JOC*Note*

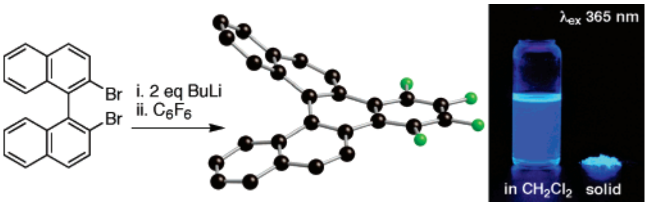
7:8,9:10-Dibenzo-1,2,3,4-tetrafluorotriphenylene: Synthesis, Structure, and Photophysical Properties of a Novel [5]Helicene

Darryl J. Morrison,[†] Tyler K. Trefz,[†] Warren E. Piers,[*,†]
Robert McDonald,[‡] and Masood Parvez[†]

Department of Chemistry, University of Calgary, 2500
University Drive N.W., Calgary, Alberta, Canada T2N 1N4

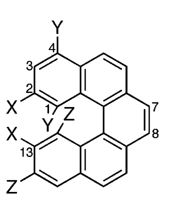
wpiers@ucalgary.ca

Received March 30, 2005

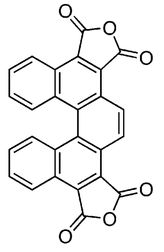


A simple and efficient synthesis and crystallographic analysis of the partially fluorinated 7:8,9:10-dibenzo-1,2,3,4-tetrafluorotriphenylene, **1**, in 59% yield is reported. Compound **1** is a blue luminescent [5]helicene in both solution and the solid state. It spontaneously forms enantiomorphous single crystals from racemic samples and exhibits slipped, face-to-face F⋯C_π contacts within the stacks of molecules.

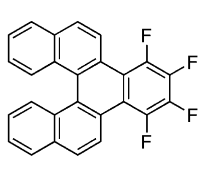
The helicenes[1] have attracted general interest due to their repeating structural motif of ortho-fused aromatic rings that result in a unique combination of features: helical chirality, inherently strong chromophores, and the possibility for steric/electronic interactions between overlapping rings. Helicene derivatives have thus found applications as ligands in asymmetric catalysis,[2,3] as enantioselective fluorescent sensors,[4] as components in chiral discotic liquid crystalline materials,[5] and in organic materials exhibiting second-order nonlinear optical properties.[6] Helicenes derived from all benzene rings have traditionally been prepared by the oxidative photocyclization of stilbene derivatives, although yields for these key steps are generally poor (e.g., 8−34% for some [5]helicene derivatives).[1,7] Renewed interest in helical molecules has led to the development of higher yielding synthetic routes, including Diels−Alder cyclizations between 1,4-distyryl benzene derivatives and quinone dienophiles,[8] cyclotrimerizations of benzynes,[9] Sn-mediated tandem radical cyclization of (*Z*,*Z*)-1,4-bis(2-iodostyryl)-benzene derivatives,[10] and Co(I)-catalyzed isomerization of aromatic (*Z*,*Z*)-dienetriynes.[11]



X = Y = Z = H **I**
X = PPh_2, Y = Z = H **II**
X = CN; Y = Z = H **III**
Y = CN; Z = Me; X = H **IV**



V



1

Despite the importance of helicene derivatives in many areas, X-ray crystallographic studies of helicenes are relatively rare. Structurally characterized examples of [5]helicene **I**, the smallest member of the helicene family, and its derivatives are particularly uncommon. Kuroda reported the unequivocal crystal structures of two racemic crystal forms of the parent [5]helicene **I** in 1982.[12] To our knowledge, the only other crystallographic studies of [5]helicene derivatives are the diphosphine [5]Heliphos **II**,[2] three mixed cyano- and methyl-substituted [5]helicenes (of which **III** and **IV** are examples),[7] and the dianhydride **V**,[13] all of which crystallized in racemic crystal forms. We now report a simple and efficient synthesis and crystallographic study of 7:8,9:10-dibenzo-1,2,3,4-tetrafluorotriphenylene **1**, a blue luminescent [5]helicene that spontaneously forms enantiomorphous single crystals from racemic samples.

