Enantioselective Photocyclization of Amides to β-Lactam Derivatives in Inclusion Crystals with an Optically Active Host

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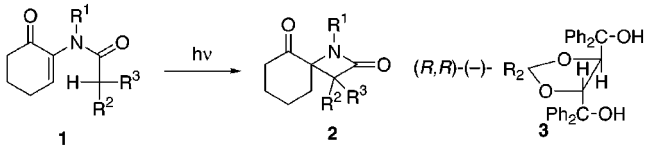
Irradiation of inclusion crystals of 2-(*N*-acyl-*N*-alkylamino)cyclohex-2-enones and *N*,*N*-dimethylphenylglyoxylamide with chiral host molecules gave the optically active *N*-alkyl-1-azaspiro[3.5]nonane-2,5-diones and 3-hydroxy-1-methyl-3-phenylazetidin-2-one, respectively. The crystal structure of the 1:1 inclusion complex of *N*,*N*-dimethylphenylglyoxylamide with (−)-*trans*-1,4-bis[3-(*o*-chlorophenyl)-3-hydroxy-3-phenylprop-1-ynyl]-2,3,5,6-tetrachloro-2,5-cyclohexadiene-1,4-diol was analyzed by X-ray diffraction.

It has been reported that the photocyclization reaction of 2-(*N*-acyl-*N*-alkylamino)cyclohex-2-enones (**1**) in acetone gives the racemic *N*-alkyl-1-azaspiro[3.5]nonane-2,5-diones (**2**).[1] However, no enantioselective photoreaction of **1** has yet been accomplished. To achieve high enantioselectively, we studied the photoreaction of inclusion crystals of **1** with the chiral hosts **3**, derived from tartaric acid. We first found that irradiation of **1** with (*R*,*R*)-(−)-*trans*-4,5-bis(hydroxydiphenylmethyl)-2,2-dimethyl-1,3-dioxacyclopentane (**3a**), (*R*,*R*)-(−)-*trans*-4,5-bis(hydroxydiphenylmethyl)-1,4-dioxaspiro[4.4]nonane (**3b**), or (*R*,*R*)-(−)-*trans*-2,3-bis(hydroxydiphenylmethyl)-1,4-dioxaspiro[5.4]decane (**3c**)[2] proceeds enantioselectively to give optically active **2**. In particular, the enantioselectivity of irradiation of the *N*-isopropyl derivative **1d** with **3a** and **3b** was very high, yielding the corresponding optically pure product **2d**.



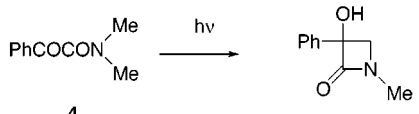
1 **2** **3**

a: R^1=PhCH$_2$, R^2=Ph, R^3=H
b: R^1=PhCH$_2$, R^2=Me, R^3=H
c: R^1=PhCH$_2$, R^2=R^3=H
d: R^1=iPr, R^2,R^3= -(CH$_2$)$_5$-

a: R_2 = Me$_2$
b: R_2 = -(CH$_2$)$_4$-
c: R_2 = -(CH$_2$)$_5$-

Recrystallization of **3a**−**c** and 2-(*N*-benzoyl-*N*-benzylamino)cyclohex-2-enones **1a** from ether or toluene gave 1:1 or 2:1 inclusion crystals. Irradiation for 8 h with stirring of a 2:1 inclusion crystal of **3c** and **1a** in water containing hexadecyltrimethylammonium bromide as a surfactant gave (−)-**2a** in 97% ee in 42% yield and recovered unchanged **1a**, respectively (Table 1). A similar procedure was applied to the other compounds of **1**. Compounds **1a**−**d** formed crystals in the ratios indicated in Table 1. An inclusion compound of **3a** and **1c** was not formed. Chemical and optical yields of the reactions are summarized in Table 1. When a 1:1 inclusion crystal of **1d** with **3a** or **3b** was irradiated, optically pure (−)-**2d** was obtained in 37 or 61% yield, respectively. No reaction occurred for the inclusion compounds of **1c** with **3b** and **3c** or of **1d** with **3c**. It seems that two reaction centers of **1c** or **1d** are located in distant positions in these inclusion compounds. To understand why this is true, we intended to investigate using X-ray analysis. Unfortunately, however, no suitable inclusion crystals of **3a**−**c** and **1a**−**d** could be obtained.

