-

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(1) For a review, see: Freedman, L. D.; Doak, G. O. *Chem. Rev.* **1982**, *82*, 15. Also refer to: Wiber, M. *Gmelin Handbook of Inorganic Chemistry*; Springer-Verlag: Berlin, 1978.

(2) For example, see: Challenger, F.; Allpress, C. F. *J. Chem. Soc.* **1915**, *107*, 16.

(3) (a) Gilman, H.; Yablunky, H. L. *J. Am. Chem. Soc.* **1941**, *63*, 212. (b) Kaufmann, F.; Steinseifer, F.; Klas, N. *Chem. Ber.* **1985**, *118*, 1039. For the bismutanation using sodium dimethylbismuthide, see: (b) Wieber, M.; Saucer, I. *Z. Naturforsch., B* **1985**, *40*, 1476. (C) Wieber, M.; Rudolph, K. *Z. Naturforsch., B* **1988**, *3*, 739.

(4) Matano, Y.; Miyamatsu, T.; Suzuki, H. *Organometallics* **1996**, *15*, 1951.

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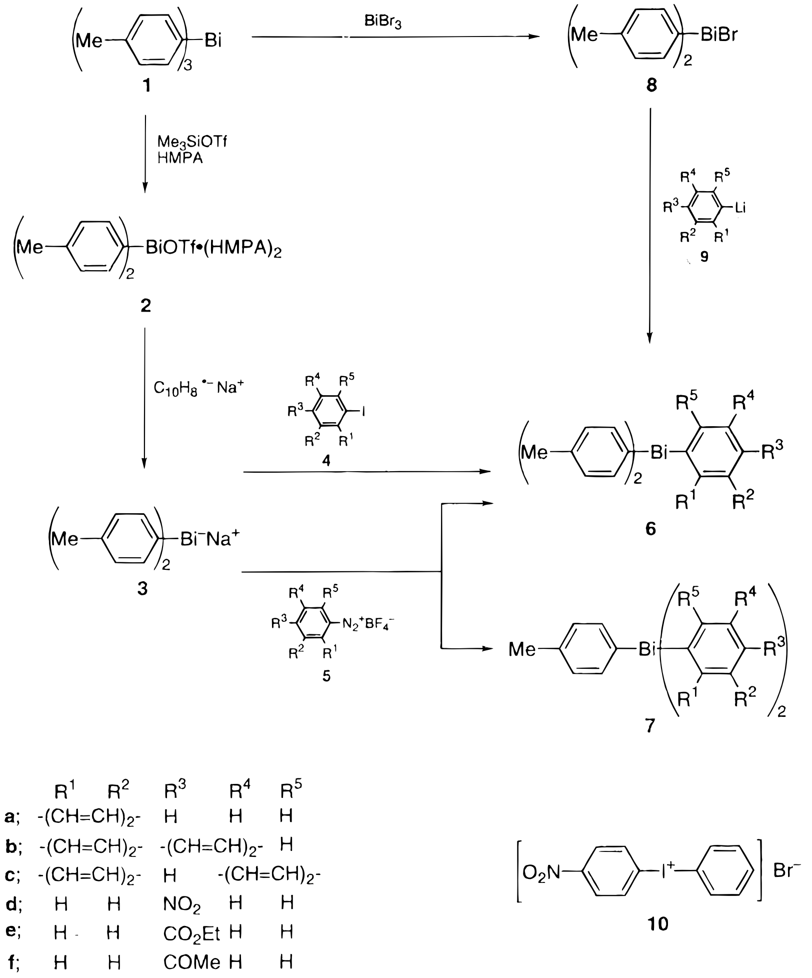
Scheme 2



