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Inclusion of Poly(ethylene glycol)s by Crystalline (*R*)-(1-Naphthyl)glycyl-(*R*)-phenylglycine

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Crystallization of (*R*)-(1-naphthyl)glycyl-(*R*)-phenylglycine [(*R,R*)-1] in the presence of oligo(ethylene glycol) dimethyl ethers 2(*n*) or poly(ethylene glycol)s (PEGs, 3(M_n)) afforded inclusion compounds. The ratio of (*R,R*)-1/the guest polymer (2 or 3) was proportional to the length of the polymer chain. The crystal structure of a hepta(ethylene glycol) dimethyl ether-included compound was disclosed by X-ray crystallography which showed that (*R,R*)-1 molecules form a sheet and the guest molecule penetrates the crystal lattice of (*R,R*)-1 through a one-dimensional channel on the sheet. Powder X-ray analysis revealed that, regardless of the length of the guest polymer, the distance between the neighboring sheets remains unchanged (12.0–12.3 Å) in these inclusion crystals. By thermal analysis, it was shown that the decomposition points of these inclusion compounds became higher with the longer PEG included. The inclusion phenomenon enabled the fractionation of PEGs with various molecular weights, among which longer PEG was preferably included.

Keywords: Molecular recognition; Polymer inclusion; Fractionation; Dipeptides; Thermal analysis; Molecular distribution

INTRODUCTION

In inclusion compounds, host molecules self-assemble to form zero- (cavity), one- (channel or canal), or two-dimensional (layered lattice) voids and guest molecules are accommodated therein. When the host molecules construct a one-dimensional channel, straight-chain polymer molecules are anticipated to be favorably included in their crystal lattice [1]. Urea and thiourea are known to be good host molecules which construct inclusion crystals with various polymers [1, 2]. The molecules assemble through hydrogen bonding to form helical ribbons, which are woven into an array of linear, hexagonal one-dimensional channels that can contain the guest polymers. Thus, the crystalline ureas included various straight-chained polymers. Farina *et al.* [3a] and Tonelli [3b] reported that perhydrotriphenylene (PHTP) molecules

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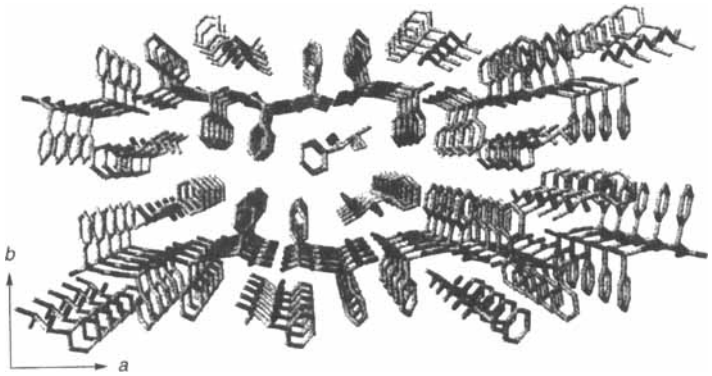
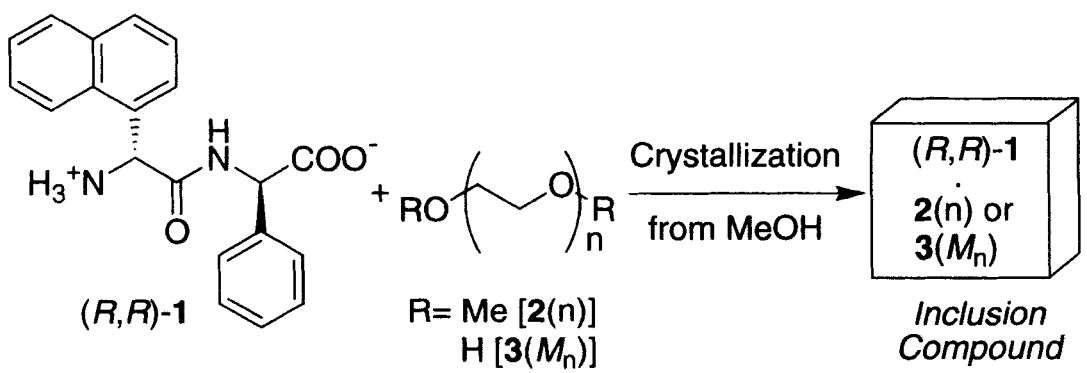


FIGURE 1 Perspective view of the inclusion compound of (*R*)-phenylglycyl-(*R*)-phenylglycine and (*S*)-isopropyl phenyl sulfoxide (the *a* – *b* plane).



SCHEME 1

stack to make the hexagonal 1D-channel, in which linear alkanes and alkanediones are included.

We have already reported a strategy for constructing one-dimensional channels by the use of simple dipeptides such as (*R*)-phenylglycyl-(*R*)-phenylglycine [4a – c] and (*R*)-(1-naphthyl)glycyl-(*R*)-phenylglycine [4d, e]. When the guest molecules are sulfoxides or ethers, the dipeptide molecules are arranged to form parallel β-sheet-like structures which stack to form a one-dimensional channel (Fig. 1).

The construction of the channel structure from the dipeptide molecules prompted us to examine the possibility that it might include a polymer. Here, we report the inclusion of an oligo(ethylene glycol) dimethyl ether and poly(ethylene glycol) (PEG) in the crystal lattice of (*R*)-(1-naphthyl)glycyl-(*R*)-phenylglycine [(*R*,*R*)-**1**].

RESULTS AND DISCUSSION

A Crystal Structure of the Inclusion Compound of 1,2-Dimethoxyethane as the Monomer Unit

First, we tried to form an inclusion compound from (*R*,*R*)-**1** and 1,2-dimethoxyethane [DME, **2**(1)] because **2**(1) is a fundamental unit of oligo(ethylene glycol) dimethyl ethers and poly(ethylene glycol)s. The inclusion compound of (*R*,*R*)-**1** and **2**(1) [(*R*,*R*)-**1** : **2**(1) = 1 : 1] could be

obtained by cocrystallization from methanol. Single-crystal X-ray analysis showed that the inclusion compound has a layer structure and the molecules of **2**(1) are placed between the layers (Fig. 2). The layer was formed by parallel β-sheet-like arrangement of (*R,R*)-**1** molecules *via* the salt formation between the amino group and the carboxyl group of (*R,R*)-**1** (Fig. 2b). One terminal COO^- group is a bridge between two $^+NH_3$ of the adjacent molecules and, on the contrary, the $^+NH_3$ linked two COO^- groups of the adjacent molecules (O···N distances; 2.68 and 2.87 Å, respectively). The layer distance is 12.1 Å, which corresponds to the strongest peak ($2\theta = 7.28°$) in its powder X-ray diffraction (PXRD) pattern.

The naphthyl and phenyl groups of (*R,R*)-**1** are situated perpendicular to the sheet, where neighboring naphthyl and phenyl groups stacked alternately in an edge-to-face manner to form the walls of the one-dimensional channel (Fig. 3a). DME molecules interact with the $^+NH_3$ of the layer *via* three-centered hydrogen bonding (O···N distances; 2.96, 3.17 Å) (Fig. 3b) in a manner similar to that of *o*-dimethoxybenzene molecules included in (*R,R*)-**1** crystals (O···N distances; 2.99, 3.09 Å) (Fig. 3c) [4d]. The conformation of the O(1)–C(2)–C(3)–O(2) unit is *gauche* (58.9°). The C(1)–O(1)–C(2)–C(3) and C(2)–C(3)–O(2)–C(4) adopt *trans* conformations (their dihedral angles: 166.2° and 170.3°, respectively).

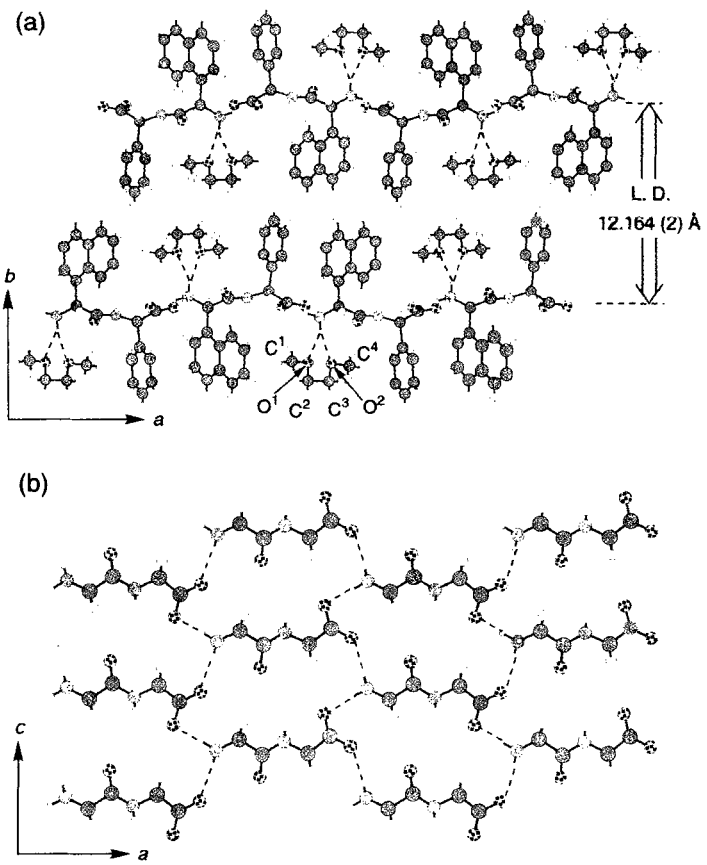


FIGURE 2 (a) Layer structure of the inclusion compound of (*R,R*)-**1**·**2**(1) (the *a*–*b* plane). Dotted lines mean hydrogen bonds between $^+NH_3$ of (*R,R*)-**1** and oxygen atoms of DME. (b) Sheet structure of dipeptide backbones of the inclusion compound (*R,R*)-**1**·**2**(1) (the *a*–*c* plane). The naphthyl and phenyl groups of (*R,R*)-**1** and **2**(1) are omitted for clarity.

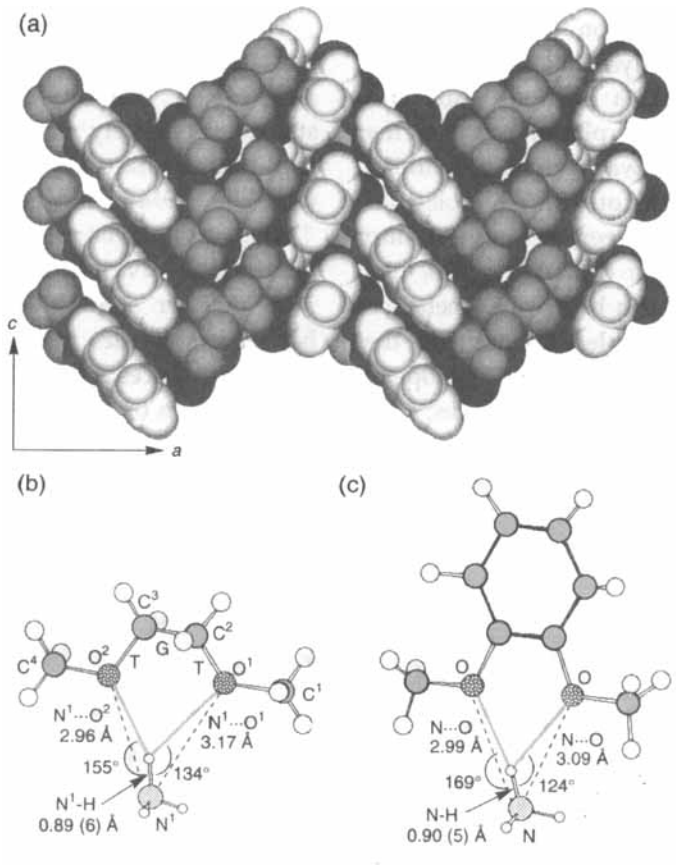


FIGURE 3 (a) CPK model of packing in the inclusion compound of (*R,R*)-**1** and **2**(1) (the *a* – *c* plane). The naphthyl and phenyl groups of (*R,R*)-**1** and **2**(1) are colored white and gray, respectively. (b) Hydrogen-bonding networks between $^{+}NH_3$ of (*R,R*)-**1** and **2**(1). (c) Hydrogen-bonding networks between $^{+}NH_3$ of (*R,R*)-**1** and 1,2-dimethoxybenzene.

Figure 3a shows that **2**(1) molecules are regularly arranged in the one-dimensional channel. Formally, the arranged molecules of **2**(1) can be regarded as an oligo(ethylene glycol) dimethyl ether by intermolecular linkage of their terminal methyl groups. This consideration led us to propose that (*R,R*)-**1** would form the inclusion crystals of an oligo(ethylene glycol) dimethyl ether [$CH_3O-(CH_2CH_2O)_n-CH_3$, **2**(*n*)].

Inclusion of Oligo(ethylene glycol) Dimethyl Ethers in (*R,R*)-1 Crystals

We investigated whether (*R,R*)-**1** molecules form an inclusion compound of **2**(*n*). As illustrated in Figure 4, the calculated molar ratios can be estimated according to an equation $(n+1)/2$, in which n is the number of the ethyleneoxy unit.

Ten kinds of oligo(ethylene glycol) dimethyl ethers **2**($n = 1-7$, 9, 12, and 20) were selected as the guest molecule. Fortunately, the inclusion compound was obtained from (*R,R*)-**1** and all of the selected **2**(*n*) by cocrystallization from methanol. The deposited crystals were collected by filtration and washed with chloroform. The host–guest molar ratios [(*R,R*)-**1**/**2**(*n*)] were determined by ^1H NMR and elemental analysis. As summarized in Table I, **2**(*n*) molecules were efficiently included in the crystal lattice of (*R,R*)-**1**. Actually, the observed ratios were proportional to the length of the polymer chain and comparable to the calculated ratios (Fig. 5).

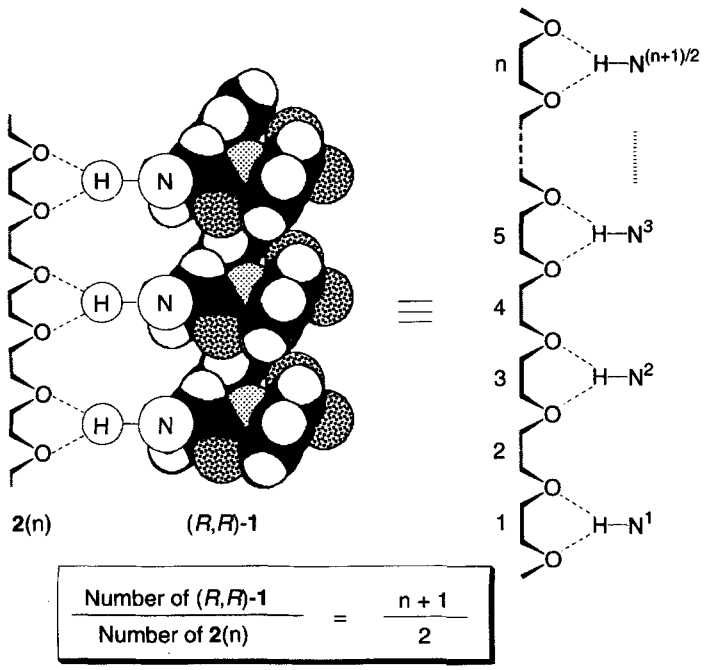


FIGURE 4 Calculation method for host–guest molar ratio between (*R,R*)-**1** and **2**(*n*).