Enantioselective photocyclization of phenylglyoxylamides to β-lactam derivatives has been reported previously, namely, irradiation of *N*,*N*-dimethylphenylglyoxylamide **4** as an inclusion complex with resolved 1,6-bis(*o*-chlorophenyl)-1,6-diphenylhexa-2,4-diyne-1,6-diol (**7**),[3] or with the optically active hosts **3** derived from tartaric acid,[4,5] to produce the optically active 3-hydroxy-1-methyl-3-phenylazetidin-2-one **5**.



4 **5**

Recently, the chiral tetraol host, (−)-*trans*-1,4-bis[3-(*o*-chlorophenyl)-3-hydroxy-3-phenylprop-1-ynyl]-2,3,5,6-tetrachloro-2,5-cyclohexadiene-1,4-diol (**6**), derived from chloranil was prepared. In addition, by complexation with **6**, various kinds of rac-guest compounds were resolved very efficiently.[6] Although the resolution of 2-methylcyclopentanone and 2-methylcyclohexanone cannot be ac-

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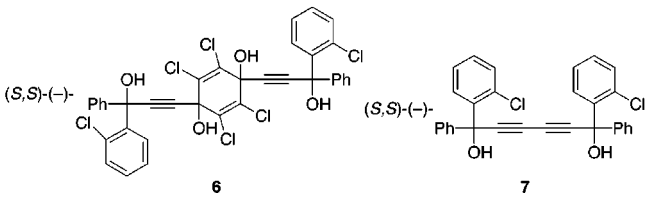
Table 1. Inclusion Compounds of 3 and 1 Prepared by Recrystallization and Their Photoreaction Products 2

		inclusion compound									photoreaction product			
							analysis % found (calcd)							
host	guest	host: guest	yield (%)	mp (°C)	appearance[c]	IR ν_{max} (Nujol, cm^{-1})	C	H	N	irradiation time (h)	cmpd	yield (%)	$[\alpha]_D$ (*c*, MeOH)	optical purity (% ee)
3a	**1a**	1:1	81	112−114	needles	3250, 1680, 1640, 1630	$C_{52}H_{51}NO_6$ 79.44 (79.46)	6.77 (6.54)	1.76 (1.78)	17	(−)-**2a**	18	−17 (0.7)	30[e]
3b	**1a**	1:1	66	126−128	needles	3250, 1680, 1645, 1635	$C_{54}H_{53}NO_6$ 79.95 (79.88)	6.52 (6.58)	1.88 (1.73)	11	(−)-**2a**	18	−27 (0.9)	64[e]
3c	**1a**	2:1	52	[b]	needles	3330, 1690, 1645, 1630	$C_{89}H_{89}NO_{10}$ 80.52 (80.22)	6.89 (6.73)	1.02 (1.05)	8	(−)-**2a**	42	−48 (0.6)	97[e]
3a	**1b**	2:1	85	[b]	needles	3440, 3200, 1675, 1620	$C_{78}H_{79}NO_{10}$ 78.83 (78.69)	6.97 (6.69)	1.12 (1.18)	21	(−)-**2a**	18	−30 (1.5)	68[f]
3b	**1b**	1:1	81	127−129	needles	3280, 1675, 1625	$C_{49}H_{51}NO_6$ 78.64 (78.48)	6.92 (6.86)	1.73 (1.87)	20	(−)-**2b**	23	−33 (1.9)	71[f]
3c	**1b**	2:1	74	155−157	needles	3350, 1695, 1620	$C_{84}H_{87}NO_{10}$ 79.64 (79.41)	6.99 (6.90)	0.86 (1.10)	40	(+)-**2b**	46	+10 (4.4)	33[f]
3a	**1c**	[a]												
3b	**1c**	2:1	81	120−123	prisms	3250, 1685, 1615	$C_{83}H_{85}NO_{10}$ 79.47 (79.34)	6.81 (6.82)	1.23 (1.12)	68	[d]			
3c	**1c**	2:1	90	157−161	prisms	3450, 3330, 1695, 1625	$C_{85}H_{89}NO_{10}$ 79.29 (79.48)	7.17 (6.89)	1.20 (1.09)	68	[d]			
3a	**1d**	1:1	94	166−169	needles	3200, 1680, 1625, 1610	$C_{47}H_{55}NO_6$ 77.73 (77.73)	7.80 (7.56)	1.94 (1.92)	13	(−)-**2d**	37	−29 (1.0)	>99.9[e]
3b	**1d**	1:1	90	125−128	needles	3200, 1680, 1630, 1610	$C_{49}H_{57}NO_6$ 77.73 (77.85)	7.83 (7.60)	2.12 (1.85)	10	(−)-**2d**	61	−27 (6.5)	>99.9[e]
3c	**1d**	2:1	83	[b]	needles	3330, 1690, 1615	$C_{84}H_{93}NO_{10}$ 79.15 (79.03)	7.53 (7.34)	1.10 (1.10)	71	[d]			

