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Absolute Stereochemistry of Chiral C₆₀ Fullerene Bis-Adducts

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Address correspondence to Prof. Nobuyuki Harada, Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, 2-1-1 Katahira, Aoba, Sendai 980-8577, Japan. E-mail: n-harada@tagen.tohoku.ac.jp **ABSTRACT** To determine the absolute configuration of chiral fullerene bis-adducts, we have studied the double Bingel reaction of C_{60} with chiral tether (2*S*,3*S*)-(–)-9 derived from (*R*,*R*)-(+)-tartaric acid, and have succeeded in isolating two possible chiral bis-adducts **10a** (5%) and **10b** (2%) in addition to the C_s-symmetrically added derivative **10c** (40%). The CD spectra of chiral bis-adducts [CD(+)281]-**10a** and [CD(–)281]-**10b** show very intense Cotton effects, which are almost of mirror image, indicating that their chiral C₆₀ π -electron systems are enantiomeric each other. The ¹H and ¹³C NMR spectra of **10a** and **10b** indicate that they have C₂symmetrical structures, and the vicinal coupling constants between two equivalent protons H-2 and H-2' were determined as 1.2 Hz for **10a** and 1.8 Hz for **10b**, respectively by the ¹³C satellite band method. From the conformational analyses, the absolute configurations of these chiral C₆₀ fullerene bis-adducts were unambiguously determined as [CD(+)281]-(*S*,*S*,^f*C*)-**10a** and [CD(–)281]-(*S*,*S*,^f*A*)-**10b**, respectively.

KEYWORDS chiral fullerene, Bingel reaction, HPLC separation, absolute stereochemistry, CD Cotton effects, ¹H and ¹³C NMR spectra, ¹³C satellite band method, conformational analysis

ullerene C_{60} is a symmetrical and achiral molecule. However, the addition reaction at two chiral positions of C_{60} skeleton makes the π -electron system of fullerene chiral. Synthetic and configurational studies of such chiral fullerene bis-adducts have been carried out by several groups. For example, Nakamura et al. first synthesized chiral bisadduct 1 using a chiral tether and assigned its absolute configuration as (^fC) on the basis of MM2/Monte Carlo calculation [1] (Chart 1). Diederich et al. also synthesized chiral bis-adduct 2 as a sole product by the double Bingel reaction and the absolute stereochemistry of [CD(+)280]-2 was determined as $(S,S, {}^{f}C)$, because the molecular mechanics calculation results indicated that among two possible diastereomeric bis-adducts, diastereomer $(S,S, {}^{f}C)$ -2 is much more stable than the other diastereomer $(S,S, {}^{f}A)$ -2 [2]. The same absolute configuration was determined by the theoretical calculation of CD spectra of chiral bisadducts and related compounds based on the π -electron SCF-CI-DV



1: Nakamura et al. (1996)



3 and related compounds: Nishimura, *et al.* (1997 and 1998)

CHART 1 Previously reported chiral C₆₀ fullerene bis-adducts.

MO method [3]. Nishimura et al. reported other chiral bis-adduct **3** using a chiral tether prepared from 2,3-butanediol [4]. They also tentatively assigned the absolute configuration of **3** as shown in Chart 1 on the basis of molecular mechanics calculation results. Shinkai et al. reported the synthesis of boroncontaining chiral bis-adduct **4**, the absolute configuration of which was assigned by comparison of its CD spectrum with that of **2** [5].

In most cases only one of two possible diastereomers has been isolated and employed for absolute configurational studies, and its stereochemistry has been determined by the assumption that the isolated diastereomer should be more stable than another non-isolated diastereomer. To compare the



[CD(+)280]- $(S,S,^{f}C)$ -**2** and related compounds: Diederich, *et al.* (1997); Harada and Diederich, *et al.* (1998)



4: Shinkai, et al. (1999)

stability of two possible diastereomers, their relative steric energies were calculated by the empirical molecular mechanics method. Therefore there are two points of ambiguity in those studies of determination of absolute configuration. The first point is that the isolated and major product is not always the more stable compound. The second is that the molecular mechanics is an empirical method to calculate steric energy, and therefore it is not guaranteed that the conformer obtained by calculation is the most stable one. In some cases, the isomer obtained by calculation as the most stable conformer may not be the true one. Therefore it is essential to isolate two possible diastereomers and to compare their data. To overcome the weak points of previous absolute configurational studies and to unambiguously determine the absolute stereochemistry of chiral C_{60} bis-adducts, we took the following strategies.