Treatment of racemic 2,2′-dibromo-1,1′-binaphthyl (DBBN)[14] with 1 equiv of *n*-BuLi in THF and quenching into excess C_6F_6 gave 2-bromo-2′-pentafluorophenyl-1,1′-binaphthyl **2** in 76% yield (Scheme 1). The structure of **2** was verified by ^1H, ^{19}F, and ^{13}C NMR spectroscopy and X-ray diffraction studies.[15] Subsequent treatment of **2** with *n*-BuLi followed by workup and chromatography

---

[*] To whom correspondence should be addressed. Tel: 403-220-5746. Fax: 403-289-948.

[†] Department of Chemistry, University of Calgary, 2500 University Drive, N.W., Calgary, Alberta, Canada T2N 1N4.

[‡] X-ray Structure Laboratory, Department of Chemistry, University of Alberta, Edmonton, Alberta, Canada T6G 2G2.

(1) Martin, R. H. *Angew. Chem., Int. Ed. Engl.* **1974**, *13*, 649−660.

(2) (a) Reetz, M. T.; Beuttenmüller, E. W.; Goddard, R. *Tetrahedron Lett.* **1997**, *38*, 3211. (b) Terfort, A.; Görls, H.; Brunner, H. *Synthesis* **1997**, 79.

(3) Dreher, S. D.; Katz, T. J.; Lam, K.-C.; Rheingold, A. L. *J. Org. Chem.* **2000**, *65*, 815.

(4) Reetz, M. T.; Sostmann, S. *Tetrahedron* **2001**, *57*, 2515.

(5) (a) Verbiest, T.; Sioncke, S.; Persoons, A.; Vyklicky, L.; Katz, T. J. *Angew. Chem., Int. Ed.* **2002**, *41*, 3882. (b) Nuckolls, C.; Katz, T. J.; Katz, G.; Collings, P. J.; Castellanos, L. *J. Am. Chem. Soc.* **1999**, *121*, 79.

(6) Clays, K.; Wostyn, K.; Persoons, A.; Maiorana, S.; Papagni, A.; Daul, C. D.; Weber, V. *Chem. Phys. Lett.* **2003**, *372*, 438.

(7) Stammel, C.; Fröhlich, R.; Wolff, C.; Wenck, H.; de Meijere, A.; Mattay, J. *Eur. J. Org. Chem.* **1999**, 1709.

(8) Carreño, M. C.; García-Cerrada, S.; Urbano, A. *J. Am. Chem. Soc.* **2001**, *123*, 7929.

(9) Peña, D.; Cobas, A.; Pérez, D.; Guitián, E.; Castedo, L. *Org. Lett.* **2003**, *5*, 1863.

(10) (a) Harrowven, D. C.; Nunn, M. I. T.; Fenwick, D. R. *Tetrahedron Lett.* **2002**, *43*, 7345. (b) Harrowven, D. C.; Nunn, M. I. T.; Fenwick, D. R. *Tetrahedron Lett.* **2002**, *43*, 3189.

(11) Teply, F.; Stará, I. G.; Stary, I.; Kollárovic, A.; Saman, D.; Rulísek, L.; Fiedler, P. *J. Am. Chem. Soc.* **2002**, *124*, 9175.

(12) Kuroda, R. *J. Chem. Soc., Perkin Trans. 2* **1982**, 789.

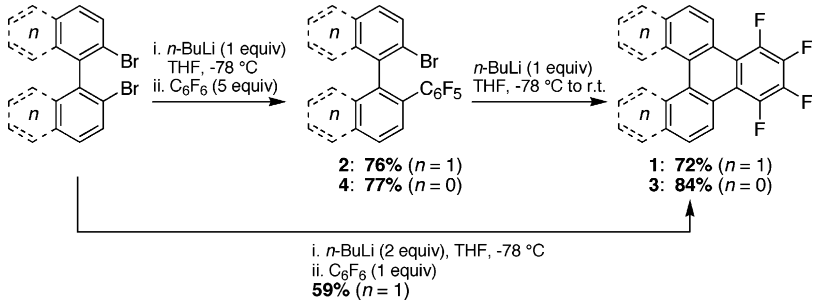
(13) Frimer, A. A.; Kinder, J. D.; Youngs, W. J.; Meador, M. B. *J. Org. Chem.* **1995**, *60*, 1658.