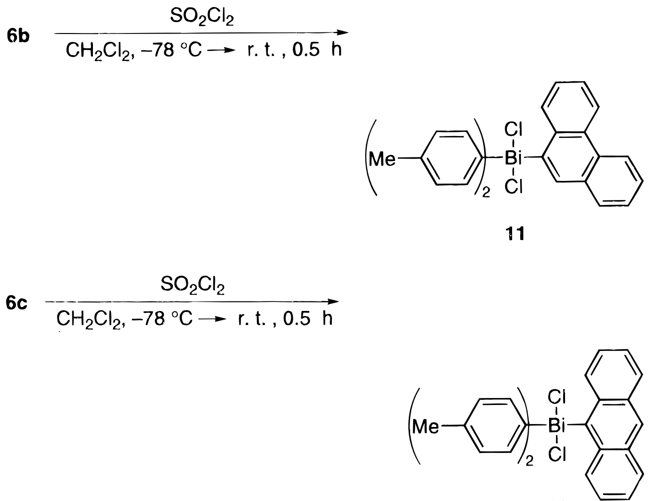
	R^1	R^2	R^3	R^4	R^5
a;	-(CH=CH)$_2$-		H	H	H
b;	-(CH=CH)$_2$-		-(CH=CH)$_2$-		H
c;	-(CH=CH)$_2$-		H	-(CH=CH)$_2$-	
d;	H	H	NO$_2$	H	H
e;	H	H	CO$_2$Et	H	H
f;	H	H	COMe	H	H

ylformamide (DMF) in the presence of bismuthane **1** gave bis(4-methylphenyl)(4-nitrophenyl)bismuthane (**6d**) and (4-methylphenyl)bis(4-nitrophenyl)bismuthane (**7d**) in 12% and 15% yields, respectively.[5] However, the thermal decomposition of arenediazonium salts occurs in a homolytic way to give a complicated mixture of products, from which each component can usually be obtained with difficulty. Hence, we examined the reaction of sodium diarylbismuthide **3** with diazonium salt **5d** in THF at −78 °C and obtained the expected products **6d** and **7d** in 15% and 6% isolated yields, respectively (Scheme 2). In this reaction, the composition of the product mixture was simple and compounds **6d** and **7d** were easily isolated by column chromatography on silica gel. Other products were those arising from simple decomposition of the starting materials.

Similar reaction of **3** with diazonium salts **5e** and **5f** in THF at −78 °C afforded bismuthanes **6e** and **6f**, both in about 10% yield. Attempts to improve the yield of bismuthanes **6** by using the iodonium salt **10**[6] in place of diazonium salt **5** met with failure. The reaction of mixed triarylbismuthanes **6b** and **6c** with sulfuryl chloride in dichloromethane at low temperature afforded the corresponding dichlorides **11** and **12** in 90% and 88% yields, respectively (Scheme 3).

In summary, we have developed a new method for the synthesis of mixed triarylbismuthanes having a nitro, carbonyl, or ester group on an aromatic ring, which are inaccessible by the known methodologies based on common organometallic reagents such as Grignard and organolithium compounds. Although the yields are unsatisfactory, this method seems to provide the only reliable direct route available at the present time to this type of mixed triarylbismuthane.

Scheme 3



Experimental Section

General Comments. All reactions were carried out under argon. Melting points were measured on a Yanagimoto hot-

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[5] Barton, D. H. R.; Bhatnagar, N. Y.; Finet, J.-P.; Motherwell, W. B. *Tetrahedron* **1986**, *42*, 3111.

[6] Kazmierczak, P.; Skulski, L. *Synthesis* **1995**, 1027.

stage apparatus and are uncorrected. Tetrahydrofuran (THF) and diethyl ether were distilled from benzophenone ketyl under argon before use. Bismuth(III) bromide and sulfuryl chloride were used as commercially received. *n*-Butyllithium was titrated against diphenylacetic acid.[7] The aromatic iodo compounds (**4a**−**c**)[8] and diazonium compounds (**5d,f**)[9] were prepared according to the reported procedures. Thin layer chromatography (TLC) was performed by using Merck precoated silica gel sheets, 60F-254. Silica gel (Wakogel) of size 200 mesh was used for column chromatography. ^{1}H NMR (200 MHz) spectra were recorded on a Varian Gemini-200 spectrometer for CDCl$_3$ solutions, with tetramethylsilane (TMS) as an internal standard. IR spectra were recorded on a Shimadzu FTIR-8100S spectrophotometer, and only prominent peaks below 2000 cm^{-1} are recorded. Electron impact mass spectra (EIMS) were obtained at 70 eV on a Shimadzu GCMS-QP2000A spectrometer, and fast atom bombardment mass spectra (FABMS) were obtained on a JEOL JMS-HS110 spectrometer using 3-nitrobenzyl alcohol as a matrix. Elemental analyses were performed at Microanalytical Laboratory of Kyoto University.

