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Journal of Fluorine Chemistry 122 (2003) 201–205
JOURNAL OF FLUORINE CHEMISTRY
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Intermolecular oxygen atom···π interaction in the crystal packing of chiral amino alcohol bearing a pentafluorophenyl group

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Received 29 January 2003; received in revised form 10 March 2003; accepted 14 March 2003

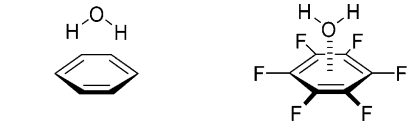
Abstract

Presence of an unique atom-to-face alcoholic oxygen atom···π interaction between a pentafluorophenyl group and alcoholic oxygen (O···π) has been demonstrated by X-ray analysis of the novel chiral amino alcohol instead of the well-known interaction between usual aromatic ring and alcoholic hydrogen atom (OH···π).

Keywords: Aromatic interaction; Attractive interaction; Chiral ligand; Amino alcohol; Asymmetric synthesis

1. Introduction

Intermolecular aromatic interactions (e.g. π–π [1], XH···π [2], cation-π [3], etc.) are relatively weak in comparison with typical hydrogen bonds, although these forces plays a significant role in molecular recognition [4] in both biological [5] and chemical systems.[1,2] Under such circumstance, perfluorophenyl groups construct occasional interactions with electron-rich aromatic rings. Thus, the mixture of hexafluorobenzene and benzene forms a 1:1 co-crystal with a face-to-face stacked arrangement [8], while the preferential geometry of the benzene dimer is edge-to-face [1,9] (i.e. CH···π interaction). Theoretical studies[3] suggest that hexafluorobenzene interacts with several electron-rich atoms (atom-to-face interaction), in contrast to the preferential XH···π or cation-π interaction of benzene. Recently, Gallivan and Dougherty investigated the mode of interaction between hexafluorobenzene and a water molecule using ab initio calculations [11] (Fig. 1). The lone pair electrons of oxygen is situated over the face of the π system [11] (oxygen atom···π interaction, i.e. O···π interaction (Fig. 1b)), whereas the hydrogen atom of a water molecule interacts with the benzene plane [12] (OH···π interaction (Fig. 1a)). Recently, O···π interactions between perfluorophenyl groups and an oxygen atom of amide carbonyl were disclosed by X-ray analysis of carbonic anhydrase II inhibitors.[4]



a: OH• • • π interaction **b**: O• • • π interaction

Fig. 1. Interaction of water with benzene (a), and with hexafluorobenzene (b).

Here, we wish to report the first observation of an O···π interaction between a pentafluorophenyl group and alcoholic oxygen in the crystal packing of the novel chiral amino alcohol, (1*R*,2*S*)-2-amino-1-(pentafluorophenyl)-2-phenylethanol (**1**) (other chiral diol or amino alcohol bearing a pentafluorophenyl group [14], fluorinated aromatic ligand for asymmetric catalyst [15]), which we prepared as a promising new chiral ligand [16]. This finding of the O···π interaction supports Dougherty's prediction [11] (Fig. 1b), because the alcoholic oxygen is similar to that

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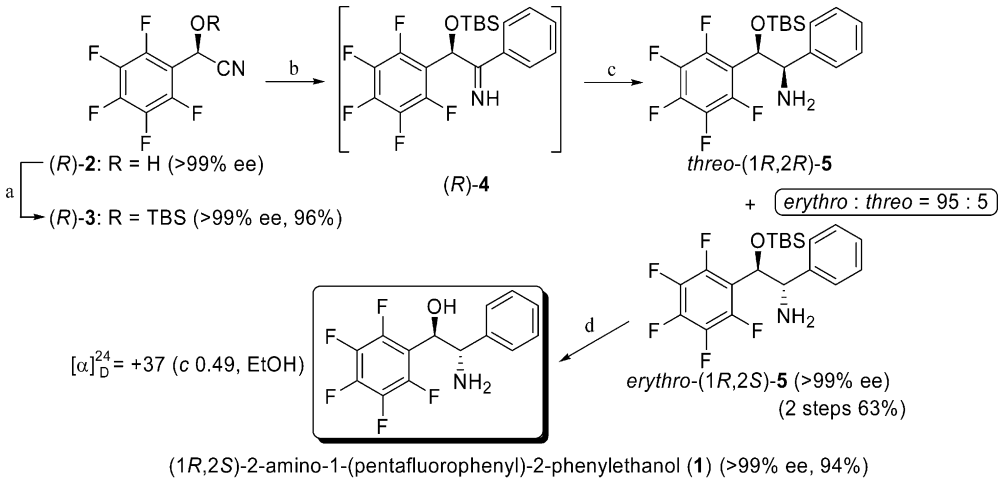
[1] Effect of an attractive interaction between a pentafluorophenyl group and an enolate anion on the diastereoselective reactions [6].