The double Bingel reaction of C_{60} with a relatively tight tether containing acetonide moiety yielded only a single diastereomer **2** [2]. However, if a loose and more flexible chiral tether with known absolute configuration is employed, two possible diastereomers would be obtained and it is relatively easy to discriminate their stereostructures by comparing ¹H NMR spectral data of two diastereomers. Since the chirality of a tether part can be used as an internal reference of absolute configuration, the absolute configuration of chiral C_{60} part would be determined. As a loose and flexible tether, we have selected (2*S*,3*S*)-(–)-2,3dimethoxybutane-1,4-diyl bis(ethyl malonate) **9**, which was prepared as shown in Scheme 1.

The double Bingel reaction of C_{60} with (2*S*,3*S*)-(–)-**9** in the presence of I₂ and 1,8-diazabicyclo[5.4.0] undec-7-ene (DBU) in toluene gave a mixture of diastereomers, which was separated and purified by successive short column chromatography on silica gel (1, toluene: 2, CHCl₃/MeOH = 97/3), flash column chromatography on silica gel (CHCl₃/ toluene = 7/1), and HPLC on silica gel (CHCl₃/ toluene = 1/4) yielding chiral bis-adducts **10a** (5%) and 10b (2%) in addition to the C_s-symmetrically added derivative 10c (40%) (Chart 2). The CD spectra of chiral bis-adducts [CD(+)281]-10a and [CD (-)281]-10b show very intense Cotton effects, which are almost of mirror image, indicating that their chiral C₆₀ π -electron systems are enantiomeric each other (Figures 1 and 2). On the other hand, the major product [CD(+)310]-10c exhibits very weak CD Cotton effects reflecting its achiral C_{60} π -electron system (Figure 3).

Both compounds **10a** and **10b** have structures of C₂-symmetry, because their ¹³C and ¹H NMR spectra show 38 carbon peaks and 7 proton signals, respectively [6,7]. On the other hand, since ¹³C and









[CD(+)281]-(*S*,*S*,^{*f*}*C*)-**10a**

[CD(-)281]-(*S*,*S*,*^fA*)-**10b**



[CD(+)310]-(S,S)-10c

CHART 2 Chiral C₆₀ fullerene bis-adducts and their absolute configurations.

¹H NMR spectra of bis-adduct **10c** exhibit 76 carbon peaks and 14 proton signals, it has an asymmetric structure [8]. To determine the absolute stereochemistry of chiral bis-adducts **10a** and **10b**, the ¹H NMR spectra of their tether parts were analyzed in detail. Both compounds **10a** and **10b** are of C₂-symmetry, and therefore H-2 and H-2' protons are equivalent to each other in ¹H NMR spectra. To observe the vicinal coupling constant between these two equivalent protons, we have applied the ¹³C satellite band method [9] and obtained $J_{2,2'} = 1.2$ Hz for **10a** and $J_{2,2'} = 1.8$ Hz for **10b**. A remarkable point in the ¹H NMR spectrum of 10a is that methylene protons (H-1a and H-1'a) appear at a low magnetic field, δ 4.958 (2 H, dd, J = 10.1, 2.9 Hz), due to the anisotropy effect of ester carbonyl group, while the remaining methylene protons (H-1b and H-1'b) are at δ 4.225 (2 H, dd, J = 10.1, 9.7 Hz). The anisotropy effect of ester carbonyl group was confirmed by observing a similar phenomenon in a model compound

(±)-11 (Chart 3): δ 4.985 (2 H, d, J = 11.0 Hz, H-1a, H-1'a), 4.014 (2 H, dd, J = 11.0, 11.0 Hz, H-1b, H-1'b). Such chemical shift data clearly indicate that the H-1a and H-1'a protons of **10a** are syn to the ester carbonyl groups in the preferred conformation. Similar phenomenon was also observed in the other diastereomer **10b**: δ 5.235 (2 H, dd, J = 11.3, 6.1 Hz, H-1a, H-1'a), 4.160 (2 H, dd, J =11.3, 3.1 Hz, H-1b, H-1'b). Other important data to deduce the preferred conformation of **10a** are NOEs observed between H-1a, H-1'a protons (lower magnetic field) and OCH₃ groups, which indicate that those moieties are close to one another. In the case of 10b, other protons H-1b and H-1'b (higher magnetic field) showed NOEs with OCH₃ groups.

The Karplus equation was next applied to the observed coupling constants. Since one coupling constant leads to four dihedral angles including plus and minus signs, the combination of three observed vicinal coupling constants (J = 9.7, 2.9, and 1.2 Hz)



FIGURE 1 CD and UV spectra of chiral fullerene derivative [CD(+)281]-(S,S,^fC)-10a in CH₂ClCH₂Cl.