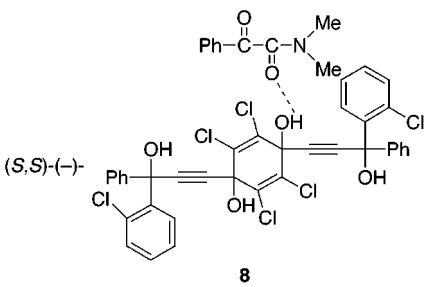
[a] Inclusion compound was not formed. [b] Melting point is not clear. [c] All crystals are colorless. [d] No reaction occurred. [e] Optical purities were determined by HPLC using a column containing the optically active solid phase Chiralcel OD. [f] Optical purities were determined by ^{1}H NMR analysis in $CDCl_3$ using the chiral shift reagent Eu(hfc)$_3$.[7]

complished by complexation with **7**, the method using **6** is applicable to their resolution.[6] Thus, host **6** is superior to **7** in asymmetric recognition of cyclic ketones. In expectation of highly enantioselective photoreactions of **4**, we studied its irradiation with **6**. We found that irradiation of **4** with **6** does indeed proceed enantioselectively to give optically active **5**.



6 **7**

Recrystallization of **6** and **4** from toluene gave a 1:1 inclusion complex **8**.



8

Irradiation for 20 h with stirring of a suspension of this powdered compound in water containing hexadecyltrimethylammonium bromide as a surfactant gave (−)-**5** in 94% ee in 23% yield.

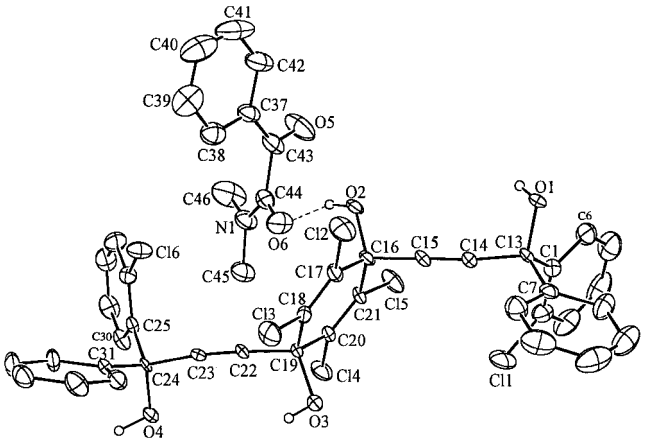


Figure 1. ORTEP drawing and atom labeling of the molecular components in **8**, the O2···O6 hydrogen bond being shown as a broken line. H atoms and numbering of C atoms in the phenyl groups have been omitted for clarity.