[2] Examples of aromatic interaction in asymmetric synthesis, see [7].

[3] The interactions in X-ray crystals have also been investigated by searching the Cambridge structural database (CSD). However, tangible examples have not been shown, and moreover, alcoholic compounds have not been contained in these searches [10].

[4] The $sp^2 \rightarrow \pi^*$ charge-transfer complexation dominate over the interaction between amide carbonyl and pentafluorophenyl groups, although the electrostatic interaction is favored in the case of oxygen atom (sp^3) of a water molecule [13].

0022-1139/$ – see front matter
doi:10.1016/S0022-1139(03)00089-7



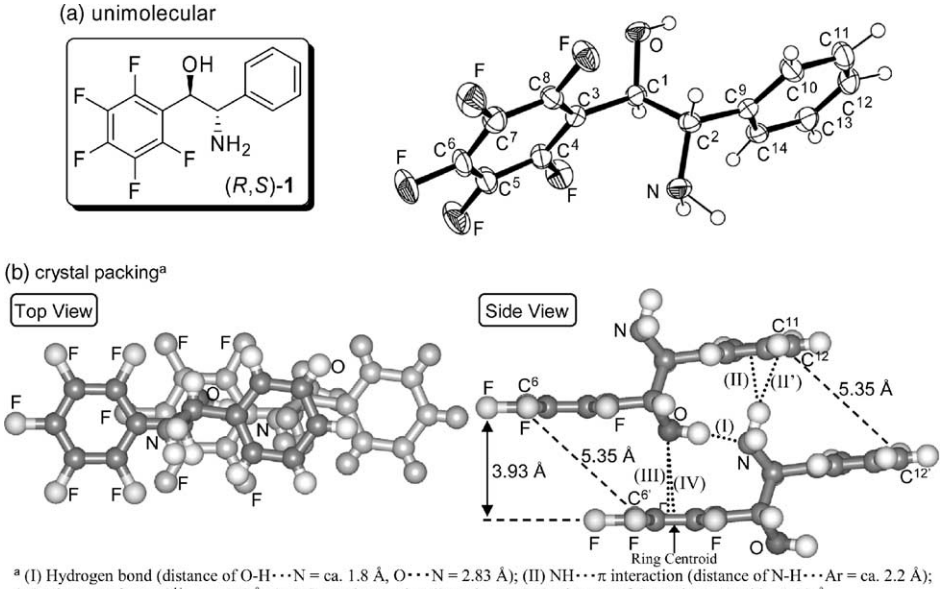
[a] Conditions: (a) TBS-OTf, DMAP, CH_2Cl_2, 0 °C; (b) PhLi (2.0 M) in cyclohexane-Et_2O (70:30), TMS-Cl, Et_2O, – 85 °C; (c) $NaBH_4$, EtOH, 0 °C; (d) TBAF, THF, 0 °C.

Scheme 1. Synthesis of (1*R*,2*S*)-**1**.

of a water molecule. Elucidation of such intra- or intermolecular interactions (intramolecular interaction between a pentafluorophenyl group and a nitrogen atom [17]) would be significant for the development of a new molecular recognition system, especially for asymmetric synthesis.[1,2]