(14) (a) Brown, K. J.; Berry, M. S.; Waterman, K. C.; Lingenfelter, D.; Murdoch, J. R. *J. Am. Chem. Soc.* **1984**, *106*, 4717. (b) Brown, K. J.; Berry, M. S.; Murdoch, J. R. *J. Org. Chem.* **1985**, *50*, 4345.

(15) See the Supporting Information for X-ray crystallographic details.

10.1021/jo0506231 CCC: $30.25

SCHEME 1. One- and Two-Step Syntheses of 1 and Tetrafluorotriphenylene 3



gave **1** as pale yellow, microcrystalline needles in 72% (55% overall from DBBN). In a one-step procedure, DBBN was treated with 2 equiv of n-BuLi at −78 °C and quenched with 1 equiv of C_6F_6 to give **1** in 59% yield. Tetrafluorotriphenylene **3**[16] was also prepared from 2,2′-dibromobiphenyl (DBBP) via the two-step procedure of monolithiation and quenching with C_6F_6 to form **4** in 77% yield, then further treatment with n-BuLi to give **3** as fairly insoluble, fine needles in 84% yield (65% overall from DBBP). Watson and co-workers recently reported a similar one-step synthesis of **3** in a comparable 68% yield.[17]

We also attempted the synthesis of optically enriched [5]helicene **1** via the two-step protocol in Scheme 1. Thus, (R)-(+)-**2** prepared from (R)-(+)-DBBN (>98% ee) was treated with n-BuLi in THF to give (+)-**1** with very low specific rotation at all wavelengths tested ($[\alpha]_\lambda \leq 5$). It is known that although [5]helicene (−)-**I** can be isolated in optically enriched form ($[\alpha]_{436} = -4950$, $[\alpha]_{578} = -1670$)[18] by its separation from the 1:1 complex of (+)-**I**:(+)-TAPA at 0 °C, the barrier for its racemization is low ($\Delta\Delta G$ = 24.1 kcal·mol^{-1} at 293 K)[19] and proceeds at ambient temperatures ($t_{1/2} \sim 14$ h at 298 K and 62.7 min at 330 K).[20] We therefore assume that **1** prepared from optically pure starting materials is nearly racemic based on the low specific rotation observed.

Single needle crystals (0.71 × 0.07 × 0.05 mm) of **1** suitable for X-ray diffraction were obtained by very slow evaporation of a CH_2Cl_2/hexane solution, and the molecular structure is shown in Figure 1. Racemic **1** crystallizes in the chiral, noncentrosymmetric space group $P2_1$ (No. 4) in which there are two molecules of (P)-**1** in the unit cell related by a 2-fold crystallographic rotation axis. To our knowledge, compound **1** is the only example of a [5]helicene derivative that spontaneously forms homochiral crystals from a racemic sample. The twisted central C(5)−C(6)−C(7)−C(16)−C(17)−C(26) ring of the tetrafluorotriphenylene core of **1** has a torsion angle, C(15)−C(16)−C(17)−C(18), of 35.6° and correspondingly longer C−C bonds (average of 1.436 Å) indicative of reduced aromaticity relative to the other rings in the structure. The packing diagram (Figure 2) reveals a highly ordered structure of 1-D columns of molecules of (P)-**1** with slipped face-to-face $C{-}F\cdots C_\pi$ intermolecular interactions (F(4′)···C(1), 3.14 Å; F(1)···C(4′), 3.26 Å) along the columns.