TABLE I Polymer inclusion of poly(ethylene)glycol dimethyl ethers 2(*n*) in cocrystallization with (*R,R*)-**1**

Guest	Calcd H/G[a]	Obs H/G[b]	L.D. (Å)[c]
2(1)	1.00	1.00	12.1
2(2)	1.50	1.56	12.2
2(3)	2.00	1.92	12.2
2(4)	2.50	2.50	12.2
2(5)	3.00	3.10	12.0
2(6)	3.50	3.15	12.3
2(7)	4.00	3.80	12.1
2(9)	5.00	5.00	12.3
2(12)	6.50	6.40	12.1
2(20)	10.50	10.50	12.1

[a]The calculated values of the host/guest molar ratio, see text.
[b]The host/guest determined by ^{1}H NMR.
[c]The layer distance measured by PXRD.

In their PXRD patterns, all of these inclusion compounds showed a strong diffraction peak at a lower 2θ angle. Irrespective of the length of the included oligomers **2**(*n*), the strong peaks appear in the range 12.0–12.3 Å. Noteworthily, these values are the same as the layer distance of the inclusion compound of (*R,R*)-**1** and DME. Therefore, oligo(ethylene glycol) dimethyl ethers are suggested to be accommodated in the channel constructed by (*R,R*)-**1** molecules. When hepta(ethylene glycol) dimethyl ether **2**(7) was used as the guest molecule, we obtained a single crystal suitable for X-ray crystallographic analysis. Satisfactory measurement could be performed at 173 K. By regarding a series of the guest molecules **2**(7) in the channel as the repeating of a —C—C—O—C—C—O— moiety, their conformation could be resolved and refined isotropically.

The (*R,R*)-**1** molecules are arranged in the same manner as that of **2**(1) to form a layer structure (Fig. 6). The hydrogen-bonding distances (O···N) between COO^- and $^+NH_3$ were 2.63 and 2.80 Å, respectively. Figure 7 shows the top view of the space filling model from the *a*–*c* plane of the inclusion compound of (*R,R*)-**1** and **2**(7). A herringbone motif of naphthyl and phenyl groups constructs the wall of the one-dimensional channel and the **2**(7) molecules are

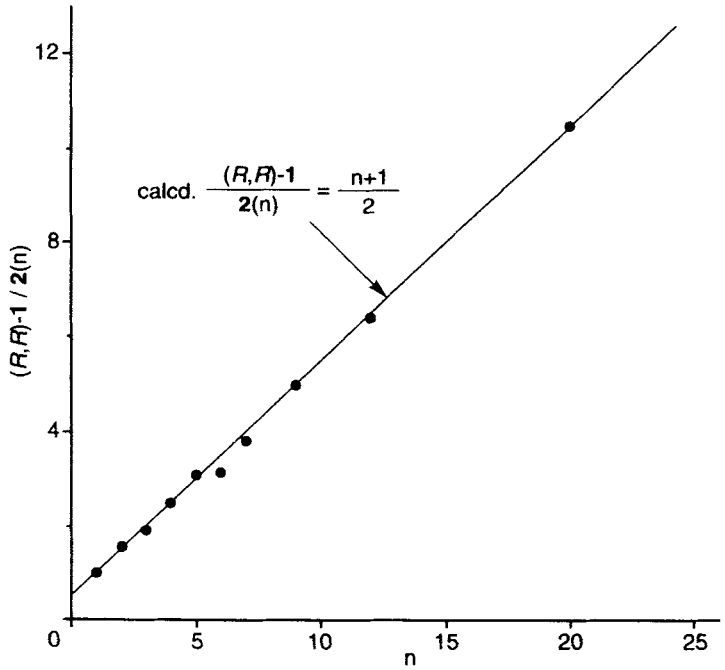


FIGURE 5 Relationship between host–guest ratio and the repetition number of monomer units (ethyleneoxy groups) of **2**(*n*).

accommodated in the channel *via* hydrogen bonding (O···N distance; 2.98 Å).

Figure 8a shows the conformation of **2**(7) to be $G^+TG^-G^+TG^-$, where T, G^+, and G^- denote *trans*, right-handed *gauche*, and left-handed *gauche* conformations, respectively. The repeating unit of **2**(7) has 5.610(2) Å, which corresponds to the *c* axis of the unit cell. A similar conformation of a PEG chain was observed in a PEG–$HgCl_2$ complex (Form II) in which the repeating unit is 5.88 Å long (Fig. 8b) [5]. In a similar manner that Hg^{2+} captures the oxygens of PEG as a Lewis acid, the ammonio proton of (*R,R*)-**1** acts as a Brønsted acid to capture the oxygens of **2**(7). Thus, the included PEGs adopt a conformation which meets not only the spatial restriction of the inclusion channel, but also the requirement of the above-mentioned hydrogen bonding. It should be noted that the ethyleneoxy unit seems to be essential for the inclusion in the one-dimensional channel because a tris(trimethylene glycol) dimethyl ether [$CH_3O(CH_2CH_2CH_2O)_3CH_3$] was not included at all.

Inclusion of Poly(ethylene glycol)s in (*R,R*)-1 Crystals

Previously, we reported that (*R,R*)-**1** formed an inclusion compound with such alcoholic guests as allyl alcohols and α-hydroxy esters. In these cases, the one-dimensional channel was not constructed, but a zero-dimensional pocket-type cavity was formed [6]. Therefore, it is doubtful whether poly(ethylene glycol)s are included in the crystal lattice of (*R,R*)-**1**. Indeed, tri(ethylene glycol), tetra(ethylene glycol), and tri(ethylene glycol) monomethyl ether did not afford the corresponding inclusion crystals. A thought occurred to us: If the poly(ethylene glycol)s **3**(M_n) are fairly long, their terminal hydroxyl groups might have little influence over the present inclusion. Hence, we tried to form an inclusion compound from PEG with high molecular weight. As summarized in Table II, PEGs of molecular weight 400–20,000 succeeded in affording the corresponding inclusion compounds. The ratios of (*R,R*)-**1**:PEG are in good

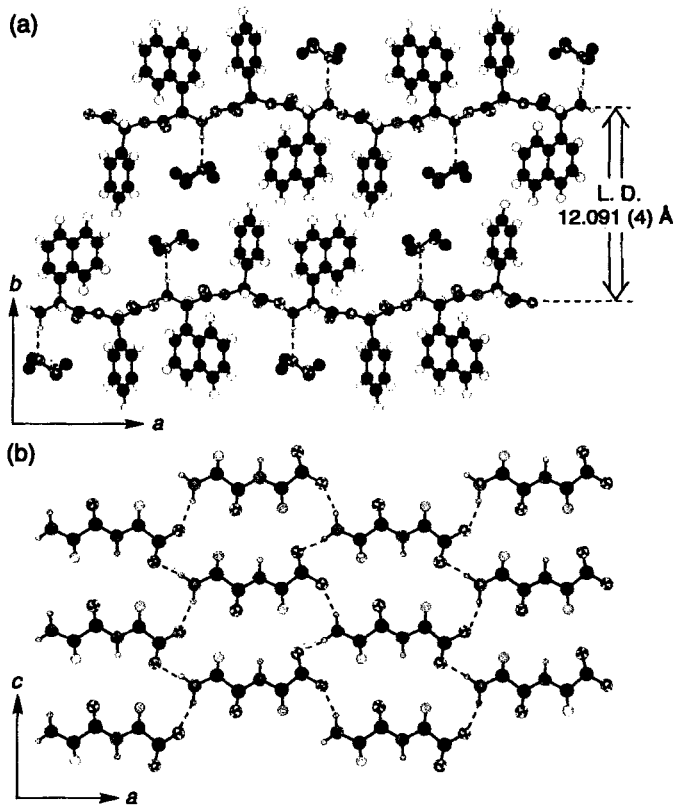


FIGURE 6 (a) Layer structure of the inclusion compound of (*R,R*)-**1**·**2**(7) (the $a-b$ plane). Dotted lines mean hydrogen bonds between $^{+}NH_3$ of (*R,R*)-**1** and oxygen atoms of **2**(7). (b) Sheet structure of dipeptide backbones of the inclusion compound (*R,R*)-**1**·**2**(7) (the $a-c$ plane). The naphthyl and phenyl groups of (*R,R*)-**1** and **2**(7) are omitted for clarity.

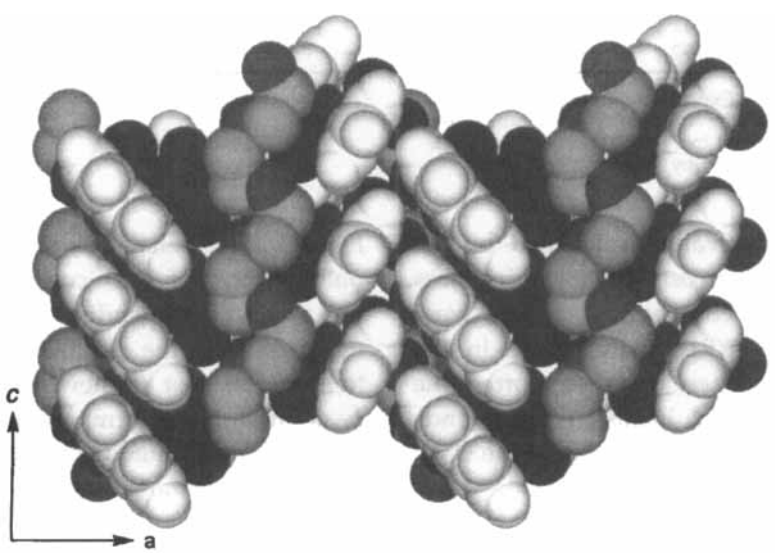


FIGURE 7 CPK model of packing in the inclusion compound of (*R,R*)-**1** and **2**(7) (the $a-c$ plane). The naphthyl and phenyl groups of (*R,R*)-**1** and **2**(7) are colored white and gray, respectively. Hydrogen atoms of **2**(7) are not shown because the guest chains are refined isotropically.

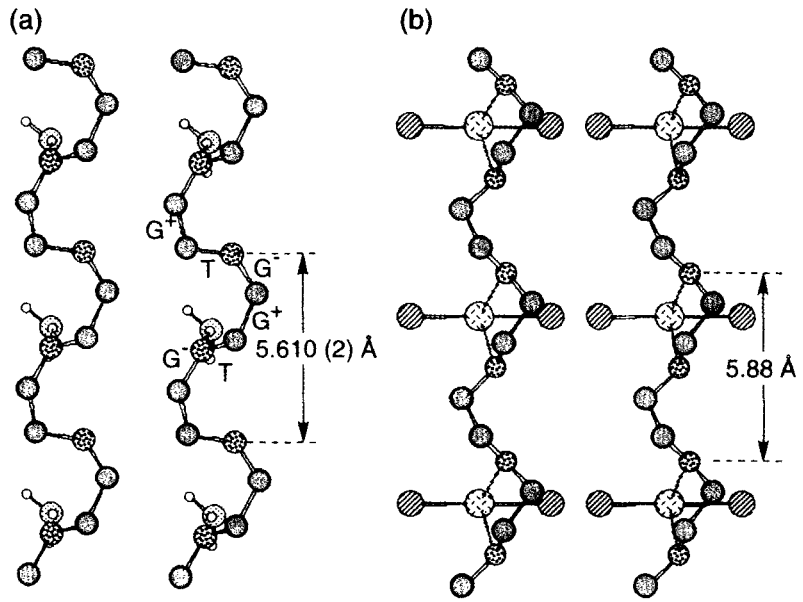


FIGURE 8 (a) Stereopair showing the conformation of 2(7) in the (*R,R*)-**1**·2(7) and the association of 2(7) and $^{+}NH_3$ of (*R,R*)-**1** in the channel. Dotted lines mean hydrogen-bonding. (b) Stereopair showing the conformation of PEG in the complex of PEG–$HgCl_2$ (type II). Dotted lines mean coordination bonding.

TABLE II Polymer inclusion of poly(ethylene)glycols 3(M_n) in cocrystallization with (*R,R*)-**1**

Guest	Obs. M_n[a]	Average *n*	Calcd H/G[b]	Obs H/G[c]	L.D. (Å)[d]
3(400)	397	8.60	4.80	4.80	12.0
3(600)	524	11.5	6.25	5.88	12.0
3(1,000)	842	18.7	9.85	9.85	12.1
3(4,000)	2878	64.9	33.0	31.0	12.0
3(6,000)	8517	193	97.0	90.2	12.1
3(20,000)	16772	380	191	212	12.1

[a] Determined by GPC, see Experimental Section.
[b] The calculated values of the host/guest molar ratio, see text.
[c] The host/guest determined by ^1H NMR.
[d] The layer distance measured by PXRD.

accordance with the values calculated by the equation $(n+1)/2$ (*vide post*). The layer distances of these inclusion compounds, which were assigned by their PXRD analysis, are in the range 12.0–12.1 Å. These facts supported the motion that the present inclusion compounds have a layer structure similar to that of the DME inclusion compound.

Thermal analysis was performed for the inclusion compounds of 3(M_n). In the DTA-curves of the inclusion compounds of 3(M_n), two endothermic peaks were observed as shown in Figure 9. This behavior is similar to that of urea-poly(tetrahydrofuran) complexes which show two peaks corresponding to the dissociation of the guest from the channel and the decomposition of the host framework.[2f,g] Hence, one peak at a lower temperature in the present inclusion compounds could be assigned to the vibrational-rotational energy of the guest to escape from the channel, and the other peak was regarded as corresponding to the decomposition of the host crystal lattice which was brought about by intramolecular dehydration of

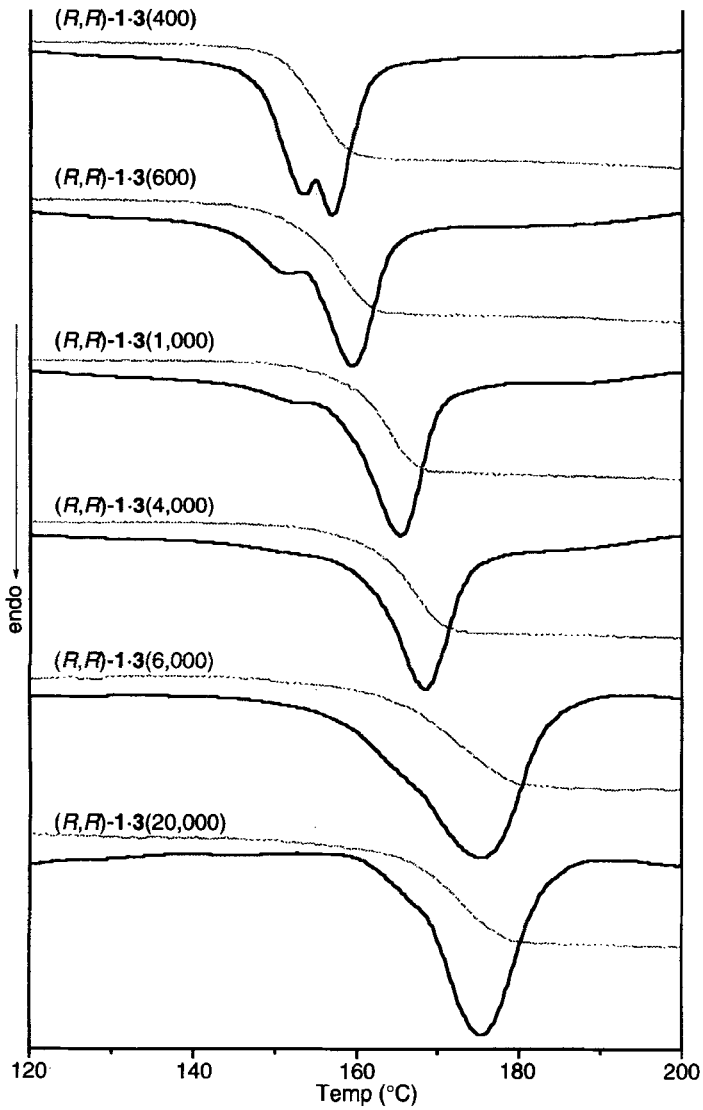


FIGURE 9 TG–DTA curves of the inclusion compounds of (*R,R*)-**1**·**3**(400, 600, 1,000, 4,000, 6,000, and 20,000). TG and DTA curves are colored gray and black, respectively.