FIGURE 2 CD and UV spectra of chiral fullerene derivative [CD(–)281]-(S,S,^fA)-10b in CH₂ClCH₂Cl.



FIGURE 3 CD and UV spectra of fullerene derivative [CD(+)310]-(S,S)-10c in CH₂CICH₂CI.

of **10a** generates $4^3 = 64$ possible conformers for the tether part. By considering the four conditions, (1) C₂-symmetrical structure of **10a**, (2) the (*S*,*S*) absolute configuration of two OCH₃ groups, (3) *syn* conformation of H-1a and H-1'a protons to ester carbonyl groups, and (4) NOEs between H-1a, H-1'a protons and OCH₃ groups, all possible conformations were checked whether they fit the chiral (^f*C*) or (^f*A*) bis-adduct skeleton (C₆₀/two cyclopropane



 $(\pm)-11$

CHART 3 Fullerene bis-adduct (\pm) -**11** having an achiral tether.

moieties) or not. These studies indicated that only one conformer of (^fC) satisfies these conditions and coupling constant data, and its absolute stereostructure is illustrated in Figure 4 (upper). Namely the absolute stereochemistry of [CD(+)281]-**10a** was unambiguously determined as $(S,S,^{f}C)$. Similar studies led to the conclusion that the other diastereomer [CD(-)281]-**10b** has the absolute stereochemistry of $(S,S,^{f}A)$ as shown in Figure 4 (lower).

Chiral bis-adduct [CD(+)281]-(*S*,*S*,^f*C*)-**10a** was treated with K₂CO₃ in EtOH/THF to yield tetraethyl ester [CD(+)281]-(^f*C*)-**12** (29%), the ¹³C and ¹H NMR spectra of which show 37 carbon peaks and 5 proton signals, respectively [10] indicating its C₂-symmetrical structure (Scheme 2). The other chiral bis-adduct of [CD(-)281]-(*S*,*S*,^f*A*)-**10b** was converted to [CD(-)281]-(^f*A*)-**12** (19%), the ¹³C and ¹H NMR spectra were identical with those of [CD(+)281]-(^f*C*)-**12**. The enantiomeric relation between [CD(+)281]-(^f*C*)-**12** and [CD(-)281]-(^f*A*)-**12** is established by the CD spectra shown in Figure 5, which are completely opposite to each other. Those CD spectra are very similar in shape to those of chiral bis-adduct [CD(+)281]-(*S*,*S*,^f*C*)-**10a** and



FIGURE 4 Stable conformations of chiral fullerene bis-adducts (S,S, ^{f}C)-**10a** and (S,S, ^{f}A)-**10b**: most parts of C₆₀ unit are omitted, and methyl ester groups are shown instead of ethyl esters for the sake of simplicity.

[CD(-)281]- $(S,S,^{f}A)$ -**10b**, concluding that CD spectra of chiral C₆₀ bis-adducts **10a**, **10b**, and **12** mainly depend on their chiral π -electron systems of C₆₀ skeleton.

Major bis-adduct [CD(+)310]-(*S*,*S*)-**10c** was similarly converted to tetra-ethyl ester **13** (23%), the ¹³C and ¹H NMR spectra of which show 39 carbon peaks and 6 proton signals, respectively (Scheme 2) [11]. However it shows no CD reflecting its achiral C_s-symmetrical structure.

In Table 1, the typical CD data and absolute configurations of previously reported chiral bis-adducts are listed together with those of [CD(+)281]- $(S,S,^{f}C)$ -**10a**. As discussed above, the CD spectra of chiral C₆₀ bis-adducts reflect mainly the absolute stereostructure of their chiral π -electron system of fullerene skeleton, irrespective of the structure of tether parts. In fact, the CD spectra of chiral bisadducts in Table 1 are similar to one another showing a Cotton effect of medium intensity around 700 nm and an intense Cotton effect of opposite sign around 280 nm. For example, the enantiomer **10a** having (^fC) absolute configuration exhibits Cotton effects, λ_{ext} 696.0 nm ($\Delta \varepsilon$ –41.2) and 280.5 (+99.3). Therefore it is concluded that (^fC) enantiomer shows negative and positive CD Cotton effects around 700 and 280 nm, respectively, and therefore (^fA) enantiomer shows CD of opposite pattern. The CD data



SCHEME 2 Conversion of chiral fullerene bis-adducts to tetra-ethyl esters.



FIGURE 5 CD and UV spectra of chiral fullerene derivative $[CD(+)281]-({}^{f}C)-12$ and $[CD(-)281]-({}^{f}A)-12$ in CH₂ClCH₂Cl.