2. Results and discussion

Synthesis of the desired amino alcohol **1** was performed as shown in Scheme 1. Enantiomerically pure (*R*)-cyanohydrin **2** [14c], which was prepared by enzymatic optical resolution, was protected with *tert*-butyldimethylsilyl trifluoromethanesulfonate (TBS-OTf) without racemization [14c]. (*R*)-Cyanohydrin **3** was treated with PhLi in the presence of tetramethylsilane (TMS)-Cl (addition of TMS-Cl was essential in this reaction [18]) in Et_2O at −85 °C to give the corresponding (*R*)-imine **4**, which without purification was reduced with $NaBH_4$ in EtOH at 0 °C to give amino alcohol **5** in 90% de. After purification by silica-gel column chromatography, the major diastereomer (1*R*,2*S*)-**5** was obtained in >99% ee and 63% yield (two steps). Treatment with 1.1 eq. of tetrabutylammonium fluoride (TBAF) gave enantiomericallly pure **1** in 94% yield ($[\alpha]_D^{24} = +37$ (*c* 0.49, EtOH)). The relative configuration of **1** was determined to be *erythro* by X-ray analysis (Fig. 2). The diastereoselectivity is consistent with that in the reduction of similar imines derived from chiral cyanohydrins [14a,19].



[a] (I) Hydrogen bond (distance of O-H···N = ca. 1.8 Å, O···N = 2.83 Å); (II) NH···π interaction (distance of N-H···Ar = ca. 2.2 Å); (II') Distance of H···C^{11} = ca. 2.6 Å; (III) O···π interaction (See Fig. 3); (IV) Distance of O···Ring centroid = 3.08 Å.

Fig. 2. X-ray analysis of (1*R*,2*S*)-**1**.

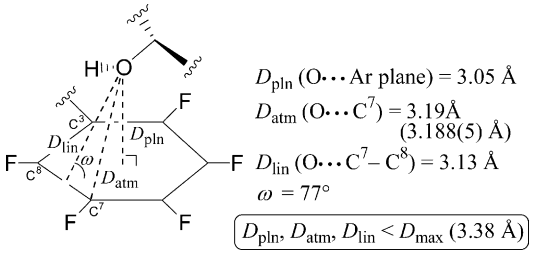


Fig. 3. Analysis of location of alcohol's oxygen toward the pentafluorophenyl group in the crystal packing of **1** for confirmation of O···π interaction.

We initially expected that the intra- and/or intermolecular π–π interaction(s) between the pentafluorophenyl and phenyl groups [1,8] operates predominantly to regulate the crystal packing of the amino alcohol **1**, because we had observed a similar π–π stacking between pentafluorophenyl and naphthyl groups [14c]. However, those interactions were not observed in this crystal structure. As shown in Fig. 2a, the dihedral angle (C^3–C^1–C^2–C^9) is 172°, which indicates that pentafluorophenyl and phenyl groups are in the anti conformation.

As shown in Fig. 2b (side view), the two molecules are stacked head-to-head, and the same distances of $C^6 \cdots C^{6'}$ and $C^{12} \cdots C^{12'}$ (5.35 Å) indicate the slipped-parallel orientation. The crystal packing (up and down) seems to be regulated principally by the O–H···N hydrogen bond[5] (Fig. 2b (I)), which, however, would not be sufficient for the parallelization of the two stacked molecules. Judging from the packing structure, the atom···π interactions could assist the parallelization, one of which would be an N–H···π interaction[6] (Fig. 2b (II)), which is known to be weak [2b]. The other example we discuss here is the interaction between alcoholic oxygen and the pentafluorophenyl group (O···π interaction (III)). The distance between the oxygen and the ring centroid is 3.08 Å (Fig. 2b (IV)), which is slightly longer than that for the reported amide oxygen (2.9 Å),[4] probably due to the different nature of sp^2 and sp^3 oxygen atoms.

The conventional method, based on only the distance between the oxygen atom and the aromatic ring,[3,4] was insufficient. Thus, the presence of such an O···π interaction was examined at several distances, which were defined by Nishio et al. for survey of XH···π (especially CH···π) interactions [2].[7] The obtained distances are D_{pln} (O···Ar plane), D_{atm} (O···C^7), D_{lin} (O···C^7–C^8), and D_{max} (3.38 Å) {[sum of the van der Waals radii [21] (3.22 Å) of oxygen r_w^O (1.52 Å) and carbon r_w^C (1.70 Å)] × 1.05} (Fig. 3) [2]. Existence of the O···π interaction was judged by comparison of a couple of conditions in which (a) all the distances of D_{pln}, D_{atm}, and D_{lin} are shorter than D_{max}, and (b) angle ω [O–(C^6–C^7)–Ar plane] is smaller than 90° [2]. In fact, all the distances D_{pln}, D_{atm}, D_{lin} were shorter than D_{max} as well as the sum of the van der Waals radii of oxygen r_w^O and carbon r_w^C. In addition, ω was smaller than 90°. These data satisfied Nishio's definition for the presence of an O···π interaction.