Solid and solution samples of **1** are pale yellow and exhibit blue fluorescence (Figure 3). There is a large Stokes shift ($\Delta\lambda$ = 163 nm) between the absorption maximum (λ_{max} = 254 nm, ϵ = 41000 M^{-1} cm^{-1}) and the emission maximum (λ_{em} = 417, 435(sh) nm). Although the quantum yield for fluorescence of **1** was modest (φ_F = 0.15 relative to 9,10-diphenylanthracene in CH_2Cl_2 at room temperature), the emission band is narrow ($h_{1/2}$ = 47 nm) indicating a high purity blue emission. Interestingly, the emission of **1** is only slightly red-shifted relative to the parent (nonfluorinated) 7:8,9:10-dibenzotriphenylene[21] ($\lambda_{max} \sim 260$ nm; λ_{em} = 410, 426(sh) nm) indicating that the fluorine atoms in **1** do not exert a

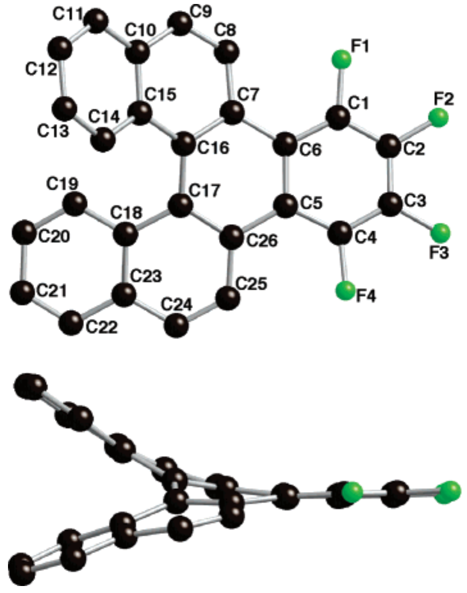


FIGURE 1. (Top) Crystalmaker depiction of the molecular structure of **1**. Selected bond lengths for the triphenylene core (Å): C(1)−C(2), 1.354(4); C(1)−C(6), 1.397(4); C(2)−C(3), 1.374(5); C(3)−C(4), 1.369(4); C(4)−C(5), 1.402(4); C(5)−C(6), 1.442(4); C(5)−C(26), 1.461(4); C(6)−C(7), 1.453(4); C(7)−C(8), 1.428(4); C(7)−C(16), 1.407(4); C(8)−C(9), 1.347(4); C(9)−C(10), 1.421(4); C(16)−C(17), 1.457(4); C(17)−C(18), 1.462(4); C(17)−C(26), 1.398(4); C(23)−C(24), 1.415(4); C(24)−C(25), 1.351(4); C(25)−C(26), 1.426(4). Average distance around the C(10)−C(15) ring, 1.396 Å. Average distance around the C(18)−C(23) ring, 1.393. Average C−F distance, 1.351 Å. (Bottom) Side view of **1** showing the P helical twist in the [5]helicene unit. Torsion angle: C(15)−C(16)−C(17)−C(18) = 35.6°.

---

[16] Tetrafluorotriphenylene **3** was originally prepared via a related ring-closing reaction between an aryllithium and an aromatic fluorine. Beaumont, C. A.; Bowen Jones, J.; Brown, D. S.; Massey, A. G.; Watkin, J. *J. Organomet. Chem.* **1988**, *344*, 1.

[17] During the preparation of this paper, the synthesis and X-ray crystal structure of 1,2,3,4-tetrafluorotriphenylene **3** was reported: Cho, D. M.; Parkin, S. R.; Watson, M. D. *Org. Lett.* **2005**, *7*, 1067.

[18] Goedicke, C.; Stegemeyer, H. *Tetrahedron Lett.* **1970**, *11*, 937.

[19] Janke, R. H.; Haufe, G.; Würthwein, E.-U.; Borkent, J. H. *J. Am. Chem. Soc.* **1996**, *118*, 6031.

[20] Gao, J. P.; Meng, X. S.; Bender, T. P.; MacKinnon, S.; Grand, V.; Wang, Z. Y. *Chem. Commun.* **1999**, 1281.