 TABLE 1
 Reported CD spectral data and absolute configurations of chiral fullerene bis-adducts

	This study	Nakamura et al.	Diederich et al.	Nishimura et al.	Shinkai et al.
Compound	10a	1	2	3	4
Abs. config.	$(S, S, {}^{f}C)$	$(R,R,^{f}C)$	$(S, S, {}^{f}C)$	$(S, S, {}^{\mathrm{f}}A)$	$({}^{f}C), ({}^{f}A)$
$CD \Delta \varepsilon$	-41.2	+4.69	-37.0	-36.3	+26.7
λ (nm)	696.0	720.0	706.0	737.0	726.0
$CD \Delta \varepsilon$	+99.3	-75.0	+89.0	+60.6	-73.2
λ (nm)	280.5	288.0	281.0	284.0	287.0

of chiral bis-adduct $(S,S,^{f}C)$ -**2** studied by Diederich et al. agree with the present conclusion. However, the CD data of bis-adducts $(R,R,^{f}C)$ -**1** and $(S,S,^{f}A)$ -**3** disagree with our conclusion, which implies that their absolute configurations might be revised.

In conclusion, we have succeeded in the unambiguous determination of absolute configurations of chiral C_{60} fullerene bis-adducts by intensive NMR studies of two key diastereomers.

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 H. Uehara, M. Sato, and J. Nishimura, *Enantiomer* 1998,
 3, 231–239.
- [5] T. Ishi-i, K. Nakashima, S. Shinkai, and A. Ikeda, J. Org. Chem. 1999, 64, 984–990.
- [6] Bis-adduct $[CD(+)281]-(2'S,3'S,^{+}C)-10a$: ¹H NMR (600 MHz, CDCl₃) δ 1.456 (6 H, t, J = 7.1 Hz), 3.435 (2 H, dd, J = 9.7, 2.9 Hz), 3.661 (6 H, s), 4.225 (2 H, dd, J = 10.1, 9.7 Hz), 4.490 (2 H, dq, J = 10.7, 7.1 Hz), 4.541 (2 H, dq, J = 10.7, 7.1 Hz), 4.958 (2 H, dd, J =10.1, 2.9 Hz); ¹³C satellite band, ¹H NMR (600 MHz, CDCl₃) δ 3.435 (0.02 H, dddd, J = 142.8, 9.7, 2.9, 1.2 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 14.22, 48.89, 59.39, 61.32, 63.68, 69.12, 71.67, 75.47, 129.56,

135.68, 139.03, 139.60, 140.49, 141.05, 141.05, 141.07, 141.44, 142.02, 142.21, 142.31, 143.62, 143.74, 144.21, 144.46, 144.53, 144.70, 144.84, 144.95, 145.02, 145.09, 145.34, 145.63, 145.67, 145.67, 146.48, 146.71, 162.75, 163.23; FAB-MS, calcd for $C_{76}H_{22}O_{10}$: 1094.1213. Found: 1094.1216.