The position of the oxygen in close proximity to the pentafluorophenyl group is reasonably explained not only by the assistance of the two interactions of O–H···N and N–H···π but also that of an O···π interaction, because (a) the strength of the N–H···π interaction (<2 kcal mol^{-1}) [22] is estimated to be similar to that of an O···π interaction for a water molecule [11] and is not enough to parallelize both aromatic rings, and (b) the stronger O–H···N interaction is not directed to the pentafluorophenyl group to regulate the geometry.

3. Conclusion

The observed distance of the O···Ar (C_6F_5) plane (i.e. D_{pln} = 3.05 Å) was rather shorter than that calculated for a water oxygen and hexafluorobenzene (3.20 Å) [11], despite the lower electron density of the former alcoholic oxygen located nearby the pentafluorophenyl group. These results conclude us that the pentafluorophenyl group has the potential to interact with an alcoholic oxygen atom. These findings will be significant in the design of novel ligands for asymmetric synthesis and/or supramolecular systems for advanced materials.

4. Experimental

4.1. General methods

All reactions were carried out under a nitrogen atmosphere with dry and freshly distilled solvents under anhydrous conditions, unless otherwise noted. Tetrahydrofuran (THF) was distilled from sodium, and dichloromethane (CH_2Cl_2) was distilled from calcium hydride. Reactions were monitored by thin-layer chromatography with precoated silica-gel plates (Merck 60 F_{254}, plate length 40 mm). As a usual workup procedure, the reaction mixture was extracted with ethyl acetate (EtOAc). The organic layer was dried over $MgSO_4$, filtrated with suction, and then concentrated in vacuo. Preparative column chromatography was carried out by using silica-gel (Fuji Silysia BW-127 ZH, 100–270 mesh). ^1H and ^{13}C NMR spectra were measured at 200 MHz (or 300 MHz) and 50 MHz (or 75 MHz), respectively, and chemical shifts are given relative to tetramethylsilane. ^{19}F NMR spectra were measured at 188 MHz (or 282 MHz) and chemical shifts are given relative to C_6F_6.

[5] The distance between hydrogen and nitrogen atoms is ca. 1.8 Å and that for oxygen and nitrogen atoms is 2.83 Å which is within a range of general O–H ···N hydrogen bonds [20].

[6] The distance between the hydrogen atom and the nearest carbon atom in the phenyl group is ca. 2.6 Å (Fig. 2b (II)). This distance is within a range of general N–H ···Ar interaction [2b].

[7] In addition to distance parameters, an angle parameter θ was considered in the CH ···π interaction [2]. However, atomic natures, especially electronic distribution, differ vastly between hydrogen and oxygen atoms. Therefore, angle parameter θ was excluded here.

4.2. (R)-2-(tert-Butyldimethylsiloxy)-2-(pentafluorophenyl)acetonitrile ((R)-3)

TBS-OTf, 2.60 ml, 11.3 mmol was slowly added to a solution of cyanohydrin (*R*)-**2** (1.67 g, 7.50 mmol, >99% ee) (obtained by the lipase LIP-catalyzed transesterification of cyanohydrin (±)-**2** as reported in [14c]) and 4-(dimethylamino)pyridine (DMAP, 1.85 g, 10.0 mmol) in CH_2Cl_2 (20 ml) under cooling in an ice bath. After being stirred for 2.5 h, the mixture was acidified (pH 4) by addition of 3% aqueous HCl. The resulting mixture was treated in a usual manner. The residual mixture was purified by column chromatography (SiO_2, hexane/EtOAc (10:1)) to give (*R*)-**3** (2.62 g, 96% yield, 99% ee) as a colorless oil: $R_f = 0.58$ (SiO_2, hexane/EtOAc (4:1)); ^{1}H NMR (200 MHz, $CDCl_3$) δ 0.13 (s, 3H), 0.26 (s, 3H), 0.90 (s, 9H), 5.77 (s, 1H); ^{13}C NMR (50 MHz, $CDCl_3$) δ −5.4, −5.3, 18.1, 25.4, 54, 111.2 (m), 116.6, 135.2 (m), 140.4 (m), 142.4 (m), 147.5 (m); ^{19}F NMR (188 MHz, $CDCl_3$) δ 1.7–2.1 (m, 2F), 11.6 (t, $J = 20.3$ Hz, 1F), 20.0–20.2 (m, 2F); IR (neat) 2958, 2934, 2862, 1513, 1003 cm^{-1}. Anal. Calcd for $C_{14}H_{16}NOSi$: C, 49.84; H, 4.78; N, 4.15. Found: C, 49.47; H, 4.47; N, 4.49; HPLC analysis: retention time = 10.7 and 11.5 min for (*S*)- and (*R*)-**3** (0.5:99.5), respectively. The conditions of HPLC are as follows: Daicel CHIRALCEL OD-H, 4.6 mm × 25 cm, hexane/*i*-PrOH (300:1), 0.5 ml min^{-1}, UV 254 nm; $[\alpha]_D^{28} = +30.1$ (*c* 0.87, $CHCl_3$).

