Supplementary Material of B204515J

Electrochiroptical response of a hexaarylethane derivative with the helical π-skeleton: Drastic UV-Vis and CD spectral changes upon electrolysis of dispiro[4,5-dibromo-9,10-dihydrophenanthrene-9,9':10,9"-bi[9H]xanthene]

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Crystallographic Details

rac-**1a**: $C_{38}H_{22}Br_2O_2$, *M* 670.40, colorless rod from $CHCl_3$, 0.4 x 0.15 x 0.15 mm, monoclinic $P2_1/c$, a = 12.462(6), b = 15.613(7), c = 15.042(7) Å, β = 111.35(1) °, *U*= 2725(2) $Å^3$, D_C (*Z* = 4) = 1.633 g cm^{-1}. A total of 6174 unique data points ($2\theta_{max}$ = 55.0 °) were measured at *T* = 153 K on a Rigaku Mercury CCD camera apparatus (Mo-Kα radiation, λ= 0.71069 Å). Numerical absorption correction was applied (μ = 30.19 cm^{-1}). The structure was solved by the direct method and refined by the full-matrix least-squares method on *F* with anisotropic temperature factors for non-hydrogen atoms. Hydrogen atoms were located at the calculated positions. The final *R* and *Rw* values are 0.036 and 0.043 for 3567 reflections with $I > 3\sigma I$ and 379 parameters. The maximum residual electron density is 0.46 e $Å^{-3}$. Atom numbering system is shown in Figure S1.

rac-**1b**: $C_{40}H_{28}O_2$, *M* 540.66, colorless plate from $CHCl_3$, 0.4 x 0.2 x 0.05 mm, monoclinic, $P2_1/n$, a = 10.059(5), b = 14.309(7), c = 19.109(10) Å, β = 97.049(7)¡, *U*= 2729(2) $Å^3$, D_C (*Z* = 4) = 1.315 g cm^{-1}. A total of 6033 unique data points ($2\theta_{max}$ = 55.0 °) were measured at *T* = 153 K on a Rigaku Mercury CCD camera apparatus (Mo-Kα radiation, λ= 0.71069 Å). Numerical absorption correction was applied (μ = 0.79 cm^{-1}). The structure was solved by the direct method and refined by the full-matrix least-squares method on *F* with anisotropic temperature

factors for non-hydrogen atoms. Hydrogen atoms were located at the calculated positions. The final *R* value is 0.057 for 2925 independent reflections with $I > 3\sigma I$ and 379 parameters. The maximum residual electron density is 0.33 e $Å^{-3}$. Atom numbering system is shown in Figure S2.

(*R*)-**6**: $C_{46}H_{40}Br_2O_6$, *M* 848.63, colorless platelet from $CHCl_3$-EtOH, 0.1 x 0.1 x 0.03 mm, monoclinic, *C*2, a = 21.614(2), b = 9.8916(8), c = 9.6740(4) Å, β = 113.459(2) °, *U*= 1897.3(2) $Å^3$, D_C (*Z* = 2) = 1.485 g cm^{-1}. A total of 2261 unique data points ($2\theta_{max}$ = 55.0 °) were measured at *T* = 123 K on a Rigaku Mercury CCD camera apparatus (Mo-Kα radiation, λ= 0.71069 Å). Numerical absorption correction was applied (μ = 21.39 cm^{-1}). The structure was solved by the Patterson method and refined by the full-matrix least-squares method on *F* with anisotropic temperature factors for non-hydrogen atoms. Hydrogen atoms were located at the calculated positions. The final *R* value is 0.032 for 1777 independent reflections with $I > 3\sigma I$ and 245 parameters. The maximum residual electron density is 0.37 e $Å^{-3}$. The axial chirality of this diasteromer was determined to be (*R*) based on the relative stereochemistry to the two asymmetric carbons derived from (*R*)-1,3-butanediol. This molecule is located on the crystallographic 2-fold axis. Atom numbering system is shown in Figure S3b.