Reaction of Sodium Bis(4-methylphenyl)bismuthide 3 with Iodoarenes 4a,b. General Procedure. Bis(4-methylphenyl)bismuth trifluoromethanesulfonate−HMPA complex **2** was prepared by stirring tris(4-methylphenyl)bismuthane (**1**; 0.48 g, 1.0 mmol), (trimethylsily)trifluomethanesulfonate (0.19 mL, 1.0 mmol), methanol (0.5 mL), and HMPA (0.35 mL, 2.0 mmol) in dichloromethane (5 mL) for 2 h at room temperature. Removal of the volatiles under reduced pressure left complex **2** as a colorless solid residue, which was dissolved in THF and cooled to −78 °C. Then a solution of sodium naphthalenide, prepared from sodium metal (0.07 g, 3 mmol) and naphthalene (0.41 g, 3.2 mmol) in THF (8 mL) at 0 °C, was added dropwise and stirred for 5 min to generate bismuthide **3**. To the resulting deep green solution, iodo compound **4** was added in one portion with vigorous stirring at −78 °C, and the mixture was allowed to gradually warm to room temperature over 45 min. After the reaction mixture was stirred for an additional 1 h at room temperature, saturated aqueous ammonium chloride was slowly added to the reaction mixture. The organic phase was separated and filtered through a Celite bed. The aqueous phase was extracted with benzene (5 mL × 3), and the extract was washed with brine (15 mL × 2). The combined organic phase was dried over anhydrous magnesium sulfate and evaporated *in vacuo* to leave an oily residue, which was chromatographed on silica gel or recrystallized from methanol to give **6** as pale yellow crystals.

Bis(4-methylphenyl)(1-naphthyl)bismuthane (6a). Yield: 46%. Mp: 133−134 °C (lit.[7] 129−130 °C). ^{1}H NMR: δ 2.31 (s, 6H), 7.17 (d, $J = 8$ Hz, 4H), 7.60 (d, $J = 8$ Hz, 4H), 7.3−7.5 (m, 3H), 7.8−8.0 (m, 4H). EIMS (m/z): 426 (M$^+$ − tolyl − H), 391 (M$^+$ − naphthyl), 336 (M$^+$ − 2 tolyl), 300 (M$^+$ − tolyl − naphthyl), 209. IR (KBr): 1485, 1383, 1184, 1009, 790, 769 cm^{-1}. Anal. Calcd for $C_{24}H_{21}Bi$: C, 55.60; H, 4.08. Found: C, 55.44; H, 4.04.

Bis(4-methylphenyl)(9-phenanthryl)bismuthane (6b). Yield: 52%. Mp: 165−170 °C. ^{1}H NMR: δ 2.32 (s, 6H), 7.18 (d, $J = 8$ Hz, 4H), 7.5−7.6 (m, 5H), 7.65 (d, $J = 8$ Hz, 4H), 8.0−8.1 (m, 1H), 8.28 (s, 1 H), 8.7−8.8 (m, 2H). FABMS (m/z): 569 (M$^+$ + 1), 477 (M$^+$ − tolyl), 300 (M$^+$ − tolyl − phenanthryl), 391 (M$^+$ − phenanthryl), 386 (M$^+$ − 2 tolyl), 286, 209. IR (KBr): 1485, 1442, 1051, 1008, 794, 744, 715 cm^{-1}. Anal. Calcd for $C_{28}H_{23}Bi$: C, 59.16; H, 4.08. Found: C, 59.11; H, 4.06.