(*R,R*)-**1** to form the corresponding diketopiperazine (3.9–4.1% weight loss in TG). The final formation of the diketopiperazine was confirmed by ^{1}H NMR and IR spectra.

The distinct features of the present PEG inclusion compounds are as follows: (i) the decomposition point becomes higher with the increasing molecular lengths of the guest **3**(M_n). This is probably because the mobility of (*R,R*)-**1** in the inclusion compounds is more retarded as **3**(M_n) becomes longer; (ii) The decomposition of the host lattice occurs immediately after the guest molecules escape from the crystal lattice. This means that the penetrating **3**(M_n) acts not only as a filler of the channel, but also as a supporting rope to adhere to (*R,R*)-**1** molecules and, as a result, makes the sheet structure more tight.

Fractionation of PEGs by (*R,R*)-1 Crystals

We were also interested in the possibility that a mixture of PEGs with various molecular weights might be fractionated by inclusion into (*R,R*)-**1** crystals. As a preliminary experiment, we investigated the fractionation of **2**(4) and **2**(12). After a mixture (82.0 mg) of **2**(4) and **2**(12) (49:51, wt/wt) was dissolved in methanol (1.5 mL) containing (*R,R*)-**1** (0.40 mmol), the resulting solution was allowed to stand at room temperature. After about one week, the deposited crystals were collected by filtration and washed with chloroform (10 mL). The filtrate and the washing were combined and evaporated to give a mixture (61.4 mg) of **2**(4) and **2**(12) which were not included in the crystalline (*R,R*)-**1**. The collected crystals were dissolved in 1 M hydrochloric acid and the released **2**(4) and **2**(12) (20.2 mg) were extracted with chloroform. The ratio of **2**(4):**2**(12) was determined by GPC. Interestingly, a large difference was observed between the ratios of the included mixture [**2**(4):**2**(12) = 14:86] and the not-included one [**2**(4):**2**(12) = 68:32]. In a similar manner, PEGs with a broad distribution of molecular weight were fractionated. The PEG mixtures were

TABLE III Comparison of the polymolecularity index[a] of PEG before and after inclusion compound formation

	Commercial PEG	After the inclusion: Include PEG	After the inclusion: Not-included PEG
Sample A			
M_n	1950	2628	1718
M_w	12758	14251	11633
M_w/M_n	6.54	5.42	6.77
Sample B			
M_n	525	801	453
M_w	746	1021	639
M_w/M_n	1.42	1.27	1.41

[a] Determined by GPC, see Experimental Section.

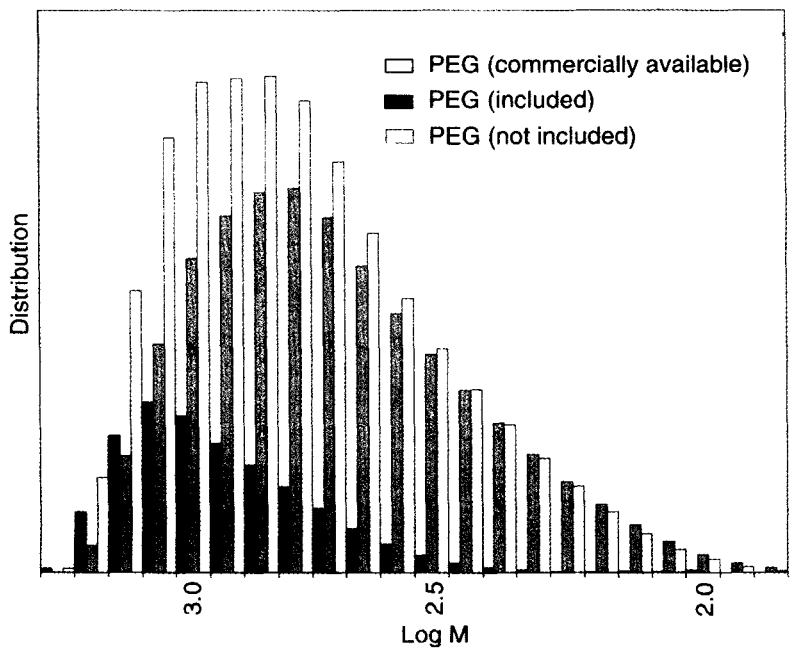


FIGURE 10 Molecular weight distribution of standard (white), included (black), and not included (gray) **3** (sample B) which obtained by crystallization of (*R,R*)-**1**·**3** (sample B).

prepared by mixing commercially available PEGs (from Nacalai Tesque, Inc.): Sample A is a 1:1 mixture of commercial PEG #1,000 and PEG #20,000. Sample B is a 1:1:1 mixture of PEG #600, PEG #1,000, and PEG #1,500. In the case of Sample A, the ratio of the included PEGs #1,000 and #20,000 was 35:65, while the not-included one was 56:44. In Table III, M_n, M_w, and M_w/M_n are shown. The favorable inclusion of the PEG with a higher molecular weight was also observed in the inclusion of Sample B, as summarized in Figure 10 and Table III.

Since analogous phenomena were reported in the formation of urea-poly(tetrahydrofuran) inclusion compounds [2f, g], it seems to be generally conclusive that, in the crystallization process, the inclusion of the polymer with higher molecular weight is more favorable.

This is reasonably explained in terms of entropic advantage in the inclusion: inversely speaking, the inclusion of the polymer with higher molecular weight is smaller in entropy loss.

CONCLUSION

(*R*)-(1-Naphthyl)glycyl-(*R*)-phenylglycine [(*R*, *R*)-**1**] formed an inclusion compound with such polymers as oligo(ethylene glycol) dimethyl ethers or poly(ethylene glycol)s. The polymer molecules are accommodated *via* hydrogen bonding in the one-dimensional channel surrounded by naphthyl and phenyl groups which are perpendicularly situated on the sheet of the glycylglycine backbones. As a result, the polymer adheres to (*R*,*R*)-**1** molecules of the crystal lattice to retard the mobility of (*R*,*R*)-**1** molecules. By the use of the inclusion by (*R*,*R*)-**1** crystals, a mixture of **2**(*n*) or **3**(M_n) with broad molecular weight distribution was fractionated to improve the polydispersity index (M_w/M_n) to some extent.

EXPERIMENTAL SECTION

General Procedure

The melting points were uncorrected. Elemental analyses were performed at the Chemical Analysis Center, Chiba University, Japan. (*R*)-(1-Naphthyl)glycyl-(*R*)-phenylglycine was prepared according to our previous literature [4d]. Molecular distribution was determined by Shodex GPC OHPak SB-802HQ or SB-804HQ, using H_2O as an eluent (0.5 mL min^{-1}). The calibration curve required for the analysis of the distribution of the molecular masses was obtained from the commercial PEG standards.

Synthesis of Penta(ethylene glycol) Dimethyl Ether [2(5)]

To a suspension of NaH (60%, 0.629 g, 15.7 mmol) in dry DMF (12 mL), ethylene glycol monomethyl ether (651 mg, 8.56 mmol) was added dropwise at 0°C. Then, the resulting suspension was stirred at 0°C for 1 h, and tri(ethylene glycol) ditosylate [7] (1.83 g, 4.0 mmol) was added at 0°C. The reaction mixture was further stirred at room temperature for 20 h. Then, the reaction was quenched with water (5 mL) at 0°C, the mixture was extracted with $CHCl_3$ (30 mL × 3), the extract was dried with $MgSO_4$ and concentrated in *vacuo*. The oily residue was chromatographed upon SiO_2 employing ethyl acetate as an eluent. The final purification was performed by distillation [Kugelrohr, 180°C (3.2 mmHg), lit. [7, 8] 100–108°C (0.27 mmHg) and 120–123°C (0.25 mmHg), respectively] to afford **2**(5) (698 mg, 2.62 mmol, 65%) as a colorless oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 3.68–3.62 (*m*, 16H, CH_2), 3.60–3.54 (*m*, 4H, $CH_3OCH_2CH_2$), 3.38 (*s*, 6H, CH_3O); IR (KBr) 3568, 2874, 1945, 1458, 1350, 1299, 1249, 1200, 1109, 947, 852 cm^{-1}. Anal. Calcd for $C_{14}H_{30}O_7$: C, 54.12; H, 9.84. Found: C, 53.95; H, 10.08.

Synthesis of Hexa(ethylene glycol) Dimethyl Ether [2(6)]

According to the procedure described above, **2**(6) was prepared from ethylene glycol monomethyl ether and tetra(ethylene glycol) ditosylate [7]. Distillation [Kugelrohr, 195°C (1.3 mmHg), lit. [8] 145–148°C (0.1 mmHg)] of the product gave **2**(6) (91% yield) as a colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.67–3.62 (*m*, 20H, CH_2), 3.58–3.52 (*m*, 4H, $CH_3OCH_2CH_2$), 3.38 (*s*, 6H, CH_3O); IR (KBr) 3517, 2873, 1962, 1459, 1350, 1299, 1249,1200, 1109, 948, 851 cm^{-1}; MS(FAB+) *m/z* 311 (MH^+, 47), 235 (4), 209 (3), 191 (3), 147 (17), 103 (60), 59 (100), 45 (17). Anal. Calcd for $C_{14}H_{30}O_7$: C, 54.18; H, 9.74. Found: C, 53.89; H, 9.75.

Synthesis of Hepta(ethylene glycol) Dimethyl Ether [2(7)]

According to the procedure described above, **2**(7) was prepared from di(ethylene glycol) monomethyl ether and tri(ethylene glycol) ditosylate [7]. The crude oil was chromatographed upon SiO_2 employing hexane-ethyl acetate 1.1 : 1 (v/v) as eluent to afford **2**(7) (64% yield) as a colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.67–3.63 (*m*, 24H, CH_2), 3.57–3.53 (*m*, 4H, $CH_3OCH_2CH_2$), 3.38 (*s*, 6H, CH_3O); IR (KBr) 3503, 2872, 1968, 1458, 1350, 1299, 1248, 1200, 1110, 948, 852 cm^{-1}; HRMS Calcd for $C_{16}H_{35}O_8$: 355.2332 (MH^+). Found: 355.2331. Anal. Calcd for $C_{16}H_{34}O_8$: C, 54.22; H, 9.67. Found: C, 54.42; H, 9.93.

Synthesis of Nona(ethylene glycol) Dimethyl Ether [2(9)]

According to the procedure described above, **2**(9) was prepared from tri(ethylene glycol) monomethyl ether and tri(ethylene glycol) ditosylate [7]. The crude oil was chromatographed upon SiO_2 employing hexane-ethyl acetate 1.5 : 1 (v/v) as eluent to afford **2**(9) (74% yield) as a colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.60–3.56 (*m*, 32H, CH_2), 3.50–3.46 (*m*, 4H, $CH_3OCH_2CH_2$), 3.31 (*s*, 6H, CH_3O); IR (KBr) 3503, 2872, 1968, 1458, 1350, 1297, 1250, 1200, 1109, 948, 851 cm^{-1}; HRMS Calcd for $C_{20}H_{43}O_{10}$: 443.2856 (MH^+). Found: 443.2852. Anal. Calcd for $C_{20}H_{42}O_{10}$: C, 54.28; H, 9.57. Found: C, 54.26; H, 9.72.

Synthesis of Dodeca(ethylene glycol) Dimethyl Ether [2(12)]

According to the procedure described above, **2**(12) was prepared from tetra(ethylene glycol) monomethyl ether and tetra(ethylene glycol) ditosylate [7]. The crude oil was chromatographed upon SiO_2 employing ethyl acetate as eluent to afford **2**(12) (528 mg, 0.918 mmol, 61%) as a colorless oil.

^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.66–3.62 (*m*, 44H, OCH_2CH_2O), 3.58–3.52 (*m*, 4H, $CH_3OCH_2CH_2$), 3.38 (*s*, 6H, CH_3); IR (KBr) 3560, 2871, 1972, 1459, 1350, 1297, 1253, 1108, 1041, 948, 851 cm^{-1}; HRMS Calcd for $C_{26}H_{55}O_{13}$: 575.3642 (MH^+). Found: 575.3628. Anal. Calcd for $C_{26}H_{54}O_{13}$: C, 54.34; H, 9.47. Found: C, 53.98; H, 9.47.

Synthesis of Eicosa(ethylene glycol) Dimethyl Ether [2(20)]

To a suspension of NaH (60%, 22.5 mg, 0.563 mmol) in dry DMF (1 mL) was added dodeca(ethylene glycol) (67.1 mg, 0.123 mmol) at 0°C. The suspension was stirred at 0°C for 20 min, tetra(ethylene glycol) monomethyl ether monotosylate (109 mg, 0.30 mmol) was added at 0°C, and the reaction mixture was stirred at room temperature for 12 h. Then, the reaction was quenched with brine (2 mL) at 0°C, and the resulting mixture was extracted with $CHCl_3$ (5 mL × 3). The extraction was dried with $MgSO_4$ and concentrated in *vacuo* [(70–150°C) (0.8 mmHg)]. The residue was chromatographed upon SiO_2 employing acetone as eluent to afford

pale yellow oil. Further purification by preparative GPC (column; JAIGEL-1H+2H; eluent $CHCl_3$) afforded **2**(20) (70.6 mg, 0.0761 mmol, 62%) as colorless waxy solid: ^1H NMR ($CDCl_3$, 300 MHz) δ 3.67–3.62 (*m*, 76H, OCH_2CH_2O), 3.58–3.53 (*m*, 4H, $CH_3OCH_2CH_2$), 3.38 (*s*, 6H, CH_3); IR (KBr) 3560, 2882, 1972, 1467, 1360, 1281, 1113, 1108, 945, 845 cm^{-1}; HRMS Calcd for $C_{42}H_{87}O_{21}$: 927.5739 (MH^+). Found: 927.5690. Anal. Calcd for $C_{42}H_{86}O_{21}$: C, 54.41; H, 9.35. Found: C, 54.41; H, 9.40.

Preparation of Inclusion Compounds

To a solution of (*R,R*)-**1** (0.10 mmol) in methanol (0.35 mL) was added excess guest molecules [**2**(*n*) or **3**(M_n)] at room temperature. After several days, the precipitated crystals were collected by filtration, washed with chloroform (3 mL), and dried in *vacuo* to afford the inclusion compounds.