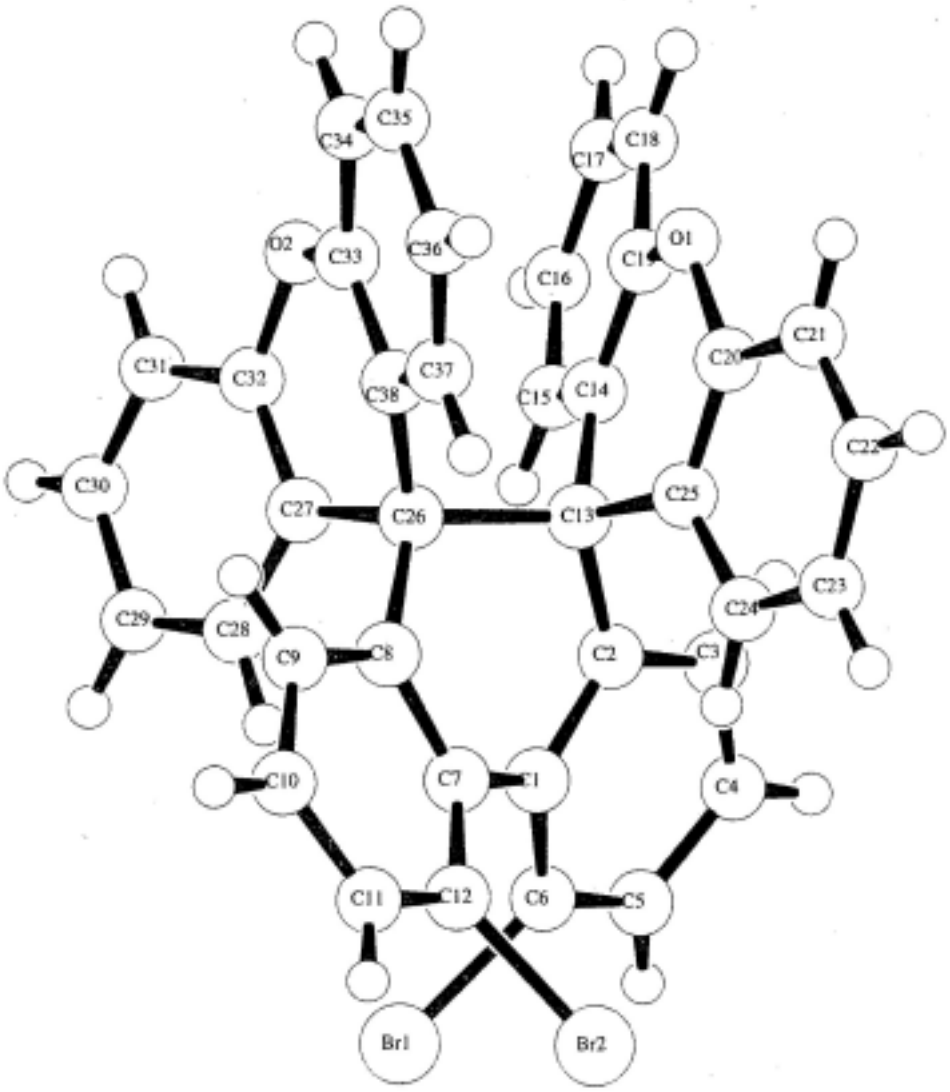

Figure S1. Ortep drawing of *rac*-**1a** showing the atom numbering system. The intramolecular contact between two Br groups is 3.423(1) Å, and the twisting angle around the biphenyl axis is 41.4(1) °.