4.3. (1R,2S)-2-(tert-Butyldimethylsiloxy)-2-(pentafluorophenyl)-1-phenylethylamine (erythro-5)

A solution of TBS ether (*R*)-**3** (1.69 g, 5.00 mmol) in Et_2O (17 ml) was stirred at −85 °C for 10 min. After addition of chlorotrimethylsilane (TMS-Cl, 0.95 ml, 7.5 mmol), a solution of phenyllithium (1.8 M in cyclohexane–ether (7:3), 3.7 ml, 7.50 mmol) was added dropwise over 10 min. After the mixture being stirred for additional 1.5 h at the temperature, a suspension of $NaBH_4$ (284 mg, 7.50 mmol) in EtOH (10 ml) was added at 0 °C. After being stirred for additional 1.5 h at the temperature, the combined mixture was acidified (pH 4) with 3% aqueous HCl, and then saturated aqueous $NaHCO_3$ solution was added to become pH 9. The resulting mixture was treated in a usual manner. The residual oil was purified by column chromatography (SiO_2, hexane/EtOAc (20:1) to (5:1)) to give (1*R*,2*S*)-**5** (1.30 g, 63% yield) and (1*R*,2*R*)-**5**, (1*R*,2*S*)-**5**/(1*R*,2*R*)-**5** = 95:5, which was determined by ^{1}H NMR spectra.

For (1*R*,2*S*)-**5**. Colorless oil; $R_f = 0.35$, (hexane/EtOAc (4:1)); ^{1}H NMR (200 MHz, $CDCl_3$) δ −0.39, (s, 3H), −0.29 (s, 3H), 0.66 (s, 9H), 1.43 (s, 2H), 4.31 (d, $J = 8.2$ Hz, 1H), 4.94 (d, $J = 8.2$ Hz, 1H), 7.26–7.35 (m, 5H); ^{19}F NMR (188 MHz, $CDCl_3$) δ −0.43 (s, 2F), 6.72 (t, 1F), 20.4–20.9 (m, 2F); ^{13}C NMR (50 MHz, $CDCl_3$) δ −5.9, −5.6, 17.9, 25.5, 60.6, 72.5, 116.4 (m), 127.5, 127.9, 128.4, 135.0 (m), 138.1 (m), 140.0 (m), 142.7 (m), 142.9, 147.6 (m); IR (neat) 3394 (NH_2), 2955, 2858, 1520, 1503, 999 cm^{-1}. Anal. Calcd for $C_{19}H_{21}F_5NOSi$: C, 57.54; H, 5.79; N, 3.36. Found: C, 57.18; H, 5.83; N, 3.50.

For (1*R*,2*R*)-**5**. Yellow oil; $R_f = 0.36$ (hexane/EtOAc (4:1)); ^{1}H NMR ($CDCl_3$) δ −0.17, (s, 3H), 0.07 (s, 3H), 0.88 (s, 9H), 1.48 (d, 2H), 4.38 (d, $J = 8.4$ Hz, 1H), 4.98 (d, $J = 8.4$ Hz, 1H), 7.20–7.27 (m, 5H).