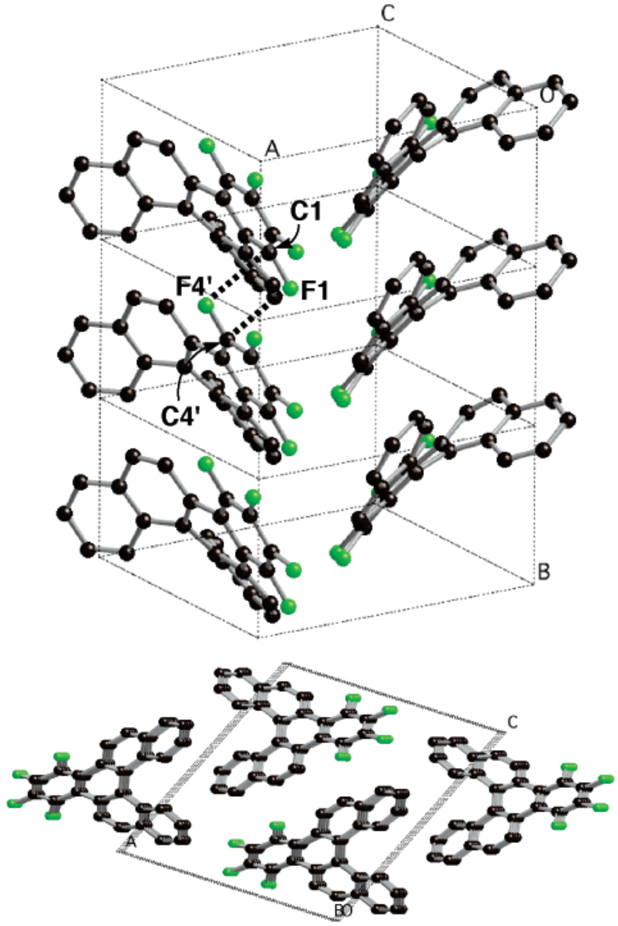


FIGURE 2. Top: View of homochiral (*P*) 1-D stacks of **1**. The dominant intermolecular interactions along a stack are slipped, face-to-face F···C_π contacts: F(4′)···C(1), 3.136 Å; F(1)···C(4′), 3.255 Å. Bottom: View of the packing diagram of **1** showing 1-D columns down the *b* axis.

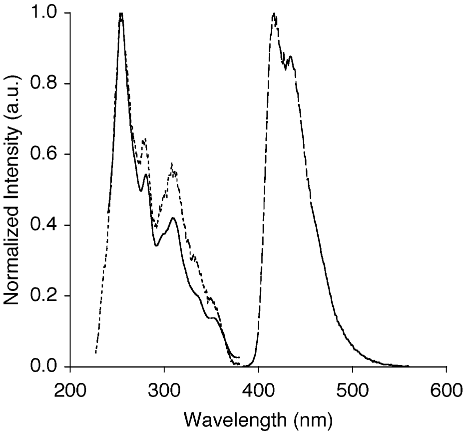


FIGURE 3. Solution UV−vis (solid line; left), excitation (λ_{em} = 417 nm; dashed, left), and emission (λ_{ex} = 254 nm; dashed, right) spectra of **1** in CH_2Cl_2 at 298 K.

significant electronic perturbation of the [5]helicene chromo-/fluorophore.

In conclusion, we have prepared a novel [5]helicene **1** and tetrafluorotriphenylene **3** in high yield by lithium−bromine exchange of 2,2′-dibromobiaryls and nucleophilic substitutions of aromatic fluorines via one- or two-step protocols. Higher [*n*]helicenes should be accessible from suitable 2,2′-dihalobiaryls using this methodology. The partially fluorinated [5]helicene **1** is a rare example of a structurally characterized [5]helicene and is the only derivative known to crystallize in an enantiomorphous space group. The ease of preparation of **1**, its high purity blue emission combined with a large Stokes shift, and the ease of substitution of aromatic fluorine atoms should allow access to derivatives suitable as fluorescent labels.