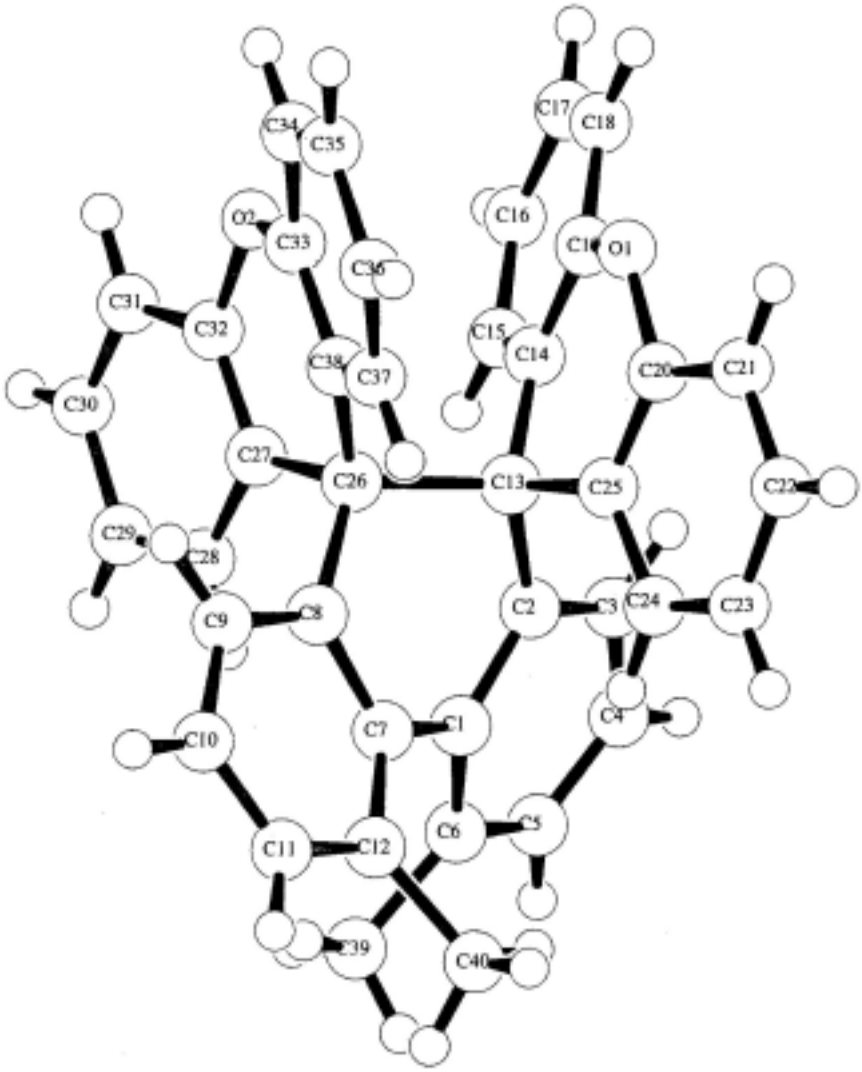

Figure S2. Ortep drawing of *rac*-**1b** showing the atom numbering system. The intramolecular contact between two Me groups is 3.017(7) Å, and the twisting angle around the biphenyl axis is 40.2(1) °.