Reaction of Bis(4-methylphenyl)bismuth Bromide (8) with Aryllithiums 9a−c. Typical Procedure. 9-Anthryllithium (**9c**) was generated by stirring *n*-butyllithium (1.43 M, 0.77 mL, 1.1 mmol) and 9-bromoanthracene (0.26 g, 1.0 mmol) in diethyl ether (10 mL) for 2 h at room temperature. To the solution of **9c** thus obtained was added with vigorous stirring at −78 °C a suspension of bis(4-methylphenyl)bismuth bromide (**8**), generated from tris(4-methylphenyl)bismuthane (0.32 g, 0.67 mmol) and bismuth(III) bromide (0.15 g, 0.33 mmol) in diethyl ether (10 mL), and the resulting mixture was allowed to gradually warm to room temperature. After the reaction mixture was stirred at room temperature for 1 h, the mixture was worked up as usual and the product was recrystallized from methanol to afford bis(4-methylphenyl)(9-anthryl)bismuthane (**6c**) as crystals (0.29 g, 51%). Mp: 148−151 °C. ^{1}H NMR: δ 2.31 (s, 6H), 7.10 (d, $J = 8$ Hz, 4H), 7.1−7.3 (m, 4H), 7.70 (d, $J = 8$ Hz, 4H), 8.0−8.1 (m, 2H), 8.3−8.4 (m, 2H), 8.50 (s, 1H). FABMS (m/z): 569 (M$^+$ + 1), 476 (M$^+$ − tolyl − H), 300 (M$^+$ − tolyl − anthryl), 391 (M$^+$ − anthryl), 386 (M$^+$ − 2 tolyl), 268, 209. IR (KBr): 1486, 1445, 1385, 1310, 1262, 1010, 885, 790, 727 cm^{-1}. Anal. Calcd for $C_{28}H_{23}Bi$: C, 59.16; H, 4.08. Found: C, 58.71; H, 4.07.

Bis(4-methylphenyl)(1-naphthyl)bismuthane (**6a**) and bis(4-methylphenyl)(9-phenanthryl)bismuthane (**6b**) were prepared similarly from the corresponding iodoarenes **4a** and **4b** in 80% and 51% yields, respectively.

Reaction of Sodium Bis(4-methylphenyl)bismuthide (3) with Arenediazonium Tetrafluoroborates 5d,f. Typical Procedure. To a solution of diarylbismuth triflate−HMPA complex **2** (0.90 g, 1.0 mmol) in THF (5 mL) was added dropwise at −78 °C a solution of sodium naphthalenide, prepared from sodium metal (0.07 g, 3.0 mmol) and naphthalene (0.41 g, 3.2 mmol) in THF (8 mL), and the resulting mixture was stirred for 5 min. 4-Nitrophenyldiazonium tetrafluoroborate (**5d**; 0.24 g, 1.0 mmol) was added portionwise to the stirred bismuthide solution at −78 °C and then allowed to warm to room temperature over 45 min. After the reaction mixture was stirred for an additional 1 h at room temperature, the mixture was worked up as usual to afford an oily residue, which was passed through a silica gel column using hexane−ethyl acetate (10:1) as the solvent to elute compounds **6d** and **7d** in this order in 15% and 5% yields, respectively.

Bis(4-methylphenyl)(4-nitrophenyl)bismuthane (6d). Mp: 92−94 °C (lit.[5,11] 129−132 °C). ^{1}H NMR: δ 2.32 (s, 6H), 7.24 (d, $J = 8.0$ Hz, 4H), 7.60 (d, $J = 8.0$ Hz, 4H), 7.90 (d, $J = 8.0$ Hz, 2H), 8.12 (d, $J = 8.0$ Hz, 2H). FABMS (m/z): 422 (M$^+$ − tolyl), 391 (M$^+$ − $C_6H_4NO_2$), 300 (M$^+$ − tolyl − $C_6H_4NO_2$), 209. Anal. Calcd for $C_{20}H_{18}BiNO_2$: C, 46.79; H, 3.53; N, 2.73. Found: C, 46.19; H, 3.40; N, 2.72.