- [7] Bis-adduct [CD(–)281]-(2'*S*,3'*S*,^f*A*)-**10b**: ¹H NMR (600 MHz, CDCl₃) δ 1.444 (6 H, t, J = 7.1 Hz), 3.441 (2 H, dd, J = 6.1, 3.1 Hz), 3.579 (6 H, s), 4.160 (2 H, dd, J = 11.3, 3.1 Hz), 4.500 (2 H, dq, J = 10.7, 7.1 Hz), 4.530 (2 H, dq, J = 10.7, 7.1 Hz), 5.235 (2 H, dd, J = 11.3, 6.1 Hz); ¹³C satellite band, ¹H NMR (600 MHz, CDCl₃) δ 3.441 (0.02 H, dddd, J = 142.6, 6.1, 3.1, 1.8 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 14.23, 49.00, 58.93, 63.57, 65.41, 69.09, 71.50, 80.71, 130.25, 136.56, 138.41, 139.01, 140.92, 140.96, 141.01, 141.63, 141.68, 141.79, 142.26, 142.44, 143.60, 143.65, 144.43, 144.48, 144.58, 144.72, 144.75, 145.02, 145.02, 145.10, 145.10, 145.32, 145.64, 145.72, 146.44, 146.69, 162.48, 163.09; FAB-MS, calcd for C₇₆H₂₂O₁₀: 1094.1213. Found: 1094.1233.
- [8] Bis-adduct [CD(+)310]-(2'S,3'S)-10c: ¹H NMR (600 MHz, CDCl₃) δ 1.388 (3 H, t, J = 7.1 Hz), 1.406 (3 H, t, J = 7.1Hz), 3.286 (1 H, ddd, J = 6.8, 3.5, 1,8 Hz), 3.604 (3 H, s), 3.676 (3H, s), 3.779 (1 H, ddd, J = 8.0, 3.5, 1.6 Hz), 4.209 (1 H, dd, J = 11.4, 1.6 Hz), 4.394 (1 H, dq, J = 10.8, 7.1 Hz), 4.400 (1 H, dq, J = 10.7, 7.1 Hz), 4.476 (1 H, dq, J = 10.8, 7.1 Hz), 4.480 (1 H, dq, J = 10.7, 7.1 Hz), 4.521 (1 H, dd, J = 11.6, 6.8 Hz), 4.733 (1 H, dd, J = 11.6, 1.8 Hz), 4.964 (1 H, dd, J = 11.4, 8.0 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 14.07, 14.19, 48.88, 49.08, 58.87, 58.93, 62.70, 63.38, 63.45, 63.64, 67.04, 67.09, 70.27, 70.29, 79.85, 80.27, 134.49, 136.84, 136.98, 137.66, 137.92, 138.19, 138.39, 139.12, 140.96, 141.06, 141.29, 141.34, 141.96, 142.35, 142.36, 142.39, 143.15, 143.20, 143.63, 143.67, 143.89, 144.00, 144.11, 144.28, 144.28, 144.29, 144.36, 144.44, 144.46, 144.66, 144.72, 144.75, 144.81, 145.05, 145.17, 145.22, 145.26, 145.29, 145.32, 145.34, 145.38, 145.41, 145.52, 145.69, 145.76, 145.92, 145.99, 146.02, 146.16, 146.22, 147.37, 147.46, 147.56, 147.56, 148.92, 150.04, 162.45, 162.75, 162.86, 162.96; FAB-MS, calcd for C₇₆H₂₂O₁₀: 1094.1213. Found: 1094.1235.
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- [10] [CD(+)281]-([†]C)-12: ¹H NMR (400 MHz, CDCl₃) δ 1.433
 (6 H, t, J = 7.1 Hz), 1.435 (6 H, t, J = 7.1 Hz), 4.416
 (2 H, dq, J = 10.7, 7.1 Hz), 4.467 (2 H, dq, J = 10.7, 7.1 Hz), 4.493 (4 H, q, J = 7.1 Hz); ¹³C NMR (100 MHz,

CDCl₃) δ 14.10, 14.19, 48.78, 63.07, 63.23, 69.27, 71.87, 130.93, 137.81, 138.21, 138.55, 140.73, 141.00, 141.60, 141.84, 141.99, 142.26, 142.39, 142.54, 143.42, 143.61, 144.25, 144.41, 144.41, 144.44, 144.69, 144.81, 144.99, 145.12, 145.16, 145.27, 145.48, 145.60, 146.39, 146.62, 162.96, 163.74; UV-Vis (ClCH₂CH₂Cl) λ_{max} 316.5 nm (ε 35,200), 255.5 (102,000); CD (ClCH₂CH₂Cl) λ_{ext} 700.5 nm ($\Delta \varepsilon$ -37.0), 486.5 (+23.0), 378.0 (-26.2), 338.5 (+60.9), 280.5 (+98.3), 262.0 (-146.1); FAB-MS, calcd for C₇₄H₂₀O₈: 1036.1158. Found: 1036.1161.

[11] Achiral tetra-ethyl ester **13**: ¹H NMR (400 MHz, CDCl₃) δ 1.389 (6 H, t, J = 7.1 Hz), 1.457 (6 H, t, J = 7.1 Hz), 4.403 (2 H, dq, J = 10.8, 7.1 Hz), 4.435 (2 H, dq, J = 10.8, 7.1 Hz), 4.454 (2 H, dq, J = 10.6, 7.1 Hz), 4.510 (2 H, dq, J = 10.6, 7.1 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 14.10, 14.13, 49.46, 63.11, 63.14, 67.80, 70.50, 134.97, 136.94, 137.37, 138.41, 139.65, 141.07, 141.28, 142.47, 142.82, 143.36, 143.59, 143.69, 144.22, 144.28, 144.37, 144.55, 144.67, 145.04, 145.22, 145.28, 145.37, 145.73, 145.82, 145.89, 145.94, 146.20, 147.27, 147.38, 147.45, 148.66, 163.05, 163.47; UV-Vis (CICH₂CH₂CI) λ_{max} 321.5 nm (ε 34,500), 259.5 (111,000); APCI-MS, calcd for C₇₄H₂₀O₈: 1036.1158. Found: 1036.1158.