X-ray Analyses

Powder X-ray diffractions were obtained with a MAC Science MXP18 diffractometer using graphite-monochromated Cu Kα radiation (40 kV, 300 mA). The spectra were measured at room temperature between 2° and 50° in the 2θ scan mode with steps of 0.01° in 2θ and 4°/min. Diffraction data were presented in *d* Å and relative reflecting intensities are in parentheses.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine • 1,2-Dimethoxyethane Complex [(R,R)-1·2(1)]

Colorless plate; dec 129.9–131.1°C; ^1H NMR (D_2O, 300 MHz) δ 8.12–8.04 (*m*, 3H, Ar*H*), 7.74–7.59 (*m*, 4H, Ar*H*), 7.38–7.35 (*d* like, 5H, Ar*H*), 5.94 (*s*, 1H, H_2NCHCO), 5.16 (*s*, 1H, CONHC*H*), 3.59 (*s*, 4H, CH_2), 3.35 (*s*, 6H, CH_3O); IR (KBr) 3370, 2940, 1668, 1571, 1488, 1373, 1238, 1091, 1030, 967, 856, 792, 769, 697 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.0 (1.00), 4.82 (0.41), 4.04 (0.43). Anal. Calcd for $C_{20}H_{18}N_2O_3 \cdot C_4H_{10}O_2$: C, 67.91; H, 6.65; N, 6.60. Found: C, 67.81; H, 6.46; N, 6.75.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine • Di(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(2)]

Colorless plate; dec 142.0–143.0°C (sintering); ^1H NMR (D_2O, 300 MHz) δ 8.13–8.05 (*m*, 3H, Ar*H*), 7.74–7.59 (*m*, 4H, Ar*H*), 7.37–7.34 (*d* like, 5H, Ar*H*), 5.94 (*s*, 1H, H_2NCHCO), 5.16 (*s*, 1H, CONHC*H*), 3.67–3.58 (*m*, 6H, CH_2), 3.36 (*s*, 4H, CH_3O); IR (KBr) 3360, 3060, 2930, 1670, 1572, 1490, 1373, 1092, 793, 768, 748, 695 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.2 (1.00), 4.05 (0.47). Anal. Calcd for $1.56C_{20}H_{18}N_2O_3 \cdot C_6H_{14}O_3$: C, 68.14; H, 6.47; N, 6.66. Found: C, 67.95; H, 6.17; N, 6.84.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine • Tri(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(3)]

Colorless plate; dec 150.9–152.7°C (sintering); ^1H NMR (D_2O, 300 MHz) δ 8.13–8.06 (*m*, 3H, Ar*H*), 7.76–7.61 (*m*, 4H, Ar*H*), 7.38–7.35 (*d* like, 5H, Ar*H*), 5.97 (*s*, 1H, H_2NCHCO), 5.17 (*s*, 1H, CONHC*H*), 3.70–3.59 (*m*, 7.2H, CH_2), 3.36 (*s*, 3.5H, CH_3O); IR (KBr) 3360, 3050, 2940, 1674, 1576, 1496, 1372, 1237, 1092, 792, 769, 696 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.2 (1.00), 4.83 (0.50), 4.05 (0.32). Anal. Calcd for $1.92C_{20}H_{18}N_2O_3 \cdot C_8H_{18}O_4$: C, 67.95; H, 6.46; N, 6.56. Found: C, 67.71; H, 6.24; N, 6.72.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine • Tetra(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(4)]

Colorless plate; dec 154.9–158.5°C (sintering); ^1H NMR (D_2O, 300 MHz) δ 8.15–8.07 (*m*, 3H, Ar*H*), 7.77–7.61 (*m*, 4H, Ar*H*), 7.39–7.37 (*d* like,

5H, Ar*H*), 5.98 (*s*, 1H, H_2NCHCO), 5.19 (*s*, 1H, CONHC*H*), 3.69–3.61 (*m*, 6H, CH_2), 3.38 (*s*, 2.2H, CH_3O); IR (KBr) 3360, 3050, 2920, 1674, 1574, 1501, 1376, 1309, 1236, 1100, 790, 766, 695 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.2 (0.69), 4.85 (1.00), 4.05 (0.40). Anal. Calcd for $2.50C_{20}H_{18}N_2O_3 \cdot C_{10}H_{22}O_5$: C, 68.10; H, 6.38; N, 6.62. Found: C, 68.26; H, 6.27; N, 6.72.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · Penta(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(5)]

Colorless crystals; dec. 144.7°C (DSC, sintering); ^1H NMR (D_2O, 300 MHz) δ 8.14–8.05 (*m*, 3H, Ar*H*), 7.76–7.60 (*m*, 4H, Ar*H*), 7.42–7.32 (*d* like, 5H, Ar*H*), 5.97 (*s*, 1H, H_2NCH(Nap)CO), 5.17 (*s*, 1H, NHC*H*(Ph)COOH), 3.70–3.65 (*m*, 5.16H, CH_2), 3.63–3.58 (*m*, 1.29H, $CH_3OCH_2CH_2$), 3.37 (*s*, 1.94H, CH_3O); IR (KBr) 3359, 3060, 2924, 1676, 1577, 1498, 1377, 1098, 792, 770, 698 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.0 (0.84), 5.13 (0.29), 4.82 (1.00), 4.26 (0.35), 4.02 (0.49), 3.78 (0.35), 3.22 (0.21). Anal. Calcd for $3.10C_{20}H_{18}N_2O_3 \cdot C_{12}H_{26}O_6$: C, 68.22; H, 6.33; N, 6.67. Found: C, 67.94; H, 6.03; N, 6.44.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · Hexa(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(6)]

Colorless crystals; dec. 146.0°C (DSC, sintering); ^1H NMR (D_2O, 300 MHz) δ 8.14–8.04 (*m*, 3H, Ar*H*), 7.76–7.60 (*m*, 4H, Ar*H*), 7.41–7.30 (*d* like, 5H, Ar*H*), 5.94 (*s*, 1H, H_2NCH(Nap)CO), 5.16 (*s*, 1H, NHC*H*(Ph)COOH), 3.69-3.62 (*m*, 6.35H, CH_2), 3.61–3.58 (*m*, 1.26H, CH_3, OCH_2CH_2), 3.39 (*s*, 1.90H, CH_3O); IR (KBr) 3414, 3144, 3051, 2924, 1686, 1560, 1508, 1491, 1377, 1260, 1100, 808, 733, 699 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.3 (1.00), 4.81 (0.35), 4.41 (0.75), 3.94 (0.67), 3.37 (0.56). Anal. Calcd for $3.15C_{20}H_{18}N_2O_3 \cdot C_8H_{18}O_4$: C, 67.82; H, 6.41; N, 6.47. Found: C, 67.53; H, 6.43; N, 6.40.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · Hepta(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(7)]

Colorless crystals; dec 145.8–147.3°C (sintering); ^1H NMR (D_2O, 300 MHz) δ 8.12–8.05 (*m*, 3H, Ar*H*), 7.74–7.68 (*m*, 4H, Ar*H*), 7.35–7.31 (*d* like, 5H, Ar*H*), 5.95 (*s*, 1H, H_2NCHCO), 5.16 (*s*, 1H, CONHC*H*), 3.72–3.09 (*m*, 8.8H, CH_2), 3.36 (*s*, 2.4H, CH_3O); IR (KBr) 3360, 3050, 2915, 1672, 1572, 1493, 1370, 1309, 1236, 1104, 970, 794, 696 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.1 (1.00), 4.85 (0.70), 4.02 (0.41). Anal. Calcd for $3.80C_{20}H_{18}N_2O_3 \cdot C_{16}H_{34}O_8$: C, 68.00; H, 6.35; N, 6.55. Found: C, 67.77; H, 6.28; N, 6.64.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · Nona(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(9)]

Colorless crystals; dec. 156.0°C (DSC); ^1H NMR (D_2O, 300 MHz) δ 8.15–8.05 (*m*, 3H, Ar*H*), 7.75–7.60 (*m*, 4H, Ar*H*), 7.42–7.32 (*d* like, 5H, Ar*H*), 5.96 (*s*, 1H, H_2NCH(Nap)CO), 5.18 (*s*, 1H, NHC*H*(Ph)COOH), 3.71–3.65 (*m*, 6.40H, CH_2), 3.63–3.59 (*m*, 0.80H, $CH_3OCH_2CH_2$), 3.37 (*s*, 1.20H, CH_3O); IR (KBr) 3415, 3054, 2930, 2867, 1686, 1571, 1560, 1491, 1381, 1099, 777, 734, 699 cm^{-1}; Powder X-ray analysis [Å (I/I_0)] 12.3 (1.00), 4.84 (0.32), 4.62 (0.51), 4.48 (0.55), 4.42 (0.49), 3.97 (0.68), 3.40 (0.59). Anal. Calcd for $5.00C_{20}H_{18}N_2O_3 \cdot C_{20}H_{42}O_{10}$: C, 68.17; H, 6.29; N, 6.62. Found: C, 67.90; H, 6.24; N, 6.59.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · Dodeca(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(12)]

Colorless crystals; dec. 153.1°C (DSC); ^1H NMR (D_2O, 300 MHz) δ 8.16–8.07 (*m*, 3H, Ar*H*), 7.77–7.60 (*m*, 4H, Ar*H*), 7.44–7.33 (*d* like, 5H, Ar*H*), 5.97 (*s*, 1H, H_2NCH(Nap)CO), 5.17 (*s*, 1H, NHC*H*(Ph)COOH), 3.71–3.65 (*m*, 4.08H, CH_2), 3.63–3.59 (*m*, 1.36H, $CH_3OCH_2CH_2$), 3.37 (*s*, 2.04H. CH_3O); IR (KBr) 3355, 3060, 2921, 1676,

1578, 1499, 1378, 1098, 792, 769, 697 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.1 (1.00), 4.85 (0.87), 4.04 (0.51). Anal. Calcd for $6.40C_{20}H_{18}N_2O_3 \cdot C_{26}H_{54}O_{13}$: C, 68.14; H, 6.29; N, 6.60. Found: C, 67.86; H, 6.14; N, 6.65.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · Eicosa(ethylene glycol) Dimethyl Ether Complex [(R,R)-1 · 2(20)]

Colorless crystals; dec. 156.7°C (DSC); ^1H NMR ($D_2O + DCl$, 300 MHz) δ 8.18–8.09 (*m*, 3H, Ar*H*), 7.80–7.63 (*m*, 4H, Ar*H*), 7.45–7.33 (*d* like, 5H, Ar*H*), 6.01 (*s*, 1H, H_2NCH(Nap)CO), 5.32 (*s*, 1H, NHC*H*(Ph)COOH), 3.76–3.68 (*m*, 7.23H, CH_2), 3.68–3.61 (*m*, 0.38H, $CH_3OCH_2CH_2$), 3.40 (*s*, 0.57H, CH_3O); IR (KBr) 3354, 3060, 2920, 1677, 1589, 1499, 1377, 1098, 790, 697 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.13 (1.00), 5.09 (0.29), 4.83 (0.83), 4.03 (0.67), 3.22 (0.18). Anal. Calcd for $10.50C_{20}H_{18}N_2O_3 \cdot C_{42}H_{86}O_{21}$: C, 68.20; H, 6.25; N, 6.63. Found: C, 67.96; H, 6.18; N, 6.62.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #400 Complex [(R,R)-1 · 3(400)]

Colorless crystals; dec. 153.0°C (DSC); ^1H NMR (D_2O, 300 MHz) δ 8.17–8.07 (*m*, 3H, Ar*H*), 7.77–7.61 (*m*, 4H, Ar*H*), 7.42–7.33 (*d* like, 5H, Ar*H*), 5.98 (*s*, 1H, H_2NCH(Nap)CO), 5.18 (*s*, 1H, NHC*H*(Ph)COOH), 3.75–3.61 (*m*, 8.80H, CH_2); IR (KBr) 3368, 3060, 2923, 1676, 1578, 1499, 1377, 1260, 1098, 792, 697 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.03 (0.93), 7.91 (0.16), 5.08 (0.21), 4.84 (1.00), 4.03 (0.50), 3.78 (0.21), 3.22 (0.13). Anal. Calcd for $C_{20}H_{18}N_2O_2 \cdot C_4H_8O_2 \cdot 0.22H_2O$: C, 67.30; H, 6.32; N, 6.42. Found: C, 67.20; H, 6.17; N, 6.50.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #600 Complex [(R,R)-1 · 3(600)]

Colorless crystals; dec. 152.8°C (DSC); ^1H NMR (D_2O, 300 MHz) δ 8.17–8.07 (*m*, 3H, Ar*H*), 7.78–7.61 (*m*, 4H, Ar*H*), 7.42–7.33 (*d* like, 5H, Ar*H*), 5.99 (*s*, 1H, H_2NCH(Nap)CO), 5.19 (*s*, 1H, NHC*H*(Ph)COOH), 3.75–3.61 (*m*, 4.08H, CH_2), 3.63–3.59 (*m*, 1.36H, $CH_3OCH_2CH_2$), 3.37 (*s*, 2.04H, CH_3O); IR (KBr) 3355, 3060, 2922, 1676, 1590, 1499, 1377, 1256, 1098, 791, 696 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.0 (0.82), 7.91 (0.14), 5.09 (0.29), 4.84 (1.00), 4.03 (0.51), 3.78 (0.20), 3.22 (0.14). Anal. Calcd for $0.94C_{20}H_{18}N_2O_3 \cdot C_4H_8O_2 \cdot 0.15H_2O$: C, 67.60; H, 6.27; N, 6.50. Found: C, 67.21; H, 6.23; N, 6.63.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #1,000 Complex [(R,R)1 · 3(1,000)]

Colorless crystals; dec. 156.7°C (DSC); ^1H NMR (CD_3OD, 300 MHz) δ 8.22 (*d* like, 1H, Ar*H*), 8.01–7.95 (*m*, 2H, Ar*H*), 7.75–7.71 (*m*, 1H, Ar*H*), 7.67–7.51 (*m*, 3H, Ar*H*), 7.47–7.42 (*m*, 2H, Ar*H*), 7.31–7.18 (*m*, 3H, Ar*H*), 5.85 (*s*, 1H, H_2NCHCO), 5.25 (*s*, 1H, CONHC*H*), 3.66–3.49 (*m*, 8.00H, CH_2); IR (KBr) 3354, 3060, 2921, 1677, 1589, 1499, 1378, 1252, 1098, 791, 697 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.1 (0.74), 7.92 (0.14), 5.08 (0.31), 4.84 (1.00), 4.03 (0.51), 3.78 (0.24), 3.22 (0.17). Anal. Calcd for $C_{20}H_{18}N_2O_3 \cdot C_4H_8O_2 \cdot 0.088H_2O$: C, 67.98; H, 6.22; N, 6.61. Found: C, 67.76; H, 6.14; N, 6.65.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #4,000 Complex [(R,R)-1 · 3(4,000)]

Colorless crystals; dec. 157.5°C (DSC); ^1H NMR ($D_2O + DCl$, 300 MHz) δ 8.51–8.33 (*m*, 3H, Ar*H*), 8.08–7.92 (*m*, 4H, Ar*H*), 7.69–7.56 (*d* like, 5H, Ar*H*), 6.23 (*s*, 1H, H_2NCHCO), 5.44 (*s*, 1H, CONHC*H*), 4.02–3.86 (*m*, 8.51H, CH_2); IR (KBr) 3354, 3060, 2922, 1676, 1588, 1499, 1377, 1253, 1097, 791, 696 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.0 (0.85), 7.91 (0.15), 5.07 (0.26), 4.84 (1.00), 4.04 (0.53), 3.77 (0.23), 3.22 (0.15). Anal. Calcd for $0.94C_{20}H_{18}N_2O_3 \cdot C_4H_8O_2 \cdot 0.022H_2O$: C, 67.98; H, 6.25; N, 6.54. Found: C, 67.60; H, 6.35; N, 6.45.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #6,000 Complex [(R,R)-1 · 3(6,000)]