To study of the photoreaction mechanism, an X-ray crystal structural analysis of the 1:1 inclusion complex **8** has been performed. The crystal structure of this 1:1 complex of **6** and **4** is shown in Figure 1. Bond lengths and angles are within the range of values found for compounds of this type. The structure consists of hydrogen-bonded (1:1) entities of host to guest with an O2−H···O6 distance of 2.664(6) Å and an O2−H···O6 angle

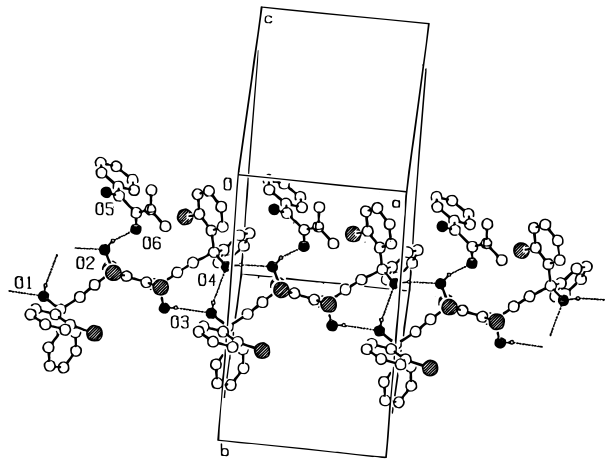


Figure 2. Molecular packing of **8** with a hydrogen-bonding network: O2···O6 = 2.664(6) Å, O1···O4^{i} = 3.171(6) Å, O4···O2^{ii} = 2.914(5) Å, and O3···O1^{ii} = 3.003(5) Å. Symmetry codes: (i) $x + 1$, y, z; (ii) $x - 1$, y, z.

of 162°. The host is conformationally rigid with the two hydroxyl groups at each acetylenic moiety located on the same side of the molecular framework. Because of the bulky chlorine atoms at the ortho position of the phenyl rings, the torsion angles of O1−C13−C1−C2 and O4−C24−C2−C26 are 172.0 and 176.7°, respectively. There are short C_{ortho}−H···O(hydroxyl) contacts at both *o*-chlorophenyl rings [C6−H6···O1 = 2.741(7) Å and C30−H30···O4 = 2.744(7) Å].

The conformation of the guest molecule can be described by the dihedral angle between the best-fit plane through the phenyl ring, including also the adjacent C=O atoms, and the plane through the atoms of the amide group (C44, C45, C46, N1, and O6); the value is 65.1°. The conformation is also greatly influenced by four C−H···O interactions ranging from 2.716(9) to 3.075(9) Å. The short Cl6···C38 (guest) distance of 3.714(8) Å indicates a rather tight packing between guest and host.

Surely the most interesting feature in this crystal structure is the hydrogen-bond network illustrated in Figure 2. The molecules are linked by intermolecular chains of O−H···O along the *a* axis. Each oxygen atom of each hydroxyl group in the host is included in this pattern simultaneously as donor and as acceptor, with the exception of the O3 atom which acts only as a donor. Also forming an integral part of this hydrogen-bonded chain is the guest molecule, which is capable of preorganizing its conformation to occupy a well-defined chiral environment provided by the host molecule. In a figurative way, we can speak about chiral polymeric chains being formed by hydrogen bonding. It is interesting that the benzoyl carbonyl O5 oxygen atom of the guest molecule is not included in the previously described hydrogen-bonding arrangement.

The enantioselectivity of **4** is controlled by the conformation about the O5=C43−C44=O6 bonds. The observed torsion angle is 113.9(8)°, and in the absence of a mirror-symmetry-related molecule, a single enantiomer of **5** is thus formed in the subsequent photoreaction.

The chiral arrangement of the prochiral molecule **4** in the inclusion complex of **6** and **4** also was clarified by the measurement of CD spectra in the solid state.[8,9] The chiral host molecule (−)-**6** showed neither strong absorption nor CD peaks in the 400−200 nm region. In contrast, however, the inclusion compound of prochiral (−)-**6** and **4** exhibited a rather strong CD spectrum, suggesting that this spectrum originates primarily from **4** (Figure 3). Thus, the prochiral compound exhibits chirality when frozen in a chiral conformation in the solid state. This result is expected on the basis of our X-ray crystal structural analysis.