Experimental Section

2-Bromo-2′-pentafluorophenyl-1,1′-binaphthyl (2). *n*-BuLi (2.11 mL, 3.37 mmol, 1.6 M in hexane) was added dropwise to a dry THF solution (35 mL, 0.1 M) of racemic 2,2′-dibromo-1,1′-binaphthyl (1.39 g, 3.37 mmol) under an argon atmosphere at −78 °C. The resulting yellow solution was stirred for 1 h at −78 °C and then transferred via cannula to a solution of C_6F_6 (1.95 mL, 16.9 mmol) in dry THF (20 mL) at 0 °C. The yellow color was rapidly consumed upon addition to the C_6F_6 solution, and the resulting light peach colored mixture was stirred for 2 h at room temperature. The reaction mixture was quenched with water (30 mL) and repeatedly extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layer was then dried over $MgSO_4$, filtered, and concentrated in vacuo. Silica gel column chromatography gave analytically pure **2** (100% hexanes; R_f = 0.18) as a colorless oil that solidified on standing (1.28 g, 76%). Recrystallization from pentane at −35 °C gave block crystals suitable for X-ray diffraction studies. ^{1}H NMR (399.9 MHz, $CDCl_3$): δ 8.12 (d, J_{H-H} = 8.5 Hz, 1H), 8.04 (d, J_{H-H} = 8.2 Hz, 1H), 7.83 (d, J_{H-H} = 8.2 Hz, 1H), 7.74 (d, J_{H-H} = 8.8 Hz, 1H), 7.69 (d, J_{H-H} = 8.8 Hz, 1H), 7.59 (m, 1H), 7.52 (d, J_{H-H} = 8.5 Hz, 1H), 7.45 (m, 1H), 7.38 (m, 1H), 7.28−7.20 (m, 3H). ^{19}F NMR (*o*- and *m*-fluorines appear diastereotopic due to presumed hindered rotation of the C_6F_5 ring at room temperature; 282.4 MHz, $CDCl_3$): δ −136.6 (dm, J_{F-F} = 23.3 Hz, *o*-F), −137.5 (dm, J_{F-F} = 23.3 Hz, *o*-F), −155.0 (t, J_{F-F} = 21.2 Hz, *p*-F), −162.5 (m, *m*-F), −163.0 (m, *m*-F). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 138.2, 135.1, 134.1, 134.0, 132.6, 132.2, 130.2, 130.1, 129.1, 128.5, 128.20, 128.16, 127.43, 127.37, 127.0, 126.9, 126.80, 126.76, 126.5, 125.4, 123.5 (aryl *C*-F resonances were not located). Anal. Calcd for $C_{26}H_{12}F_5Br$: C, 62.55; H, 2.42. Found: C, 62.03; H, 2.09.

(*R*)-(+)-2. Preparation of (*R*)−(+)-**2** was performed as described above using (*R*)−(+)-2,2′-dibromo-1,1′-binaphthyl ($[\alpha]^{20}_D$ = 34 {*c* 0.010, pyridine}; >98% ee). Samples for optical rotation were recrystallized from pentane at −35 °C. No degradation of the optical purity was expected during this synthesis since 2-bromo-2′-lithio-1,1′-binaphthyl is known to be configurationally stable at −78 °C. (*R*)-(+)-**2**: $[\alpha]^{20}_D$ = +46 (*c* 0.060, CH_2Cl_2).

7:8,9:10-Dibenzo-1,2,3,4-tetrafluorotriphenylene (1). *n*-BuLi (1.80 mL, 2.88 mmol, 1.6 M in hexane) was added dropwise to a dry THF solution (60 mL, 0.05 M) of **2** (1.40 g, 2.80 mmol) under an argon atmosphere at −78 °C. The resulting yellow solution was allowed to warm slowly to room temperatue over a period of 18 h. The reaction mixture was quenched with water (30 mL) and repeatedly extracted with CH_2Cl_2 (3 × 50 mL), and the combined organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. Silica gel column chromatography gave **1** (100% hexanes; R_f = 0.34) as a pale yellow solid with blue fluorescence (0.80 g, 72%). Samples for elemental analysis and spectrophotometric studies were obtained by layering CH_2Cl_2 solutions with hexane at −35 °C and isolation of the microcrystalline needles by vacuum filtration. Single-needle crystals suitable for X-ray diffraction were obtained by very slow evaporation of a CH_2Cl_2/hexane solution at room temperature. UV−vis (CH_2Cl_2) λ_{max}/nm (ϵ/M^{-1} cm^{-1}): 254 (41000), 279 (21400), 310 (16900). Emission (CH_2Cl_2, λ_{ex} = 254 nm, 9.00 × 10^{-7} M) λ_{em}/nm: 417, 435 (sh). ^{1}H NMR (300.1 MHz, $CDCl_3$): δ 8.99 (d, J_{H-H} = 9.1 Hz, 2H), 8.07 (d, J_{H-H} = 9.1 Hz, 2H), 7.97 (d, J_{H-H} = 7.9 Hz, 2H), 7.90 (d, J_{H-H} = 8.6 Hz, 2H), 7.53 (m, 2H), 7.23