Colorless crystals; dec. 159.0°C (DSC); ^{1}H NMR (D_2O + DCl, 300 MHz) δ 8.13–8.06 (*m*, 3H, Ar*H*), 7.75–7.63 (*m*, 4H, Ar*H*), 7.40–7.33 (*d* like, 5H, Ar*H*), 5.98 (*s*, 1H, H_2NCH(Nap)CO), 5.47 (*s*, 1H, NHC*H*(Ph)COOH), 3.70–3.62 (*m*, 8.60H, CH_2); IR (KBr) 3358, 3060, 2958, 2921, 1676, 1577, 1508, 1377, 1309, 1261, 1137, 1097, 956, 792, 769, 696 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.07 (0.83), 7.91 (0.15), 5.07 (0.23), 4.84 (1.00), 4.03 (0.50), 3.22 (0.13). Anal. Calcd for $0.93C_{20}H_{18}N_2O_3 \cdot C_4H_8O_2 \cdot 0.2H_2O$: C, 67.41; H, 6.29; N, 6.47. Found: C, 67.13; H, 6.16; N, 6.55.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #20,000 Complex [(R,R)-1 · 3(20,000)]

Colorless crystals; dec. 162.4°C (DSC); ^{1}H NMR (D_2O + DCl, 300 MHz) δ 8.13–8.06 (*m*, 3H, Ar*H*), 7.75–7.63 (*m*, 4H, Ar*H*), 7.40–7.33 (*d* like, 5H, Ar*H*), 5.99 (*s*, 1H, H_2NCH(Nap)CO), 5.51 (*s*, 1H, NHC*H*(Ph)COOH), 3.70–3.62 (*m*, 7.20H, CH_2); IR (KBr) 3353, 3060, 2921, 1676, 1577, 1508, 1376, 1309, 1252, 1137, 1097, 969, 791, 769, 696 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.07 (0.85), 7.90 (0.15), 5.07 (0.25), 4.84 (1.00), 4.03 (0.48), 3.21 (0.15). Anal. Calcd for $1.06C_{20}H_{18}N_2O_3 \cdot C_4H_8O_2 \cdot 0.1H_2O$: C, 68.12; H, 6.19; N, 6.68. Found: C, 67.81; H, 5.93; N, 6.89.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · Tetra(ethylene glycol) Dimethyl Ether Dodeca(ethylene glycol) Dimethyl Ether [(R,R)-1 · 2(4) · 2(12)] Complex

To a solution of (*R,R*)-**1** (0.40 mmol) in methanol (1.5 mL) was added **2**(12) and **2**(4) (49:51 v/v, 82.0 mg) at room temperature. After several days, the precipitated crystals were collected by filtration, washed with chloroform (10 mL), and dried in *vacuo* to afford an inclusion compound [(*R,R*)-**1**·**2**(4)·**2**(12); 143 mg]: colorless crystals; dec. 156.0°C (DSC); ^{1}H NMR (D_2O, 300 MHz) δ 8.15–8.06 (*m*, 3H, Ar*H*), 7.79–7.62 (*m*, 4H, Ar*H*), 7.42–7.32 (*d* like, 5H, Ar*H*), 5.96 (*s*, 1H, H_2NCH(Nap)CO), 5.18 (*s*, 1H, NHC*H*(Ph)COOH), 3.71–3.65 (*m*, 6.87H, CH_2), 3.63–3.59 (*m*, 0.70H, $CH_3OCH_2CH_2$), 3.40 (*s*, 1.09H, CH_3O); IR (KBr) 3416, 3367, 3056, 2929, 2874, 1686, 1571, 1492, 1380, 1098, 790, 734, 699 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.18 (1.00), 4.83 (0.64), 4.61 (0.61), 4.48 (0.65), 3.96 (0.77), 3.39 (0.63). Anal. Calcd for $5.30C_{20}H_{18}N_2O_3 \cdot 9.60(C_2H_4O) \cdot C_2H_6O$: C, 68.17; H, 6.29; N, 6.62. Found: C, 67.81; H, 6.20; N, 6.64.

The inclusion compound was dissolved in 1 M hydrochloric acid (1.5 mL), and extracted with chloroform (5 mL × 3). The combined organic layers were dried over anhydrous $MgSO_4$ and concentrated under reduced pressure to afford the mixture of **2**(4) and **2**(12) (34.4 mg) as a colorless oil. The ratio of **2**(4):**2**(12) was determined by GPC (Shodex GPC OHPak SB-802HQ) and the result was shown in text.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #1,000 and 20,000 Complex

To a solution of (*R,R*)-**1** (0.50 mmol) in methanol (1.5 mL) was added PEG #1,000 and 20,000 (**3**, sample A, 1:1 w/w, 153 mg) at room temperature. After several days, the precipitated crystals were collected by filtration, washed with chloroform (10 mL), and dried in *vacuo* to afford an inclusion compound [(*R,R*)-**1**·**3**; 189 mg]: colorless crystals; dec. 160.6°C (DSC); ^{1}H NMR (D_2O, 300 MHz) δ 8.13–8.06 (*m*, 3H, Ar*H*), 7.75–7.63 (*m*, 4H, Ar*H*), 7.40–7.33 (*d* like, 5H, Ar*H*), 5.96 (*s*, 1H, H_2NCH(Nap)CO), 5.19 (*s*, 1H, NHC*H*(Ph)COOH), 3.70–3.62 (*m*, 8.00H, CH_2); IR (KBr) 3353, 3060, 2921, 1677, 1578, 1508, 1499, 1377, 1098, 791, 769, 697 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.0 (0.65), 5.06 (0.28), 4.83 (1.00), 4.02 (0.49), 3.78 (0.22), 3.21 (0.16). Anal. Calcd for $C_{20}H_{18}N_2O_3 \cdot C_4H_8O_2 \cdot 0.15H_2O$: C, 67.80; H, 6.23; N, 6.59. Found: C, 67.58; H, 6.15; N, 6.53.

(*R,R*)-**1**·**3** was dissolved in 1 M hydrochloric acid (3 mL), and extracted with chloroform (10 mL × 3). The combined organic layers were dried with anhydrous $MgSO_4$ and concentrated under reduced pressure to afford **3** (38 mg) as a colorless waxy solid. The polydispersity index (M_w/M_n) was determined by GPC (Shodex GPC OHPak SB-804HQ) and the results were summarized in Table III.

(R)-(1-Naphthyl)glycyl-(R)-phenylglycine · PEG #600, 1,000, and 1,500 Complex

To a solution of (*R,R*)-**1** (0.40 mmol) in methanol (1.5 mL) was added PEG #600, 1,000, and 1,500 (**3**, sample B, 1:1:1 w/w/w, 122 mg) at room temperature. After several days, the precipitated crystals were collected by filtration, washed with chloroform (10 mL), and dried in *vacuo* to afford a inclusion compound [(*R,R*)-**1**·**3**; 123 mg]: colorless crystals; dec. 160.4°C (DSC); ^{1}H NMR (D_2O, 300 MHz) δ 8.13–8.07 (*m*, 3H, Ar*H*), 7.75–7.63 (*m*, 4H, Ar*H*), 7.40–7.33 (*d* like, 5H, Ar*H*), 5.97 (*s*, 1H, H_2NCH(Nap)CO), 5.19 (*s*, 1H, NHC*H*(Ph)COOH), 3.70–3.60 (*m*, 8.00H, CH_2); IR (KBr) 3353, 3060, 2921, 1677, 1578, 1508, 1499, 1377, 1098, 791, 769, 697 cm^{-1}; Powder X-ray diffraction [Å (I/I_0)] 12.0 (1.00), 7.86 (0.17), 5.05 (0.22), 4.81 (1.00), 3.99 (0.51), 3.76 (0.17), 3.20 (0.14). Anal. Calcd for $C_{20}H_{18}N_2O_3 \cdot C_4H_8O_2 \cdot 0.20H_2O$: C, 67.65; H, 6.25; N, 6.57. Found: C, 67.75; H, 6.08; N, 6.73.

(*R,R*)-**1**·**3** was dissolved in 1 M hydrochloric acid (3 mL), and extracted with chloroform (10 mL × 3) was performed. The combined organic layers were dried over anhydrous $MgSO_4$ and concentrated under reduced pressure to afford **3** (27 mg) as a colorless waxy solid. The results were shown in Figure 10 and Table III.

X-ray Crystallography

Data collection was performed on a Mac Science MXC18 four-circle diffractometer with graphite monochromated Cu Kα ($\lambda = 1.54178$ Å) radiation using the 2θ-ω scan technique, and the X-ray intensities were measured up to $2\theta = 140°$ at 298 or 173 K, respectively. The structures were solved and refined by a computer program package; CRYSTAN-GM ver. 6.2.1 or maXus ver. 1.1 from MAC Science Co. Ltd. The structures solved by a direct method (SIR 92 [9] on a computer program package).

The Inclusion Compound of DME [(R,R)-1 · 2(1)]

Colorless plate, crystal dimensions 0.40 × 0.25 × 0.15 mm^3, $C_{24}H_{28}N_2O_5$, Orthorhombic, $P2_12_12_1$, $a = 15.813$ (2) Å, $b = 24.328$ (4) Å, $c = 5.698$ (1) Å, $V = 2191.9$ (6) Å^3, $Z = 4$, $d_{cal} = 1.29$ g cm^{-3}, $2\theta_{max} = 140°$, temperature 298 K, 2513 reflections measured, 2410 independent, $R = 0.0514$ (1796 reflections with $|F_0| > 3\sigma(|F_0|)$); $R_w = 0.0519$, 376 parameters, with heavy atoms refined anisotropically, residual electron density 0.23/−0.23.

The Inclusion Compound of Hepta(ethylene glycol) Dimethyl Ether [(R,R)-1 · 2(7)]

Colorless plate, $C_{24}H_{26}O_5N_2$, crystal dimensions 0.15 × 0.08 × 0.06 mm^3, Orthorhombic, $P2_12_12_1$, $a = 15.817$ (4) Å, $b = 24.182$ (7) Å, $c = 5.610$ (2) Å, $V = 2146$ (1) Å^3, $Z = 4$, $d_{cal} = 1.307$ g cm^{-3}, $2\theta_{max} = 140°$, temperature 173 K, 2485 reflections measured, 2042 independent, $R = 0.106$ ($|F_0| > 1.50\sigma(|F_0|)$); $R_w = 0.089$, 259 parameters, with heavy atoms refined anisotropically except the atoms of the guest molecule, residual electron density 0.64/−0.70.

Acknowledgments

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Supplementary Material

(*R*)-(1-Naphthyl)glycyl-(*R*)-phenylglycine · 1,2-dimethoxyethane complex [(*R*,*R*)-**1**·**2**(1)]

Crystal data:	
Chemical formula	$C_{24}H_{28}O_5N_2$
Formula weight	424.50
Crystal description	Colourless plate
Crystal size/mm	0.40 × 0.25 × 0.15
a/Å	15.813 (2)
b/Å	24.328 (4)
c/Å	5.698 (1)
alpha/deg	90.000 (0)
beta/deg	90.000 (0)
gamma/deg	90.000 (0)
Volume of unit cell/Å^3	2191.9 (6)
Crystal system	Orthorhombic
Space group	$P\,2_12_12_1$ (No. 19)
Z value	4
Density: Dcalc/gcm^{-3}	1.29
Data Collection:	
Diffractometer used	Mac Science MXC18
Radiation	Cu K-alpha (lambda = 1.54178 Å)
Total reflections measured	2513
Reflection (*hkl*) limits	$0 < h < 19$
	$0 < k < 29$
	$0 < l < 6$
Unique reflections	2410
Internal consistency: Rint	0.00
Measurement temperature/K	298
Refinement:	
Least squares refinement method	Full matrix
Weight method	Count statistics
Absorption correction	none
Data reduction cut-off	3.00
Maximum theta	70
Hydrogen treatment	refall
Linear absorption coefficient/cm^{-1}	6.998
R	0.0458
R_w	0.0475
Reflections used in L.S.	1796
L.S. parameters	362
Max shift/esd	0.6305
Average shift/esd	0.0082
Maximum peak in final Fourier map	0.23
Minimum peak in final Fourier map	−0.23
Programs used to solve and refinement	CRYSTAN-GM ver.6.2.1 (Mac Science)

Fractional atomic coordinates and U(iso)

Atom	x/a	y/b	z/c	U(iso)
O(1)	0.33343(16)	0.21501(12)	0.47418(57)	0.056
O(2)	0.54669(15)	0.24858(15)	1.05410(60)	0.067
N(3)	0.18868(18)	0.22538(13)	0.71429(76)	0.050
N(4)	0.41371(18)	0.22787(14)	0.79623(69)	0.053
O(5)	0.63487(15)	0.24755(13)	0.75007(61)	0.063
C(6)	0.26463(19)	0.25143(14)	0.81721(74)	0.045
C(7)	0.5650(2)	0.2392(2)	0.8437(8)	0.053
C(8)	0.34170(19)	0.22959(12)	0.67836(76)	0.044
C(9)	0.3071(2)	0.3462(1)	0.9700(9)	0.051
C(10)	0.3077(2)	0.4050(2)	0.9413(10)	0.061
C(11)	0.4954(2)	0.2122(2)	0.6913(9)	0.051
C(12)	0.26053(19)	0.31411(13)	0.80576(82)	0.048
C(13)	0.3532(3)	0.3239(2)	1.1606(9)	0.063
C(14)	0.5055(2)	0.1497(2)	0.6924(9)	0.057
C(15)	0.2156(3)	0.3396(2)	0.6316(10)	0.063
C(16)	0.2146(3)	0.3977(2)	0.6134(12)	0.078
C(17)	0.3571(3)	0.4369(2)	1.1013(13)	0.076
O(18)	0.2342(3)	0.1119(2)	0.8601(12)	0.120
C(19)	0.5429(4)	0.1225(2)	0.8761(12)	0.079
C(20)	0.4746(3)	0.1200(2)	0.5048(12)	0.078
C(21)	0.2605(3)	0.4288(2)	0.7629(12)	0.073
C(22)	0.4006(4)	0.3562(2)	1.3075(12)	0.081
C(23)	0.4785(4)	0.0624(3)	0.5105(17)	0.098
C(24)	0.4027(4)	0.4135(2)	1.2769(14)	0.090
C(25)	0.5144(4)	0.0362(3)	0.6975(17)	0.098
C(26)	0.5485(5)	0.0653(2)	0.8801(15)	0.096
O(27)	0.1431(6)	0.1130(3)	0.4564(19)	0.198
C(28)	0.0790(8)	0.1173(6)	0.2606(25)	0.152
C(29)	0.3041(5)	0.1061(5)	1.0234(26)	0.149
C(30)	0.2285(8)	0.0671(4)	0.7118(34)	0.194
C(31)	0.1451(9)	0.0683(5)	0.5859(41)	0.238
H(3A)	0.179(3)	0.234(2)	0.565(10)	0.07(2)
H(6)	0.273(2)	0.241(2)	0.988(8)	0.04(1)
H(17)	0.352(3)	0.478(2)	1.065(11)	0.09(2)
H(3B)	0.192(2)	0.188(2)	0.708(9)	0.05(1)
H(19)	0.565(3)	0.140(2)	1.015(11)	0.09(2)
H(13)	0.348(3)	0.284(2)	1.210(9)	0.07(1)
H(15)	0.191(2)	0.322(2)	0.508(9)	0.05(1)
H(11)	0.503(2)	0.227(1)	0.520(7)	0.04(1)
H(4)	0.417(2)	0.239(2)	0.933(7)	0.032(9)
H(21)	0.259(3)	0.469(2)	0.722(12)	0.10(2)
H(16)	0.187(3)	0.412(2)	0.478(10)	0.07(2)
H(3C)	0.135(3)	0.236(2)	0.802(9)	0.06(1)
H(26)	0.569(4)	0.042(3)	1.029(13)	0.13(2)
H(22)	0.418(5)	0.339(3)	1.447(15)	0.14(3)
H(24)	0.433(4)	0.441(3)	1.382(13)	0.13(3)
H(23)	0.461(4)	0.046(3)	0.367(13)	0.12(3)
H(20)	0.460(3)	0.143(2)	0.362(10)	0.09(2)
H(25)	0.514(4)	0.004(2)	0.711(13)	0.10(2)
H(28A)	0.07809	0.15025	0.16838	0.7(2)
H(28B)	0.02429	0.11325	0.33138	1.4(5)
H(28C)	0.08849	0.08705	0.15558	0.30(9)
H(31A)	0.14198	0.03681	0.48447	0.5(2)
H(31B)	0.09748	0.06791	0.69047	10.9(158)
H(30A)	0.22898	0.03429	0.80524	0.33(9)
H(30B)	0.27558	0.06629	0.60574	12.9(120)
H(29A)	0.30951	0.13739	1.12476	0.5(1)
H(29B)	0.35561	0.10179	0.93626	2.6(14)
H(29C)	0.29511	0.07389	1.11766	0.28(7)