The mixing of powdered **6** and **4** gave the inclusion complex in a quantitative yield. Irradiation for 20 h with stirring of a suspension of this powdered 1:1 complex of **6** and **4** in water containing hexadecylmethylammonium bromide as a surfactant gave (−)-**5** in 87% ee in 15% yield.

Other phenylglyoxylamides, for example, diethylphenylglyoxylamide, did not form inclusion complexes with **6** by either recrystallization or mixing methods.

In conclusion, we have successfully performed highly enantioselective photocyclization of amides (**1**, **4**) to *β*-lactams (**2**, **5**) using host−guest interaction in chiral crystals.

Experimental Section

General Methods. The preparation of inclusion compounds of **1** and **4** with the hosts was achieved by recrystallization from toluene or ether. The host:guest ratio of each inclusion compound was determined by elemental analysis. Irradiation of the inclusion compounds as water suspensions at room temperature was performed through a Pyrex filter using a 100-W high-pressure Hg lamp. IR spectra were measured with a JASCO FT/IR-350 IR spectrometer, using Nujol mulls. ^{1}H NMR spectra were recorded in $CDCl_3$ on a JEOL JNM-LA300 (300 MHz) spectrometer. $[\alpha]_D$ values were measured with a JASCO DIP-1000S digital polarimeter. Optical purities were determined by HPLC using a hexane/2-propanol (9:1) solvent (flow rate = 1.0 mL/min), unless otherwise stated, and a column (0.46 cm × 25 cm) containing the chiral solid phase Chiralcel OC or OD, which is commercially available from Daicel Chemical Industries, Ltd., Himeji, Japan.

Preparation of 1. The compounds **1a**−**d** were prepared by the reported method.[1]

Preparation of Inclusion Compounds of 3 and 1. When a solution of **3c** (3.8 g, 7.6 mmol) and **1a** (1.2 g, 3.8 mmol) in toluene (20 mL) was kept at room temperature for 12 h, a 2:1 inclusion compound of **3c** and **1a** was obtained as colorless needles (2.6 g, 52% yield, mp is not clear). IR (Nujol) ν_{max}: 3330, 1690, 1645, 1630 cm^{-1}. Anal. Calcd for $C_{89}H_{89}NO_{10}$: C, 80.22; H, 6.73; N, 1.05. Found: C, 80.52; H, 6.89; N, 1.02. By the same procedure, the inclusion compounds of **3** and **1** were prepared (Table 1).

Photocyclization of 1 to 2. A suspension of a powdered 1:1 inclusion compound of **3c** with **1a** (2.6 g, 3.2 mmol) in water (120 mL) containing hexadecyltrimethylammonium bromide (0.04 g) as a surfactant was irradiated with stirring for 8 h. The reaction product was filtered, dried, and chromatographed on silica gel using AcOEt/hexane (1:4) as the eluent to give (−)-**2a** in 97% ee as a colorless oil (0.27 g, 26% yield). $[\alpha]_D$ −48 (*c* 0.6, MeOH). By the same procedure, inclusion compounds of **3** and **1** gave optically active **2**, in the yields and optical purities shown in Table 1. Products **2a**−**d**[1] are known compounds.

Preparation of (−)-*trans*-1,4-Bis[3-(*o*-chlorophenyl)-3-hydroxy-3-phenylprop-1-ynyl]-2,3,5,6-tetrachloro-2,5-cy-

(7) $Eu(hfc)_3$ is available from Aldrich Chemical Company, Inc., Milwaukee, WI. The methylene proton signal of the benzyl group of **2b** and its (−)- and (+)-enantiomers appeared at δ 4.18 and 4.78, respectively, in $CDCl_3$ in the presence of 0.1 molar equiv of $Eu(hfc)_3$.