---

(21) Grellmann, K.-H.; Hentzschel, P.; Wismontski-Knittel, T.; Fischer, E. *J. Photochem.* **1979**, *11*, 197.

(m, 2H). ^{19}F NMR (282.4 MHz, $CDCl_3$): δ −138.4 (dm, J_{F-F} = 16.0 Hz, 2F), −157.0 (dm, J_{F-F} = 16.0 Hz, 2F). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 145.6 (dm, $^1J_{C-F}$ = 257 Hz, aryl *C*-F), 139.5 (dm, $^1J_{C-F}$ = 254 Hz, aryl *C*-F), 132.6, 130.8, 129.9, 128.9, 127.7, 127.1, 126.9, 126.1, 125.3, 122.8 (m), 117.0 (m). HRMS (EI, 70 eV): calcd for $C_{26}H_{12}F_4$ 400.08751, found 400.09053. Anal. Calcd for $C_{26}H_{12}F_4$: C, 78.00; H, 3.02. Found: C, 77.12; H, 2.88.

One-Step Synthesis of 1. *n*-BuLi (1.64 mL, 2.62 mmol, 1.6 M in hexane) was added dropwise to a dry THF solution (30 mL, 0.05 M) of 2,2′-dibromo-1,1′-binaphthyl (0.54 g, 1.31 mmol) under argon at −78 °C. The resulting yellow solution was stirred for 1 h at −78 °C. C_6F_6 (0.175 mL, 1.52 mmol) was then added to the solution, and the reaction mixture was allowed to warm to room temperature slowly over a period of 18 h. Workup and purification by silica gel column chromatography as described above gave **1** as a pale yellow solid (0.31 g, 59%).

Attempted Synthesis of Optically Active 1. The synthesis of **1** via either the sequential two-step synthesis of **1** from (*R*)-(+)-2,2′-dibromo-1,1′-binaphthyl and (*R*)-(+)-**2** or the one-step protocol from (*R*)-(+)-2,2′-dibromo-1,1′-binaphthyl gave samples of **1** with very low optical activity at all wavelengths tested ($[\alpha]^{20}_{\lambda}$ $\leq$ +5 {*c* 0.05, CH_2Cl_2}) indicating nearly complete racemization.

2-Bromo-2′-pentafluorophenylbiphenyl (4). *n*-BuLi (4.0 mL, 6.4 mmol, 1.6 M in hexane) was added dropwise to a dry THF solution (64 mL, 0.1 M) of 2,2′-dibromobiphenyl (2.0 g, 6.4 mmol) under an argon atmosphere at −78 °C. The resulting yellow solution was stirred for 1 h at −78 °C and then transferred via cannula to a solution of C_6F_6 (3.7 mL, 32 mmol) in dry THF (20 mL) at 0 °C. The yellow color was rapidly consumed upon addition to the C_6F_6 solution, and the light peach colored mixture was stirred for 2 h at room temperature. The reaction mixture was quenched with water (30 mL) and repeatedly extracted with CH_2Cl_2 (3 × 50 mL), and the combined organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. Silica gel column chromatography gave **4** (100% hexanes; R_f = 0.35) as a colorless oil that solidified on standing (1.97 g, 77%). Recrystallization from diethyl ether yielded analytically pure colorless crystals. ^{1}H NMR (300.1 MHz, $CDCl_3$): δ 7.60−7.48 (m, 3H), 7.46−7.33 (m, 2H), 7.24−7.09 (m, 3H). ^{19}F NMR (*o*- and *m*-fluorines appear diastereotopic due to presumed hinder rotation of the C_6F_5 ring; 282.4 MHz, $CDCl_3$): δ −138.8 (dd, J_{F-F} = 23.3, J_{F-F}=8.3, *o*-F), −139.2 (dd, J_{F-F} = 23.3, J_{F-F}=8.3, *o*-F), −155.1 (t, J_{F-F} = 21.2, *p*-F), −162.2 (m, *m*-F), −163.0 (m, *m*-F). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 141.8, 140.5, 132.7, 132.6, 131.3, 131.0, 129.4, 129.2, 128.1, 127.1, 126.9, 125.9, 123.1 (aryl *C*-F resonances not located). Anal. Calcd for $C_{18}H_{18}F_5Br$: C, 54.16; H, 2.02. Found: C, 54.02; H, 2.10.