Anisotropic thermal parameters

Atom	*U*11	*U*22	*U*33	*U*12	*U*13	*U*23
O(1)	0.045(1)	0.062(2)	0.062(2)	0.010(1)	−0.001(1)	−0.004(1)
O(2)	0.038(1)	0.098(2)	0.064(2)	0.001(1)	0.000(1)	−0.021(2)
N(3)	0.036(1)	0.047(1)	0.069(2)	−0.004(1)	0.001(1)	−0.006(2)
N(4)	0.036(1)	0.062(2)	0.060(2)	0.007(1)	−0.001(2)	−0.012(2)
O(5)	0.040(1)	0.078(2)	0.069(2)	−0.007(1)	0.006(1)	−0.007(2)
C(6)	0.034(1)	0.041(1)	0.060(2)	0.001(1)	0.001(2)	−0.002(2)
C(7)	0.035(1)	0.057(2)	0.066(3)	0.005(1)	−0.002(2)	−0.010(2)
C(8)	0.036(1)	0.035(1)	0.060(2)	0.001(1)	0.002(2)	0.002(2)
C(9)	0.044(1)	0.043(2)	0.065(3)	−0.001(1)	0.008(2)	−0.002(2)
C(10)	0.053(2)	0.043(2)	0.085(3)	0.000(2)	0.008(2)	−0.007(2)
C(11)	0.034(1)	0.057(2)	0.062(3)	0.003(1)	0.002(2)	−0.009(2)
C(12)	0.037(1)	0.041(1)	0.068(2)	0.003(1)	0.005(2)	−0.001(2)
C(13)	0.067(2)	0.056(2)	0.066(3)	−0.003(2)	−0.002(2)	−0.007(2)
C(14)	0.038(1)	0.057(2)	0.075(3)	0.001(1)	0.007(2)	−0.006(2)
C(15)	0.053(2)	0.049(2)	0.088(4)	0.006(2)	−0.015(2)	−0.005(2)
C(16)	0.070(3)	0.059(2)	0.104(5)	0.015(2)	−0.018(3)	0.021(3)
C(17)	0.066(3)	0.049(2)	0.113(5)	−0.010(2)	0.013(3)	−0.015(3)
O(18)	0.115(3)	0.078(3)	0.168(6)	0.006(2)	0.002(4)	−0.001(3)
C(19)	0.083(3)	0.071(3)	0.084(4)	−0.006(3)	0.001(3)	0.005(3)
C(20)	0.075(3)	0.067(3)	0.093(4)	0.011(2)	−0.008(3)	−0.026(3)
C(21)	0.065(2)	0.041(2)	0.113(4)	0.007(2)	0.000(3)	0.009(3)
C(22)	0.095(3)	0.076(3)	0.073(4)	−0.008(3)	−0.017(3)	−0.013(3)
C(23)	0.089(4)	0.084(4)	0.122(6)	0.001(3)	0.003(4)	−0.042(4)
C(24)	0.088(3)	0.077(3)	0.105(5)	−0.016(3)	−0.016(4)	−0.028(4)
C(25)	0.089(4)	0.059(3)	0.146(7)	0.000(3)	0.040(5)	0.001(4)
C(26)	0.111(4)	0.066(3)	0.111(6)	0.003(3)	0.010(5)	0.020(3)
O(27)	0.251(9)	0.107(5)	0.237(10)	−0.038(5)	−0.051(9)	−0.011(6)
C(28)	0.152(9)	0.143(8)	0.162(10)	−0.029(6)	0.007(9)	−0.010(9)
C(29)	0.100(5)	0.137(7)	0.211(12)	−0.010(5)	−0.016(7)	0.056(9)
C(30)	0.21(1)	0.08(1)	0.29(2)	0.02(1)	−0.05(1)	−0.05(1)
C(31)	0.23(1)	0.08(1)	0.40(3)	−0.03(1)	−0.08(2)	0.01(1)

Intramolecular bond lengths (Å)

O(1)–C(8)	1.223(6)	O(2)–C(7)	1.254(6)
N(3)–C(6)	1.479(5)	N(3)–H(3A)	0.89(6)
N(3)–H(3B)	0.92(5)	N(3)–H(3C)	1.01(5)
N(4)–C(8)	1.323(5)	N(4)–C(11)	1.474(5)
N(4)–H(4)	0.83(4)	O(5)–C(7)	1.244(5)
C(6)–C(8)	1.547(5)	C(6)–C(12)	1.528(5)
C(6)–H(6)	1.01(5)	C(7)–C(11)	1.548(6)
C(9)–C(10)	1.440(6)	C(9)–C(12)	1.423(6)
C(9)–C(13)	1.416(7)	C(10)–C(17)	1.429(8)
C(10)–C(21)	1.387(8)	C(11)–C(14)	1.529(6)
C(11)–H(11)	1.05(5)	C(12)–C(15)	1.369(7)
C(13)–C(22)	1.370(8)	C(13)–H(13)	1.00(5)
C(14)–C(19)	1.373(8)	C(14)–C(20)	1.380(8)
C(15)–C(16)	1.418(7)	C(15)–H(15)	0.92(5)
C(16)–C(21)	1.350(8)	C(16)–H(16)	0.96(6)
C(17)–C(24)	1.358(10)	C(17)–H(17)	1.02(6)
O(18)–C(29)	1.452(14)	O(18)–C(30)	1.382(15)
C(19)–C(26)	1.394(9)	C(19)–H(19)	0.97(7)
C(20)–C(23)	1.403(9)	C(20)–H(20)	1.02(6)
C(21)–H(21)	1.00(6)	C(22)–C(24)	1.407(9)

C(22)–H(22)	0.94(9)	C(23)–C(25)	1.366(13)
C(23)–H(23)	0.95(8)	C(24)–H(24)	1.01(7)
C(25)–C(26)	1.369(12)	C(25)–H(25)	0.80(6)
C(26)–H(26)	1.07(8)	O(27)–C(28)	1.511(17)
O(27)–C(31)	1.314(18)	C(28)–H(28A)	0.960(14)
C(28)–H(28B)	0.959(13)	C(28)–H(28C)	0.959(14)
C(29)–H(29A)	0.960(13)	C(29)–H(29B)	0.959(11)
C(29)–H(29C)	0.961(13)	C(30)–C(31)	1.50(3)
C(30)–H(30A)	0.959(14)	C(30)–H(30B)	0.959(16)
C(31)–H(31A)	0.961(17)	C(31)–H(31B)	0.960(19)

Intramolecular bond angles (deg.)

C(6)–N(3)–H(3A)	114.9(32)	C(6)–N(3)–H(3B)	113.5(26)
C(6)–N(3)–H(3C)	111.8(27)	H(3A)–N(3)–H(3B)	102.3(45)
H(3A)–N(3)–H(3C)	105.2(42)	H(3B)–N(3)–H(3C)	108.4(37)
C(8)–N(4)–C(11)	123.9(4)	C(8)–N(4)–H(4)	121.8(25)
C(11)–N(4)–H(4)	114.1(25)	N(3)–C(6)–C(8)	106.8(4)
N(3)–C(6)–C(12)	112.1(3)	N(3)–C(6)–H(6)	112.0(21)
C(8)–C(6)–C(12)	110.8(3)	C(8)–C(6)–H(6)	107.8(21)
C(12)–C(6)–H(6)	107.3(23)	O(2)–C(7)–O(5)	125.8(4)
O(2)–C(7)–C(11)	116.7(4)	O(5)–C(7)–C(11)	117.4(5)
O(1)–C(8)–N(4)	124.5(4)	O(1)–C(8)–C(6)	120.1(3)
N(4)–C(8)–C(6)	115.4(4)	C(10)–C(9)–C(12)	118.3(4)
C(10)–C(9)–C(13)	117.6(4)	C(12)–C(9)–C(13)	124.1(4)
C(9)–C(10)–C(17)	118.1(5)	C(9)–C(10)–C(21)	119.6(5)
C(17)–C(10)–C(21)	122.4(4)	N(4)–C(11)–C(7)	106.6(4)
N(4)–C(11)–C(14)	110.3(4)	N(4)–C(11)–H(11)	113.1(20)
C(7)–C(11)–C(14)	110.3(4)	C(7)–C(11)–H(11)	107.4(20)
C(14)– C(11)–H(11)	109.1(20)	C(6)–C(12)–C(9)	119.8(4)
C(6)–C(12)–C(15)	120.3(4)	C(9)–C(12)–C(15)	119.8(4)
C(9)–C(13)–C(22)	122.1(5)	C(9)–C(13)–H(13)	122.8(28)
C(22)–C(13)–H(13)	114.6(29)	C(11)–C(14)–C(19)	121.9(5)
C(11)–C(14)–C(20)	118.8(5)	C(19)–C(14)–C(20)	119.4(5)
C(12)–C(15)–C(16)	120.7(5)	C(12)–C(15)–H(15)	124.3(26)
C(16)–C(15)–H(15)	114.5(27)	C(15)–C(16)–C(21)	120.4(6)
C(15)–C(16)–H(16)	115.9(30)	C(21)–C(16)–H(16)	123.0(29)
C(10)–C(17)–C(24)	122.2(5)	C(10)–C(17)–H(17)	111.0(34)
C(24)–C(17)–H(17)	126.8(35)	C(29)–O(18)–C(30)	111.4(9)
C(14)–C(19)–C(26)	121.4(6)	C(14)–C(19)–H(19)	124.3(33)
C(26)–C(19)–H(19)	114.2(33)	C(14)–C(20)–C(23)	119.3(7)
C(14)–C(20)–H(20)	114.0(30)	C(23)–C(20)–H(20)	125.8(31)
C(10)–C(21)–C(16)	121.2(4)	C(10)–C(21)–H(21)	126.1(37)
C(16)–C(21)–H(21)	112.5(37)	C(13)–C(22)–C(24)	120.3(6)
C(13)–C(22)–H(22)	114.8(44)	C(24)–C(22)–H(22)	122.8(44)
C(20)–C(23)–C(25)	120.2(8)	C(20)–C(23)–H(23)	112.7(42)
C(25)–C(23)–H(23)	126.6(42)	C(17)–C(24)–C(22)	119.6(6)
C(17)–C(24)–H(24)	114.8(39)	C(22)–C(24)–H(24)	125.5(39)
C(23)–C(25)–C(26)	121.0(7)	C(23)–C(25)–H(25)	122.5(52)
C(26)–C(25)–H(25)	116.4(53)	C(19)–C(26)–C(25)	118.6(7)
C(19)–C(26)–H(26)	123.7(38)	C(25)–C(26)–H(26)	117.0(38)
C(28)–O(27)–C(31)	119.2(11)	O(27)–C(28)–H(28A)	118.2(12)
O(27)–C(28)–H(28B)	106.7(13)	O(27)–C(28)–H(28C)	107.6(11)
H(28A)–C(28)–H(28B)	107.6(13)	H(28A)–C(28)–H(28C)	107.6(14)
H(28B)–C(28)–H(28C)	109.0(13)	O(18)–C(29)–H(29A)	112.1(9)
O(18)–C(29)–H(29B)	109.0(13)	O(18)–C(29)–H(29C)	109.0(9)

H(29A)–C(29)–H(29B)	108.8(10)	H(29A)–C(29)–H(29C)	108.9(15)
H(29B)–C(29)–H(29C)	109.0(11)	O(18)–C(30)–C(31)	109.5(10)
O(18)–C(30)–H(30A)	108.4(17)	O(18)–C(30)–H(30B)	110.5(10)
C(31)–C(30)–H(30A)	106.8(12)	C(31)–C(30)–H(30B)	112.4(18)
H(30A)–C(30)–H(30B)	109.0(12)	O(27)–C(31)–C(30)	107.8(11)
O(27)–C(31)–H(31A)	108.7(20)	O(27)–C(31)–H(31B)	109.8(12)
C(30)–C(31)–H(31A)	108.4(12)	C(30)–C(31)–H(31B)	113.1(21)
H(31A)–C(31)–H(31B)	109.0(13)		

Intramolecular torsion angles (deg.)