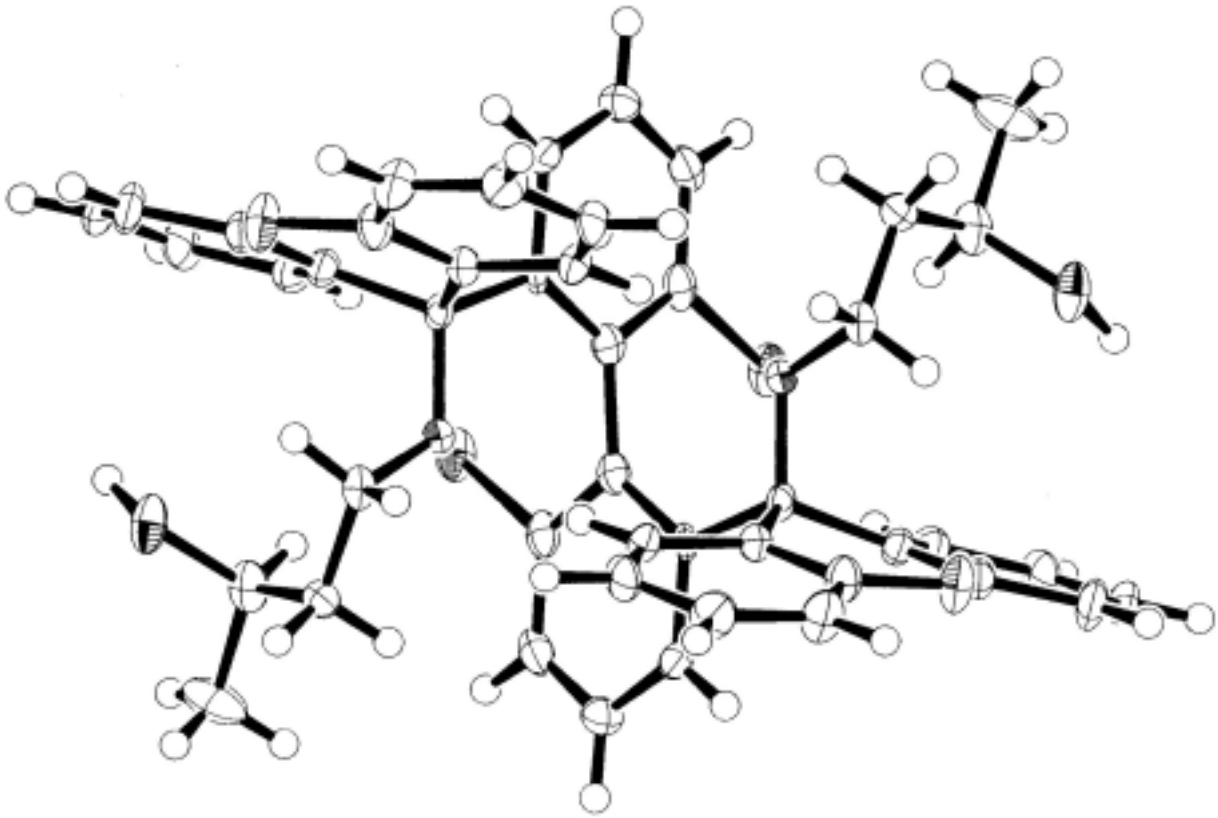

Figure S3a. Ortep drawing of (*R*)-**6**. The twisting angle around the biphenyl axis is 89.8 ° .

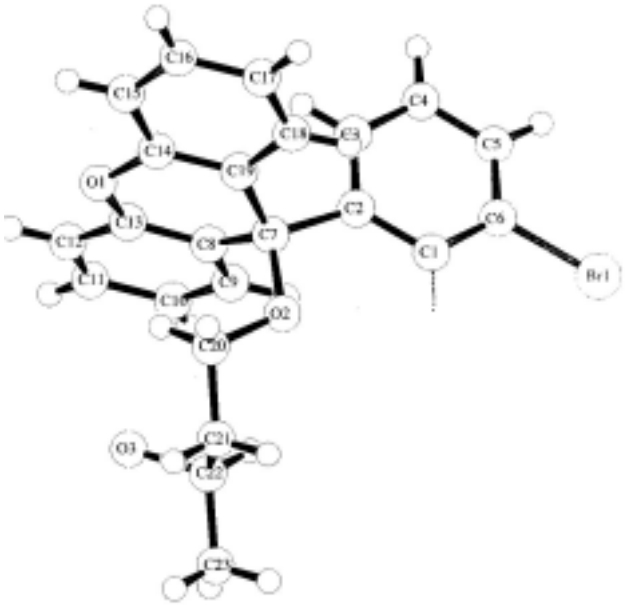

Figure S3b. Ortep drawing of (*R*)-**6** (half unit) showing the atom numbering system.