(4-Methylphenyl)bis(4-nitrophenyl)bismuthane (7d). Mp: 150−153 °C (lit.[5] 145−149 °C). ^{1}H NMR: δ 2.36 (s, 3H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.60 (d, $J = 8.0$ Hz, 2H), 7.90 (d, $J = 8.0$ Hz, 4H), 8.19 (d, $J = 8.0$ Hz, 4H). FABMS (m/z): 453 (M$^+$ − tolyl), 422 (M$^+$ − $C_6H_4NO_2$), 331 (M$^+$ − tolyl − $C_6H_4NO_2$), 300 (M$^+$ − 2 $C_6H_4NO_2$), 209. Anal. Calcd for $C_{19}H_{15}BiN_2O_4$: C, 41.92; H, 2.78; N, 5.15. Found: C, 41.83; H, 2.81; N, 5.14.

Mixed tertiary bismuthanes **6e** and **6f** were prepared similarly from the corresponding arenediazonium salts **5e** and **5f** in 10% and 11% yields, respectively.

Bis(4-methylphenyl)(4-carbethoxyphenyl)bismuthane (6e). Pale yellow oil. ^{1}H NMR: δ 2.32 (s, 6 H), 1.37 (t, $J = 6.9$ Hz, 3H), 4.35 (q, $J = 6.9$ Hz, 2H), 7.20 (d, $J = 7.6$ Hz, 4 H), 7.61 (d, $J = 7.6$ Hz, 4H), 7.81 (d, $J = 8.2$ Hz, 2H), 8.01 (d, $J = 8.2$ Hz, 2H). FABMS (m/z): 541 (M$^+$ + 1), 449 (M$^+$ −

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(7) Kofron, W. G.; Baclawski, L. *J. Org. Chem.* **1976**, *41*, 1879.

(8) Suzuki, H.; Kondo, A.; Inouye, M.; Ogawa, T. *Synthesis* **1986**, 121.

(9) Flood, D. T. *Organic Syntheses*; Wiley: New York, 1943, Collect. Vol. II, p 295.

(10) Gilman, H.; Yablunky, H. L. *J. Am. Chem. Soc.* **1941**, *63*, 207.

(11) Reference 5 reported that ^{1}H NMR (CDCl$_3$) absorptions of the aromatic protons of compounds **6d** and **7d** appeared as multiplets at δ 8.14−7.39 and 8.17−7.39, respectively. However, these compounds were found to show four sets of well-separated doublets in the aromatic proton region. Contrary to the original report, a small difference in δ values of the methyl protons of these compounds was observed; the reported value is δ 2.35 for both compounds.

tolyl), 391 ($M^+ - C_6H_4CO_2Et$), 300 (M^+ − tolyl − $C_6H_4CO_2Et$), 209, 149. IR (neat): 1715 (C=O), 1584, 1387, 1281, 1103, 795, 752 cm^{-1}. Anal. Calcd for $C_{23}H_{23}BiO_2$: C, 51.12; H, 4.29. Found: C, 51.82; H, 4.33.

(4-Acetylphenyl)bis(4-methylphenyl)bismuthane (6f). Colorless crystals. Mp: 101−104 °C. ^{1}H NMR: δ 2.32 (s, 6H), 2.56 (s, 3H), 7.21 (d, J = 7.6 Hz, 4H), 7.61 (d, J = 7.6 Hz, 4H), 7.83 (d, J = 6.0 Hz, 2 H), 7.92 (d, J = 6.0 Hz, 2H). FABMS (m/z): 511 ($M^+ + 1$), 419 (M^+ − tolyl), 391 ($M^+ - C_6H_4COMe$), 300 (M^+ − tolyl − C_6H_4COMe), 209. IR (KBr): 1684 (C=O), 1576, 1485, 1385, 1269, 1184, 1007, 953, 797 cm^{-1}. Anal. Calcd for $C_{22}H_{21}BiO$: C, 51.77; H, 4.15. Found: C, 51.70; H, 4.15.