H(3A)–N(3)–C(6)–C(8)	−63.5(37)	H(3A)–N(3)–C(6)–C(12)	58.0(37)
H(3A)–N(3)–C(6)–H(6)	178.6(45)	H(3B)–N(3)–C(6)–C(8)	53.7(30)
H(3B)–N(3)–C(6)–C(12)	175.2(30)	H(3B)–N(3)–C(6)–H(6)	−64.1(39)
H(3C)–N(3)–C(6)–C(8)	176.7(29)	H(3C)–N(3)–C(6)–C(12)	−61.8(29)
H(3C)–N(3)–C(6)–H(6)	58.9(38)	C(11)–N(4)–C(8)–O(1)	5.1(4)
C(11)–N(4)–C(8)–C(6)	−175.4(6)	C(8)–N(4)–C(11)–C(7)	155.5(5)
C(8)–N(4)–C(11)–C(14)	−84.8(5)	C(8)–N(4)–C(11)–H(11)	37.6(23)
H(4)–N(4)–C(8)–O(1)	178.5(31)	H(4)–N(4)–C(8)–C(6)	−2.0(31)
H(4)–N(4)–C(11)–C(7)	−18.4(29)	H(4)–N(4)–C(11)–C(14)	101.3(29)
H(4)–N(4)–C(11)–H(11)	−136.3(37)	N(3)–C(6)–C(8)–O(1)	27.6(4)
N(3)–C(6)–C(8)–N(4)	−151.9(5)	N(3)–C(6)–C(12)–C(9)	154.9(5)
N(3)–C(6)–C(12)–C(15)	−28.2(4)	C(12)–C(6)–C(8)–O(1)	−94.7(5)
C(12)–C(6)–C(8)–N(4)	85.8(4)	C(8)–C(6)–C(12)–C(9)	−85.8(4)
C(8)–C(6)–C(12)–C(15)	91.0(5)	H(6)–C(6)–C(8)–O(1)	148.2(25)
H(6)–C(6)–C(8)–N(4)	−31.3(25)	H(6)–C(6)–C(12)–C(9)	31.5(25)
H(6)–C(6)–C(12)–C(15)	−151.6(25)	O(2)–C(7)–C(11)–N(4)	26.6(4)
O(2)–C(7)–C(11)–C(14)	−93.1(5)	O(2)–C(7)–C(11)–H(11)	148.2(22)
O(5)–C(7)–C(11)–N(4)	−156.4(5)	O(5)–C(7)–C(11)–C(14)	84.0(5)
O(5)–C(7)–C(11)–H(11)	−34.8(22)	C(10)–C(9)–C(12)–C(6)	174.6(6)
C(10)–C(9)–C(12)–C(15)	−2.3(4)	C(12)–C(9)–C(10)–C(17)	−177.7(7)
C(12)–C(9)–C(10)–C(21)	2.8(5)	C(13)–C(9)–C(10)–C(17)	2.2(5)
C(13)–C(9)–C(10)–C(21)	−177.3(7)	C(10)–C(9)–C(13)–C(22)	−3.3(5)
C(10)–C(9)–C(13)–H(13)	169.2(33)	C(13)–C(9)–C(12)–C(6)	−5.3(4)
C(13)–C(9)–C(12)–C(15)	177.8(7)	C(12)–C(9)–C(13)–C(22)	176.6(8)
C(12)–C(9)–C(13)–H(13)	−10.9(32)	C(9)–C(10)–C(17)–C(24)	0.0(6)
C(9)–C(10)–C(17)–H(17)	−179.2(35)	C(9)–C(10)–C(21)–C(16)	−0.8(5)
C(9)–C(10)–C(21)–H(21)	−176.2(43)	C(17)–C(10)–C(21)–C(16)	179.8(8)
C(21)–C(10)–C(17)–C(24)	179.5(9)	C(21)–C(10)–C(17)–H(17)	0.2(35)
C(17)–C(10)–C(21)–H(21)	4.3(42)	N(4)–C(11)–C(14)–C(19)	−90.8(6)
N(4)–C(11)–C(14)–C(20)	88.1(5)	C(7)–C(11)–C(14)–C(19)	26.7(5)
C(7)–C(11)–C(14)–C(20)	−154.4(6)	H(11)–C(11)–C(14)–C(19)	144.5(23)
H(11)–C(11)–C(14)–C(20)	−36.7(23)	C(6)–C(12)–C(15)–C(16)	−177.1(7)
C(6)–C(12)–C(15)–H(15)	−5.9(33)	C(9)–C(12)–C(15)–C(16)	−0.3(5)
C(9)–C(12)–C(15)–H(15)	171.0(34)	C(9)–C(13)–C(22)–C(24)	2.1(6)
C(9)–C(13)–C(22)–H(22)	166.3(52)	H(13)–C(13)–C(22)–C(24)	−171.0(31)
H(13)–C(13)–C(22)–H(22)	−6.8(59)	C(11)–C(14)–C(19)–C(26)	177.2(9)
C(11)–C(14)–C(19)–H(19)	−0.1(42)	C(11)–C(14)–C(20)–C(23)	−175.9(8)
C(11)–C(14)–C(20)–H(20)	14.3(34)	C(19)–C(14)–C(20)–C(23)	3.0(6)
C(20)–C(14)–C(19)–C(26)	−1.6(6)	C(20)–C(14)–C(19)–H(19)	−179.0(42)
C(19)–C(14)–C(20)–H(20)	−166.8(35)	C(12)–C(15)–C(16)–C(21)	2.4(5)
C(12)–C(15)–C(16)–H(16)	173.3(35)	H(15)–C(15)–C(16)–C(21)	−169.7(31)
H(15)–C(15)–C(16)–H(16)	1.2(46)	C(15)–C(16)–C(21)–C(10)	−1.8(5)
C(15)–C(16)–C(21)–H(21)	174.2(38)	H(16)–C(16)–C(21)–C(10)	−172.0(38)
H(16)–C(16)–C(21)–H(21)	4.0(52)	C(10)–C(17)–C(24)–C(22)	−1.3(6)
C(10)–C(17)–C(24)–H(24)	−177.9(44)	H(17)–C(17)–C(24)–C(22)	177.8(42)

H(17)–C(17)–C(24)–H(24)	1.2(59)	C(29)–O(18)–C(30)–C(31)	167.0(15)
C(29)–O(18)–C(30)–H(30A)	50.8(12)	C(29)–O(18)–C(30)–H(30B)	−68.7(13)
C(30)–O(18)–C(29)–H(29A)	178.6(14)	C(30)–O(18)–C(29)–H(29B)	58.1(11)
C(30)–O(18)–C(29)–H(29C)	−60.8(12)	C(14)–C(19)–C(26)–C(25)	−1.1(7)
C(14)–C(19)–C(26)–H(26)	−170.7(47)	H(19)–C(19)–C(26)–C(25)	176.5(39)
H(19)–C(19)–C(26)–H(26)	7.0(59)	C(14)–C(20)–C(23)–C(25)	−1.7(7)
C(14)–C(20)–C(23)–H(23)	−174.0(47)	H(20)–C(20)–C(23)–C(25)	166.8(39)
H(20)–C(20)–C(23)–H(23)	−5.5(60)	C(13)–C(22)–C(24)–C(17)	0.3(6)
C(13)–C(22)–C(24)–H(24)	176.5(49)	H(22)–C(22)–C(24)–C(17)	−162.7(56)
H(22)–C(22)–C(24)–H(24)	13.6(73)	C(20)–C(23)–C(25)–C(26)	−1.1(7)
C(20)–C(23)–C(25)–H(25)	174.9(56)	H(23)–C(23)–C(25)–C(26)	170.1(55)
H(23)–C(23)–C(25)–H(25)	−13.9(76)	C(23)–C(25)–C(26)–C(19)	2.5(7)
C(23)–C(25)–C(26)–H(26)	172.7(44)	H(25)–C(25)–C(26)–C(19)	−173.8(53)
H(25)–C(25)–C(26)–H(26)	−3.5(67)	C(28)–O(27)–C(31)–C(30)	162.8(18)
C(31)–O(27)–C(28)–H(28A)	179.6(19)	C(31)–O(27)–C(28)–H(28B)	58.4(14)
C(31)–O(27)–C(28)–H(28C)	−58.5(14)	C(28)–O(27)–C(31)–H(31A)	45.5(14)
C(28)–O(27)–C(31)–H(31B)	−73.6(16)	O(18)–C(30)–C(31)–O(27)	62.8(13)
O(18)–C(30)–C(31)–H(31A)	−179.6(19)	O(18)–C(30)–C(31)–H(31B)	−58.7(15)
H(30A)–C(30)–C(31)–O(27)	−179.9(18)	H(30A)–C(30)–C(31)–H(31A)	−62.4(16)
H(30A)–C(30)–C(31)–H(31B)	58.6(17)	H(30B)–C(30)–C(31)–O(27)	−60.4(15)
H(30B)–C(30)–C(31)–H(31A)	57.1(16)	H(30B)–C(30)–C(31)–H(31B)	178.1(22)

(*R*)-(1-Naphthyl)glycyl-(*R*)-phenylglycine · $MeO(CH_2CH_2O)_7Me$ complex [(*R*,*R*)-**1** · **2**(7)]

Crystal data:	
Chemical formula	$C_{24}H_{26}O_5N_2$
Formula weight	422.48
Crystal description	Colourless plate
Crystal size/mm	0.15 × 0.08 × 0.06
a/Å	15.817(4)
b/Å	24.182(7)
c/Å	5.610(2)
alpha/deg	90.000(0)
beta/deg	90.000(0)
gamma/deg	90.000(0)
Volume of unit cell/Å^3	2146(1)
Crystal system	Orthorhombic
Space group	$P\,2_12_12_1$ (No. 19)
Z value	4
Density: Dcalc/gcm^{-3}	1.307
Data Collection:	
Diffractometer used	Mac Science MXC 18
Radiation	Cu K-alpha (lambda = 1.54178 Å)
Total reflections measured	2485
Reflection (*hkl*) limits	$0 < h < 19$ $0 < k < 29$ $0 < l < 6$
Unique reflections	2042
Measurement temperature/K	173
Refinement:	
Least squares refinement method	Full matrix
Weight method	Count statistics
Absorption correction	none
Data reduction cut-off	1.50
Maximum theta	70

Hydrogen treatment	refU
Linear absorption coefficient/cm^{-1}	7.532
R	0.106
R_w	0.089
Reflections used in L.S.	1071
L.S. parameters	259
Max shift/esd	0.4402
Average shift/esd	0.0060
Maximum peak in final Fourier map	0.64
Minimum peak in final Fourier map	−0.70
Programs used to solve and refinement	maXus ver. 1.1 (Mac Science)

Fractional atomic coordinates and U(iso)

Atom	*x/a*	*y/b*	*z/c*	*U*(iso)
O(1)	0.6258(3)	0.2464(3)	0.735(1)	0.059(4)
O(2)	0.5405(3)	0.2449(5)	1.050(1)	0.067(5)
O(3)	0.3250(3)	0.2138(2)	0.479(1)	0.050(3)
N(4)	0.1806(4)	0.2277(3)	0.726(2)	0.046(4)
N(5)	0.4083(5)	0.2253(3)	0.793(1)	0.046(4)
C(6)	0.3345(5)	0.2288(3)	0.677(2)	0.041(4)
C(7)	0.3078(5)	0.3463(4)	0.985(2)	0.054(5)
C(8)	0.5559(5)	0.2364(4)	0.847(2)	0.049(5)
C(9)	0.2582(5)	0.3156(3)	0.816(2)	0.048(5)
C(10)	0.2583(4)	0.2524(4)	0.828(2)	0.039(5)
C(11)	0.3546(6)	0.3238(4)	1.182(2)	0.057(6)
C(12)	0.3100(6)	0.4070(5)	0.957(2)	0.049(5)
C(13)	0.4855(5)	0.2094(3)	0.688(2)	0.044(5)
C(14)	0.2141(6)	0.3431(4)	0.643(2)	0.060(6)
C(15)	0.4982(6)	0.1457(5)	0.682(2)	0.056(6)
C(16)	0.2125(8)	0.3983(5)	0.621(2)	0.071(7)
C(17)	0.2583(9)	0.4303(4)	0.770(3)	0.083(8)
C(18)	0.3619(8)	0.4364(5)	1.109(3)	0.083(8)
C(19)	0.4039(8)	0.3556(4)	1.331(2)	0.069(7)
C(20)	0.4061(7)	0.4096(5)	1.292(3)	0.081(8)
C(21)	0.5075(8)	0.0310(4)	0.685(3)	0.078(8)
C(22)	0.4725(9)	0.0600(7)	0.510(4)	0.11(1)
C(23)	0.5409(8)	0.1192(6)	0.873(3)	0.087(9)
C(24)	0.4705(8)	0.1177(6)	0.504(3)	0.084(9)
C(25)	0.541(1)	0.0607(4)	0.861(4)	0.12(1)
O(26)	0.178(1)	0.105(1)	0.680(5)	0.29(2)
C(27)	0.243(2)	0.070(1)	0.751(5)	0.51(2)
C(28)	0.135(2)	0.085(2)	0.473(4)	0.26(3)
C(29)	0.283(2)	0.096(3)	0.989(5)	0.08(1)
O(31)	0.239(2)	0.082(3)	1.202(5)	0.32(3)
C(32)	0.149(1)	0.110(3)	0.225(5)	0.61(4)
H(4A)	0.13452	0.24240	0.81750	0.01(2)
H(4B)	0.18192	0.18810	0.73740	0.10(6)
H(4C)	0.17512	0.23800	0.56140	0.05(3)
H(10)	0.26439	0.24107	1.00786	0.02(2)
H(5)	0.411(7)	0.228(5)	0.97(2)	0.00(1)
H(14)	0.191(5)	0.326(3)	0.48(2)	0.00(2)
H(16)	0.18031	0.41628	0.49843	0.12(7)
H(17)	0.25773	0.46973	0.75094	0.07(4)
11(20)	0.43580	0.43203	1.40520	0.01(3)
11(19)	0.43769	0.33878	1.45325	0.03(3)

H(11)	0.35100	0.28458	1.20909	0.05(4)
H(13)	0.489(6)	0.221(3)	0.49(2)	0.01(2)
H(23)	0.56910	0.14034	0.99417	0.10(6)
H(25)	0.56626	0.04138	0.99666	0.3(2)
H(22)	0.44739	0.03977	0.38035	0.07(5)
H(24)	0.449(6)	0.139(4)	0.35(2)	0.00(2)
H(21)	0.519(6)	−0.013(4)	0.65(2)	0.00(2)
H(18)	0.368(5)	0.479(4)	1.09(2)	0.00(2)

Anisotropic thermal parameters

Atom	*U*11	*U*22	*U*33	*U*12	*U*13	*U*23
O(1)	0.042(3)	0.077(4)	0.057(4)	0.006(4)	0.002(3)	0.001(5)
O(2)	0.038(3)	0.128(7)	0.034(3)	0.010(5)	0.010(3)	0.001(5)
O(3)	0.041(3)	0.038(3)	0.072(5)	−0.001(3)	−0.023(3)	0.004(3)
N(4)	0.032(3)	0.056(5)	0.051(5)	−0.004(3)	−0.003(3)	0.010(4)
N(5)	0.045(4)	0.058(4)	0.034(4)	0.007(3)	0.006(3)	−0.010(4)
C(6)	0.042(4)	0.041(4)	0.039(5)	−0.007(3)	−0.012(4)	0.007(4)
C(7)	0.046(5)	0.055(6)	0.061(7)	−0.004(4)	0.027(5)	−0.017(5)
C(8)	0.024(4)	0.052(6)	0.072(7)	0.006(3)	−0.012(4)	0.017(6)
C(9)	0.027(4)	0.039(4)	0.078(7)	−0.012(3)	−0.009(5)	0.002(5)
C(10)	0.031(3)	0.035(4)	0.051(5)	0.021(4)	0.002(4)	0.006(4)
C(11)	0.067(6)	0.054(5)	0.050(6)	0.001(4)	0.003(5)	0.025(5)
C(12)	0.048(5)	0.070(7)	0.029(5)	−0.008(5)	0.009(4)	−0.006(5)
C(13)	0.031(4)	0.039(5)	0.061(6)	0.003(3)	0.006(4)	0.010(5)
C(14)	0.046(5)	0.035(5)	0.10(1)	0.003(4)	−0.011(6)	0.003(6)
C(15)	0.047(5)	0.085(7)	0.035(5)	0.004(5)	−0.003(5)	0.008(6)
C(16)	0.078(7)	0.063(8)	0.073(8)	0.033(6)	−0.008(6)	−0.009(6)
C(17)	0.106(8)	0.020(4)	0.12(1)	0.025(5)	0.053(9)	0.044(6)
C(18)	0.069(8)	0.059(7)	0.12(1)	0.010(6)	0.027(8)	0.002(8)
C(19)	0.097(8)	0.045(5)	0.065(8)	−0.008(5)	−0.033(7)	−0.004(5)
C(20)	0.057(6)	0.078(8)	0.11(1)	−0.010(6)	−0.004(8)	−0.031(9)
C(21)	0.081(8)	0.054(7)	0.010(1)	−0.007(5)	−0.021(8)	−0.017(8)
C(22)	0.08(1)	0.11(1)	0.13(1)	0.001(9)	−0.01(1)	−0.01(1)
C(23)	0.076(7)	0.076(8)	0.11(1)	0.000(6)	−0.035(8)	0.003(9)
C(24)	0.090(9)	0.070(8)	0.09(1)	0.027(7)	−0.019(8)	−0.008(8)
C(25)	0.16(1)	0.017(5)	0.18(2)	−0.004(6)	0.02(1)	0.017(8)