1,2,3,4-Tetrafluorotriphenylene (3). *n*-BuLi (2.0 mL, 3.26 mmol, 1.6 M in hexane) was added dropwise to a dry THF solution (60 mL, 0.05 M) of **4** (1.30 g, 3.26 mmol) at −78 °C under an argon atmosphere. The resulting grayish pink solution was allowed to warm slowly to room temperature over a period of stirring for 18 h. The large amount of colorless microcrystalline precipitate that had formed was collected by vacuum filtration and washed with water and then cold CH_2Cl_2, and the mixed filtrate was extracted repeatedly with CH_2Cl_2 (3 × 40 mL). The combined organic layer was dried over $MgSO_4$ and concentrated in vacuo to give a colorless powder. Silica gel column chromatography gave **3** as a colorless microcrystalline solid (100% hexanes, R_f = 0.43) that was combined with the material isolated by filtration (total yield: 0.82 g, 84.0%). Recrystallization from hot $CHCl_3$ gave analytically pure, colorless microcrystalline needles. ^{1}H and ^{19}F NMR spectral data in warm $CDCl_3$ matched that reported previously by Watson et al.[17] ^{1}H NMR (300.1 MHz, C_6D_6): δ 8.83 (m, 2H), 8.24 (m, 2H), 7.33 (m, 4H). ^{19}F NMR (282.4 MHz, C_6D_6): δ −138.5 (dm, J_{F-F} = 16.0 Hz, 2F), −157.8 (dm, J_{F-F} = 16.0 Hz, 2F). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 130.4, 128.5, 127.9, 127.8, 127.6, 126.0, 123.3 (aryl *C*-F resonances not located). Anal. Calcd for $C_{18}H_8F_4$: C, 72.00; H, 2.69. Found: C, 71.61; H, 2.69.

Quantum Yield Measurement. The relative quantum yield for fluorescence of compound **1** was determined relative to 9,10-diphenylanthracene in CH_2Cl_2 at 298 K (φ_F = 0.95).[22] A range of concentrations of solutions of **1** and 9,10-diphenylanthracene were measured such that absorbances were less than 0.10 at the excitation wavelength (λ_{ex} = 254 nm). The quantum yield was then determined by known methods.[23]

Acknowledgment. Funding for this work was provided by the Merck Frosst Centre for Therapeutic Research and NSERC of Canada through a CRD Grant to W.E.P. and a CGS-D2 Fellowship to D.J.M.

Supporting Information Available: Experimental details and tables of crystal data, atomic coordinates, bond lengths, and angles, diagrams, and anisotropic displacement parameters for **1** and **2**. This material is available free of charge via the Internet at http://pubs.acs.org.

JO0506231

---

(22) Murov, S. L.; Carmichael, I.; Hug, G. L. *Handbook of Photochemistry*, 2nd ed.; Marcel Dekker: New York, 1993.

(23) (a) Williams, A. T. R.; Winfield, S. A.; Miller, J. N. *Analyst* **1983**, *108*, 1067. (b) Demas, J. N.; Crosby, G. A. *J. Phys. Chem.* **1971**, *75*, 991.