Reaction of Arylbis(4-methylphenyl)bismuthanes 6b,c with Sulfuryl Chloride. Typical Procedure. To a dichloromethane (10 mL) solution of bismuthane **6b** (0.30 g, 0.44 mmol) was added sulfuryl chloride (0.47 μL, 0.48 mmol) at −78 °C. As the mixture was warmed gradually to room temperature, the color of the reaction mixture turned yellow. After 1 h, the solvent was removed under reduced pressure to leave dichloride **11** as a yellow solid, which was purified by recrystallization from a mixture of dichloromethane and hexane.

Bis(4-methylphenyl)(1-phenanthryl)bismuth Dichloride (11). Yield: 90%. Mp: 145−147 °C. ^{1}H NMR: δ 2.49 (s, 6H), 7.57 (d, J = 8.2 Hz, 4H), 7.6−7.8 (m, 5H), 8.0−8.1 (m, 1H), 8.25 (s, 1H), 8.65 (d, J = 8.2 Hz, 4H), 8.7−8.8 (m, 2H). FABMS (m/z): 603 (M^+ − Cl), 391 (M^+ − 2Cl − phenanthryl), 443, 268, 209. Anal. Calcd for $C_{28}H_{23}BiCl_2$: C, 52.60; H, 3.63. Found: C, 52.23; H, 3.53.

(9-Anthryl)bis(4-methylphenyl)bismuth Dichloride (12). Yield: 88%. Mp: 170−173 °C. ^{1}H NMR: δ 2.49 (s, 6H), 7.4−7.5 (m, 4H), 7.54 (d, J = 8.5 Hz, 4H), 8.0−8.1 (2H, m), 8.3−8.4 (m, 2H), 8.62 (s, 1H), 8.65 (d, J = 8.5 Hz, 4H). FABMS (m/z): 603 (M^+ − Cl), 476 (M^+ − tolyl − H), 391 (M^+ − anthryl), 268, 212, 209. IR (KBr): 1522, 1473, 1446, 1385, 1266, 1248, 1183, 997, 899, 795, 727 cm^{-1}. Anal. Calcd for $C_{28}H_{23}BiCl_2$: C, 52.60; H, 3.63. Found: C, 52.43; H, 3.50.

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Additions and Corrections

1996, Volume 15

Tetsuo Ohta, Yoichi Tonomura, Kyoko Nozaki, Hidemasa Takaya, and Kazushi Mashima*: An Anionic Dinuclear BINAP−Ruthenium(II) Complex: Crystal Structure of $[NH_2Et_2][\{RuCl((R)\text{-}p\text{-MeO-BINAP})\}_2(\mu\text{-}Cl)_3]$ and Its Use in Asymmetric Hydrogenation.

Pages 1522−1523. An improper computer operation caused wrong calculations of bond distances and angles of complex (*R*)-**2**. Selected bond lengths (Å) and angles (deg) in the caption to Figure 1 are corrected as follows: Ru−Cl(1) = 2.437(3), Ru−Cl(2) = 2.423(3), Ru−Cl(3) = 2.493(3), Ru−Cl(3*) = 2.518(3), Ru−P(1) = 2.271(3), Ru−P(2) = 2.269(3); Cl(1)−Ru−Cl(2) = 163.9(1), Cl(1)−Ru−Cl(3) = 79.8(1), Cl(1)−Ru−Cl(3*) = 79.3(1), Cl(2)−Ru−Cl(3) = 87.6(1), Cl(2)−Ru−Cl(3*) = 88.7(1), Cl(3)−Ru−Cl(3*) = 80.5(1), Cl(1)−Ru−P(1) = 96.0(1), Cl(1)−Ru−P(2) = 103.5(1), Cl(2)−Ru−P(1) = 94.8(1), Cl(2)−Ru−P(2) = 88.0(1), Cl(3)−Ru−P(1) = 94.0(1), Cl(3)−Ru−P(2) = 173.0(1), Cl(3*)−Ru−P(1) = 173.3(1), Cl(3*)−Ru−P(2) = 94.0(1), P(1)−Ru−P(2) = 91.8(1). The dihedral angle between the two naphthyl planes of **2** was 70.7°.

Supporting Information Available: Corrected tables for the crystallographic study of compound (*R*)-**2** (21 pages). Ordering information is given on any current masthead page.

OM970421Y