4.4. (1R,2S)-2-Amino-1-(pentafluorophenyl)-2-phenylethanol (erythro-1)

To a solution of amine (1*R*,2*S*)-**5** (240 mg, 0.57 mmol) in THF (2.0 ml) was added *tetra-n*-butylammonium fluoride (TBAF, 1 M in THF, 0.57 ml, 0.57 mmol), and then the mixture was stirred for 1 h at 0 °C. The resulting mixture was concentrated in vacuo, and the residual viscous oil was purified by column chromatography (SiO_2, EtOAc) to give (1*R*,2*S*)-**1** (161 mg, 94% yield) as a white solid. The optical purity was determined by HPLC analysis after acetylation (see below): mp, 174–175 °C, $R_f = 0.53$ (SiO_2, EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.85 (br, 3H), 4.32 (d, $J = 7.5$ Hz, 1H), 5.10 (d, $J = 7.5$ Hz, 1H), 7.27–7.34 (m, 5H); ^{19}F NMR (282 MHz, $CDCl_3$) δ −0.36 −0.18 (m, 2F), 7.06 (t, $J = 21.0$ Hz, 1F), 19.9 (dd, $J = 7.1$, 21.0 Hz, 2F); ^{13}C NMR (75 MHz, $CDCl_3$) δ 60.0, 71.1, 114.5 (m), 126.7, 128.2, 128.7, 135.6 (m), 138.8 (m), 141.1, 142.1 (m), 143.2 (m), 146.6 (m); IR (KBr) 3365, 3304 (OH), 3107 (NH_2), 1523, 1499, 995 cm^{-1}. Anal. Calcd for $C_{14}H_{10}F_5NO$: C, 55.45; H, 3.32; N, 4.62. Found: C, 55.69; H, 3.35; N, 4.61; $[\alpha]_D^{24} = +37$ (*c* 0.49, EtOH).

4.5. Acetylation of (1R,2S)-1 for HPLC analysis

Pyridine (0.5 ml) was added to a solution of (1*R*,2*S*)-**1** (20 mg, 0.066 mmol) and AcCl (45 μl, 0.66 mmol) in toluene (1.0 ml) at 0 °C. After removal of a cooling bath, the reaction mixture was stirred for 4 h. The mixture was acidified (pH 4) with 3% aqueous HCl, and extracted with ethyl acetate. The extract was treated in a usual manner. The residual solid was purified by column chromatography (SiO_2, hexane/EtOAc (2:1)) to give diacetate of **1** (21 mg, 0.055 mmol, 83% yield, 99.4% ee) as a white solid. The optical purity was determined by HPLC analysis: retention time = 15.8 and 48.4 min for diacetate of (1*R*,2*S*)-**1** and (1*S*,2*R*)-**1** (99.7:0.3), respectively. The conditions of HPLC are as follows: column; Daicel CHIRALCEL OD-H, ϕ 4.6 mm × 25 cm, hexane/*i*-PrOH (19:1)), 0.8 ml min^{-1}, UV 254 nm; $[\alpha]_D^{24} = +17$ (*c* 0.15, EtOH).

4.6. X-ray analysis of (1R,2S)-1

The single crystal growth was carried out in a dichloromethane–hexane mixed solvent at room temperature. Crystal data for (1*R*,2*S*)-**1**: $C_{14}H_{10}F_5NO$, Fw = 303.23, triclinic, space group *P*1, $a = 6.365(2)$, $b = 10.207(4)$, $c = 5.348(2)$ Å, $\alpha = 99.539(5)^\circ$, $\beta = 100.06(3)^\circ$, $\gamma = 101.774(2)^\circ$, $V = 327.6(2)$ Å^3, $T = 150.2$ K, $Z = 1$,

$D_{calc} = 1.537$ g cm^{-3}, $R = 0.077$ for 1205 observed reflections [$I > 3.00\sigma(I)$] and 206 variable parameters. All measurements were made on a Rigaku RAXIS-IV imaging plate area detector with Mo Kα radiation. Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-205107.

Acknowledgements

This work was partially supported by the Ministry of Education, Science, Sports and Culture of Japan. We are grateful to Dr. Toshiyuki Oshiki in Okayama University for his kind assistance in X-ray analysis of **1**, and to Toyobo Co. Ltd. (Osaka, Japan) for giving us lipase LIP. We also thank the SC-NMR Laboratory of Okayama University for ^{1}H, ^{13}C, and ^{19}F NMR measurements and the Venture Business Laboratory of Okayama University for X-ray analysis.

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