Intramolecular bond lengths (Å)

O(1)–C(8)	1.30(2)	O(2)–C(8)	1.18(2)
O(3)–C(6)	1.18(2)	N(4)–C(10)	1.48(1)
N(5)–C(6)	1.34(2)	N(5)–C(13)	1.41(2)
C(6)–C(10)	1.58(2)	C(7)–C(9)	1.44(2)
C(7)–C(11)	1.44(2)	C(7)–C(12)	1.48(2)
C(8)–C(13)	1.57(2)	C(9)–C(10)	1.53(2)
C(9)–C(14)	1.37(2)	C(11)–C(19)	1.38(2)
C(12)–C(17)	1.45(2)	C(12)–C(18)	1.38(2)
C(13)–C(15)	1.55(2)	C(14)–C(16)	1.34(2)
C(15)–C(23)	1.42(2)	C(15)–C(24)	1.28(2)
C(16)–C(17)	1.35(2)	C(18)–C(20)	1.40(2)
C(19)–C(20)	1.33(2)	C(21)–C(22)	1.33(3)
C(21)–C(25)	1.33(3)	C(22)–C(24)	1.40(3)
C(23)–C(25)	1.42(2)	O(26)–C(27)	1.37(4)

O(26)–C(28)	1.43(4)	C(27)–C(29)	1.60(5)
C(28)–C(32)	1.54(6)	C(29)–O(31)	1.43(5)
O(31)–C(32)	1.58(6)	O(2)–H(4A)	1.691(6)
N(4)–H(4A)	0.960(8)	N(4)–H(4B)	0.960(8)
N(4)–H(4C)	0.960(9)	N(5)–H(5)	1.00(11)
C(10)–H(10)	1.050(9)	C(11)–H(11)	0.96(1)
C(13)–H(13)	1.14(11)	C(14)–H(14)	1.05(9)
C(16)–H(16)	0.96(2)	C(17)–H(17)	0.96(1)
C(18)–H(18)	1.03(9)	C(19)–H(19)	0.96(2)
C(20)–H(20)	0.96(2)	C(21)–H(21)	1.09(11)
C(22)–H(22)	0.96(2)	C(23)–H(23)	0.96(2)
C(24)–H(24)	1.05(10)	C(25)–H(25)	0.98(3)

Intramolecular bond angles (deg.)

C(6)–N(5)–C(13)	124.7(9)	O(3)–C(6)–N(5)	123.3(8)
O(3)–C(6)–C(10)	121.3(8)	N(5)–C(6)–C(10)	115.4(8)
C(9)–C(7)–C(11)	126.2(9)	C(9)–C(7)–C(12)	117.4(9)
C(11)–C(7)–C(12)	116.4(9)	O(1)–C(8)–O(2)	127.8(9)
O(1)–C(8)–C(13)	113.9(10)	O(2)–C(8)–O(2)	118.3(8)
C(7)–C(9)–C(10)	119.3(9)	C(7)–C(9)–C(14)	119.6(8)
C(10)–C(9)–C(14)	121.1(9)	N(4)–C(10)–C(6)	106.3(7)
N(4)–C(10)–C(9)	112.6(7)	C(6)–C(10)–C(9)	109.8(7)
C(7)–C(11)–C(19)	123.2(9)	C(7)–C(12)–C(17)	116.7(9)
C(7)–C(12)–C(18)	117.5(10)	C(17)–C(12)–C(18)	125.8(11)
N(5)–C(13)–C(8)	105.2(8)	N(5)–C(13)–C(15)	113.1(7)
C(8)–C(13)–C(15)	109.5(7)	C(9)–C(14)–C(16)	124.0(11)
C(13)–C(15)–C(23)	119.4(10)	C(13)–C(15)–C(24)	119.8(10)
C(23)–C(15)–C(24)	120.8(12)	C(14)–C(16)–C(17)	120.3(12)
C(12)–C(17)–C(16)	122.0(10)	C(12)–C(18)–C(20)	120.7(11)
C(11)–C(19)–C(20)	117.6(12)	C(18)–C(20)–C(19)	124.4(13)
C(22)–C(21)–C(25)	115.5(13)	C(21)–C(22)–C(24)	123.6(16)
C(15)–C(23)–C(25)	114.4(14)	C(15)–C(24)–C(22)	120.1(14)
C(21)–C(25)–C(23)	125.2(17)	C(27)–O(26)–C(28)	112.8(29)
O(26)–C(27)–C(29)	107.9(28)	O(26)–C(28)–C(32)	121.8(38)
C(27)–C(29)–O(31)	114.4(33)	C(29)–O(31)–C(32)	114.0(36)
C(28)–C(32)–O(31)	91.8(31)	C(8)–O(2)–H(4A)	105.8(6)
C(10)–N(4)–H(4A)	105.9(8)	C(10)–N(4)–H(4B)	111.0(7)
C(10)–N(4)–H(4C)	110.0(7)	H(4A)–N(4)–H(4B)	110.5(8)
H(4A)–N(4)–H(4C)	110.5(7)	H(4B)–N(4)–H(4C)	109.0(8)
C(6)–N(5)–H(5)	121.4(63)	C(13)–N(5)–H(5)	113.2(63)
N(4)–C(10)–H(10)	110.1(7)	C(6)–C(10)–H(10)	110.5(7)
C(9)–C(10)–H(10)	107.5(9)	C(7)–C(11)–H(11)	117.8(10)
C(19)–C(11)–H(11)	119.0(11)	N(5)–C(13)–H(13)	112.0(45)
C(8)–C(13)–H(13)	114.4(45)	C(15)–C(13)–H(13)	102.9(44)
C(9)–C(14)–H(14)	125.8(46)	C(16)–C(14)–H(14)	108.2(47)
C(14)–C(16)–H(16)	121.8(13)	C(17)–C(16)–H(16)	117.9(13)
C(12)–C(17)–H(17)	118.4(13)	C(16)–C(17)–H(17)	119.7(15)
C(12)–C(18)–H(18)	119.4(51)	C(20)–C(18)–H(18)	119.8(51)
C(11)–C(19)–H(19)	120.9(10)	C(20)–C(19)–H(19)	121.5(12)
C(18)–C(20)–H(20)	118.0(12)	C(19)–C(20)– H(20)	117.3(14)
C(22)–C(21)–H(21)	115.5(52)	C(25)–C(21)– H(21)	127.2(52)
C(21)–C(22)–H(22)	117.6(16)	C(24)–C(22)–H(22)	118.8(17)
C(15)–C(23)–H(23)	121.1(13)	C(25)–C(23)–H(23)	124.5(16)
C(15)–C(24)–H(24)	118.7(53)	C(22)–C(24)–H(24)	121.1(54)
C(21)–C(25)–H(25)	118.8(11)	C(23)–C(25)–H(25)	116.0(17)
O(2)–H(4A)–N(4)	166.0(6)		

Intramolecular torsion angles (deg.)

C(13)–N(5)–C(6)–O(3)	7.2(9)	C(6)–N(5)–C(13)–C(8)	155.1(11)
C(13)–N(5)–C(6)–C(10)	−175.1(12)	C(6)–N(5)–C(13)–C(15)	−85.4(10)
O(3)–C(6)–C(10)–N(4)	25.7(9)	O(3)–C(6)–C(10)–C(9)	−96.4(10)
N(5)–C(6)–C(10)–N(4)	−152.0(10)	N(5)–C(6)–C(10)–C(9)	85.9(9)
C(11)–C(7)–C(9)–C(10)	−5.0(9)	C(11)–C(7)–C(9)–C(14)	177.3(16)
C(9)–C(7)–C(11)–C(19)	177.2(17)	C(12)–C(7)–C(9)–C(10)	176.3(13)
C(12)–C(7)–C(9)–C(14)	−1.4(9)	C(9)–C(7)–C(12)–C(17)	3.0(10)
C(9)–C(7)–C(12)–C(18)	−175.9(14)	C(11)–C(7)–C(12)–C(17)	−175.7(14)
C(11)–C(7)–C(12)–C(18)	5.3(10)	C(12)–C(7)–C(11)–C(19)	−4.1(10)
O(1)–C(8)–C(13)–N(5)	−152.9(11)	O(1)–C(8)–C(13)–C(15)	85.3(10)
O(2)–C(8)–C(13)–N(5)	27.5(9)	O(2)–C(8)–C(13)–C(15)	−94.3(11)
C(7)–C(9)–C(10)–N(4)	154.8(12)	C(7)–C(9)–C(10)–C(6)	−87.0(10)
C(7)–C(9)–C(14)–C(16)	−1.3(11)	C(14)–C(9)–C(10)–N(4)	−27.6(9)
C(14)–C(9)–C(10)–C(6)	90.7(11)	C(10)–C(9)–C(14)–C(16)	−179.0(16)
C(7)–C(11)–C(19)–C(20)	1.4(11)	C(7)–C(12)–C(17)–C(16)	−2.3(12)
C(7)–C(12)–C(18)–C(20)	−4.2(11)	C(18)–C(12)–C(17)–C(16)	176.5(20)
C(17)–C(12)–C(18)–C(20)	177.0(20)	N(5)–C(13)–C(15)–C(23)	−91.9(12)
N(5)–C(13)–C(15)–C(24)	88.6(12)	C(8)–C(13)–C(15)–C(23)	25.0(10)
C(8)–C(13)–C(15)–C(24)	−154.5(14)	C(9)–C(14)–C(16)–C(17)	2.2(12)
C(13)–C(15)–C(23)–C(25)	173.4(17)	C(13)–C(15)–C(24)–C(22)	−172.3(19)
C(23)–C(15)–C(24)–C(22)	8.2(13)	C(24)–C(15)–C(23)–C(25)	−7.1(14)
C(14)–C(16)–C(17)–C(18)	−0.2(12)	C(12)–C(18)–C(20)–C(19)	1.5(13)
C(11)–C(19)–C(20)–C(18)	0.0(12)	C(22)–C(21)–C(25)–C(23)	−0.1(16)
C(25)–C(21)–C(22)–C(24)	0.9(16)	C(21)–C(22)–C(24)–C(15)	−5.1(15)
C(15)–C(23)–C(25)–C(21)	3.0(15)	C(28)–O(26)–C(27)–C(29)	−177.3(35)
C(27)–O(26)–C(28)–C(32)	−104.1(42)	O(26)–C(27)–C(29)–O(31)	82.9(37)
C(32)–O(31)–C(29)–C(27)	−69.4(39)	O(31)–C(32)–C(28)–O(26)	73.9(38)
H(4A)–O(2)–C(8)–O(1)	−14.0(9)	H(4A)–O(2)–C(8)–C(13)	165.5(11)
H(4A)–N(4)–C(10)–C(6)	−179.6(9)	H(4A)–N(4)–C(10)–C(9)	−59.3(8)
H(4A)–N(4)–C(10)–H(10)	60.7(8)	H(4B)–N(4)–C(10)–C(6)	60.5(8)
H(4B)–N(4)–C(10)–C(9)	−179.2(10)	H(4B)–N(4)–C(10)–H(10)	−59.2(8)
H(4C)–N(4)–C(10)–C(6)	−60.2(8)	H(4C)–N(4)–C(10)–C(9)	60.1(8)
H(4C)–N(4)–C(10)–H(10)	−179.9(10)	C(6)–N(5)– C(13)–H(13)	30.3(51)
H(5)–N(5)–C(6)–O(3)	−162.0(73)	H(5)–N(5)–C(6)–C(10)	15.7(72)
H(5)–N(5)–C(13)–C(8)	−34.9(67)	H(5)–N(5)–C(13)–C(15)	84.6(68)
H(5)–N(5)–C(13)–H(13)	−159.7(84)	O(3)–C(6)–C(10)–H(10)	145.1(12)
N(5)–C(6)–C(10)–H(10)	−32.6(8)	C(9)–C(7)–C(11)–H(11)	−2.7(10)
C(12)–C(7)–C(11)–H(11)	175.9(15)	O(1)–C(8)–C(13)–H(13)	−29.5(52)
O(2)–C(8)–C(13)–H(13)	150.8(53)	C(7)–C(9)–C(10)–H(10)	33.3(9)
C(7)–C(9)–C(14)–H(14)	160.6(58)	C(14)–C(9)–C(10)–H(10)	−149.0(12)
C(10)–C(9)–C(14)–H(14)	−17.1(57)	C(7)–C(11)–C(19)–H(19)	−175.2(18)
H(11)–C(11)–C(19)–C(20)	−178.6(17)	H(11)–C(11)–C(19)–H(19)	4.8(11)
C(7)–C(12)–C(17)–H(17)	178.8(18)	C(7)–C(12)–C(18)–H(18)	176.1(59)
C(18)–C(12)–C(17)–H(17)	−2.4(12)	C(17)–C(12)–C(18)–H(18)	−2.7(57)
H(13)–C(13)–C(15)–C(23)	147.1(49)	H(13)–C(13)–C(15)–C(24)	−32.5(49)
C(9)–C(14)–C(16)–H(16)	−179.3(20)	H(14)–C(14)–C(16)–C(17)	−162.4(51)
H(14)–C(14)–C(16)–H(16)	16.1(49)	C(13)–C(15)–C(23)–H(23)	−9.1(12)
C(13)–C(15)–C(24)–H(24)	10.2(59)	C(24)–C(15)–C(23)–H(23)	170.4(20)
C(23)–C(15)–C(24)–H(24)	−169.3(61)	C(14)–C(16)–C(17)–H(17)	178.7(21)
H(16)–C(16)–C(17)–C(12)	−178.8(20)	H(16)–C(16)–C(17)–H(17)	0.1(13)
C(12)–C(18)–C(20)–H(20)	−172.3(21)	H(18)–C(18)–C(20)–C(19)	−178.8(60)
H(18)–C(18)–C(20)–H(20)	7.4(58)	C(11)–C(19)–C(20)–H(20)	173.9(19)
H(19)–C(19)–C(20)–C(18)	176.6(22)	H(19)–C(19)–C(20)–H(20)	−9.5(13)
C(22)–C(21)–C(25)–H(25)	178.9(25)	C(25)–C(21)–C(22)–H(22)	−179.2(24)
H(21)–C(21)–C(22)–C(24)	−164.8(63)	H(21)–C(21)–C(22)–H(22)	15.0(60)
H(21)–C(21)–C(25)–C(23)	163.6(71)	H(21)–C(21)–C(25)–H(25)	−17.4(68)

C(21)–C(22)–C(24)–H(24)	172.3(64)	H(22)–C(22)–C(24)–C(15)	175.0(25)
H(22)–C(22)–C(24)–H(24)	−7.5(61)	C(15)–C(23)–C(25)–H(25)	−176.0(22)
H(23)–C(23)–C(25)–C(21)	−174.4(28)	H(23)–C(23)–C(25)–H(25)	6.5(15)
O(2)–H(4A)–N(4)–C(10)	−162.2(25)		

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