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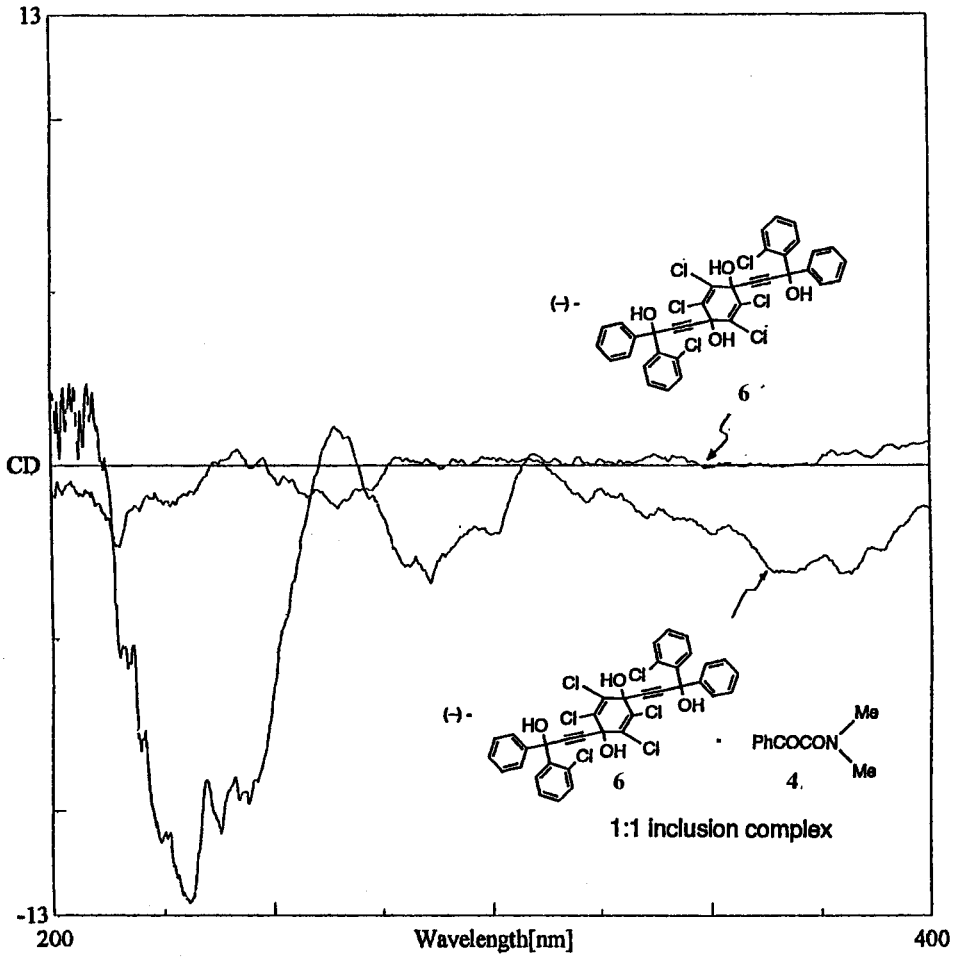

Figure 3. CD spectra of **6** and the 1:1 inclusion complex of **6** and **4** as a Nujol mull.

clohexadiene-1,4-diol (6). Compound **6** was prepared by a literature procedure.[6]

Preparation of *N,N*-Dimethylphenylglyoxylamide 4. Compound **4** was prepared by a literature procedure.[10]

Preparation of Inclusion Compounds of 6 and 4 by the Recrystallization Method. When a solution of **6** (4.13 g, 5.66 mmol) and **4** (1.00 g, 5.66 mmol) in toluene (50 mL) was kept at room temperature for 2 h, their 1:1 inclusion compound was obtained as colorless needles (3.6 g, 70% yield, mp 166−168 °C). IR (Nujol) ν_{max}: 1679, 1624 cm^{-1}. Anal. Calcd for $C_{46}H_{33}NO_6Cl_6$: C, 60.82; H, 3.66; N, 1.54. Found: C, 61.14; H, 3.74; N, 1.55.

Irradiation of *N,N*-Dimethylphenylglyoxylamide (4) to 3-Hydroxy-1-methyl-3-phenylazetidin-2-one (5). A suspension of the powdered inclusion complex of **6** and **4** (3.6 g, 4.0 mmol) in water (100 mL) containing hexadecyltrimethylammonium bromide (0.04 g) as a surfactant was irradiated with stirring for 20 h with a 100-W high-pressure Hg lamp. The reaction mixture was filtered, dried, and chromatographed on a silica gel, using AcOEt/hexane (1:9) as the eluent to give (*S*)-(−)-3-hydroxy-1-methyl-3-phenylazetidin-2-one (**5**)[3] in 94% ee as colorless crystals (0.16 g, 23% yield). $[\alpha]_D$ −34 (*c* 0.1, MeOH).

Crystal Structure Determination of the 1:1 Inclusion Complex (8) between 6 and 4 Prepared by the Recrystallization Method. Crystal Data. $C_{36}H_{22}Cl_6O_4 \cdot C_{10}H_{11}NO_2$; M = 908.43; orthorhombic, space group, $P2_12_12_1$; a = 10.666(1) Å, b = 19.037(2) Å, c = 21.509(1) Å; V = 4367.4(6) Å^3 (by least-squares refinement on diffractometer angles for 20 automatically centered reflections, λ = 1.54178 Å); Z = 4; $F(000)$ = 1864; D_x = 1.382 g cm^{-3}; μ (Cu Kα) = 3.993 mm^{-1}. Colorless needles; crystal size, 0.6 × 0.3 × 0.2 mm.

Data Collection and Processing. Intensity data were measured at room temperature on a Siemens P4 diffractometer with a graphite monochromator and in the ω scan mode. Corrections for absorption effects were made with the empirical program DIFABS.[11] The possibility of decay of the analyzed crystal was tested by the intensities of standard reflections and was found to be negligible. A total of 3935 unique reflections were measured up to $2\theta_{max}$ = 128.60° using Cu Kα radiation with R_{int} = 0.0560.

Structure Analysis and Refinement. The structure was solved by direct methods (SIR92)[12] and refined by a full-matrix least-squares approach based on F^2 using SHELXL-96.[13] The O−H hydrogen atoms were found from a difference Fourier synthesis and refined. The remaining C−H hydrogen atoms were introduced in calculated positions and allowed to ride on their parent atoms. The absolute chirality was determined using the Flack parameter [−0.01(2)].[14] The final values of the *R* factors were R1 = 0.0445 for 3373 observed reflections with $I > 2\sigma(I)$ and wR2 = 0.1083 for all data, 553 parameters. At convergence, the peaks and troughs of the difference density map did not exceed 0.267 and −0.248 e Å^{-3}, respectively. The ORTEP diagram was prepared using ORTEP-III,[15] as implemented in ORTEP-3 for Windows.[16] The hydrogen-bonding scheme was prepared using PLUTON-92.[17]

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Preparation of the Inclusion Compound of 6 and 4 by the Mixing Method. When powdered **6** (2.74 g, 3.74 mmol) and **4** (0.66 g, 3.74 mmol) were mixed for 30 min using an agate mortar and pestle, the mixture solidified to give a 1:1 inclusion complex of **6** and **4** as a colorless powder in a quantitative yield (3.4 g, mp 162−164 °C). IR (Nujol) ν_{max}: 1679, 1624 cm^{-1}.

Irradiation of the Inclusion Compound of 6 and 4 Prepared by the Mixing Method. A suspension of the powdered inclusion complex of **6** and **4** (3.4 g, 3.74 mmol) in water (100 mL) containing hexadecyltrimethylammonium bromide (0.04 g) as a surfactant was irradiated with stirring for 20 h with a 100-W high-pressure Hg lamp. The reaction mixture was filtered, dried, and chromatographed on a silica gel, using AcOEt/hexane (1:9) as the eluent to give (*S*)-(−)-3-hydroxy-1-methyl-3-phenylazetidin-2-one (**5**)[3] in 87% ee as colorless crystals (0.10 g, 15% yield). $[\alpha]_D$ −20 (*c* 0.1, MeOH).

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Supporting Information Available: Tables with crystallographic data and structure refinement, listing of atomic coordinates, anisotropic thermal parameters, bond lengths and angles, hydrogen coordinates, dihedral angles between mean planes. This material is available free of charge via the Internet at http://pubs.acs.org.

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