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The use of the Mitsunobu reaction in preparation of chiral synthons for macrocyclic frameworks

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Abstract

The Mitsunobu reaction was used for the synthesis of chiral diesters **2** and **6** and chiral diamine **4**. Five new chiral macrocyclic bisamides were synthesized, starting from these precursors.

1. Introduction

The increasing interest in the molecular recognition and complexing properties of naturally occurring macrocycles has attracted much attention in the design and synthesis of new cyclic polyaza- and polyoxamacrocycles.[1] The cavity size and rigidity of a macrocycle are important in governing the host–guest interactions and in the complexation selectivity of metal ions.[2] Among others, macrocyclic amides are used as host molecules. They act as hydrogen-bond donors and hydrogen-bond acceptors and can complex neutral molecules of biological interest.[3] The most popular method for preparation of macrocyclic amides consists in reacting a diacid dichloride with a diamine under high-dilution conditions.[4] At the beginning of the nineties we found[5] that α,ω-diamino aliphatic ethers react under ambient conditions with dimethyl α,ω-dicarboxylates, to afford the macrocyclic bisamides which can be readily transformed into diamines using, for example, $BH_3 \cdot Me_2S$. The optimum reaction conditions proposed by us are as follows: methanol as a solvent, concentration ~0.1 M, room temperature, seven days. Similar conditions have been used recently for preparing several various types of macrocyclic amides.[6,7]

The above-mentioned reaction requires α,ω-diamines and dimethyl esters of α,ω-dicarboxylic acids possessing ether oxygen atoms. More complex compounds of this type are not always readily available. The alkylation of alcohols or phenols with functionalized alkyl halides is frequently used as the most

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versatile procedure for the synthesis of these compounds.[7,8] However, in the case of more elaborate compounds this method failed sometimes.[9] The use of the triphenylphosphine/diethyl azadicarboxylate (TPP/DEAD) protocol, known as the Mitsunobu reaction,[10] for coupling phenols and alcohols to form ethers, has been recently widely used in the synthesis of macrocyclic lactones,[9] dendrons,[11] 2,2′-BINOL analogs,[12] etc. Conversion of alcohols into amines is accomplished by coupling an alcohol with phthalimide using TPP/DEAD, followed by hydrazinolysis of the phthalimide product to the desired amine.[13] Since the Mitsunobu reaction is compatibile with a large variety of different substituents it might be an attractive alternative to other common procedures.[14]

2. Results and discussion

Treatment of an alcohol with TPP/DEAD results in the formation of a reactive triphenylphosphonium intermediate which is displaced by the conjugate base of the phenol in an S_N2 type reaction. The advantage of this procedure is that transformation of the alcohol into a leaving group and its nucleophilic substitution are performed in a single reaction step. Moreover, it is usually a mild and stereochemically clean S_N2 reaction which leads to products in generally high yields. Keeping in mind these advantages, we resolved to utilize the Mitsunobu reaction in the preparation of a few chiral, interesting substrates suitable for the preparation of elaborated chiral benzophanes. In this work we would like to show that it is possible to synthesize interesting ethers as well as amines, from cheap chiral sources, using the Mitsunobu reaction which seems to be the most efficient way to form the ether linkage. Next, we decided to describe, following the procedure adapted previously, a few examples of the synthesis of chiral macrocyclic bisamides from diesters **2** and **6** and diamine **4**. We have choosen as a major substrate (*S*,*S*)-2,3-*O*-benzylidenethreitol **1**, readily available from L-tartaric acid.[15,16] Thus, the reaction of methyl salicylate with diol **1**, carried out under Mitsunobu conditions, provided chiral ester **2** in 25% yield (Scheme 1). This low yield seems to be the result of the formation of 3,4-*O*-benzylidenetetrahydrofuran as a by-product. It is well known that such intramolecular coupling of diols caused by the TPP/DEAD system leads to 3- to 7-membered cyclic ethers.[17]

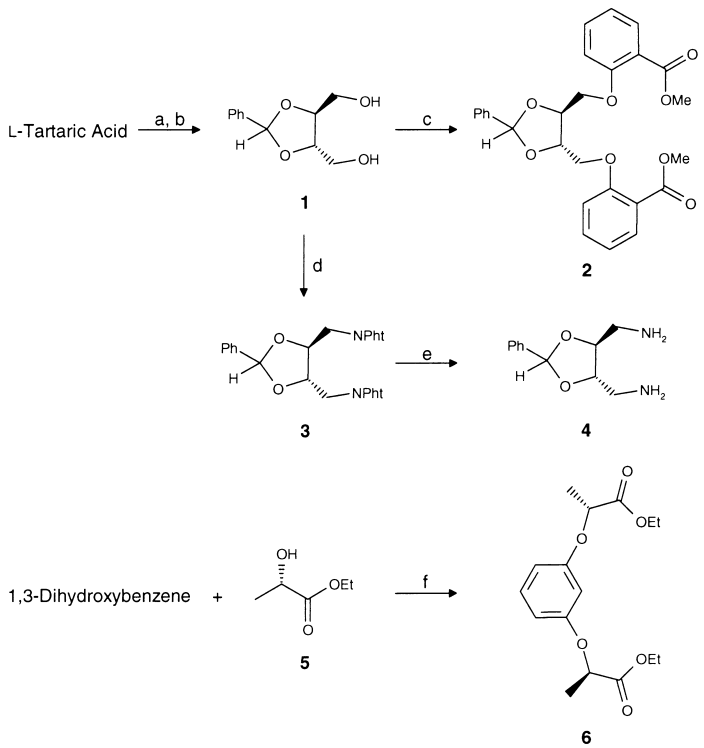
The same diol **1** was converted into diamine **4**. The Mitsunobu reaction, followed by exposure of the resulting intermediate **3** to hydrazine hydrate, resulted in formation of the chiral diamine **4** in an overall yield of 58% (Scheme 1).

Additionally we resolved to use our approach to synthesize another chiral α,ω-diester containing an aromatic nucleus. Thus, the reaction of 1,3-dihydroxybenzene with ethyl L-lactate **5** performed under Mitsunobu conditions afforded, in a convergent route, pure diastereoisomeric ester **6** in a yield of 53%, as a result of inversion of configuration of the starting ester **5** (Scheme 1). All attempts to carry out the analogous synthesis from catechol failed.

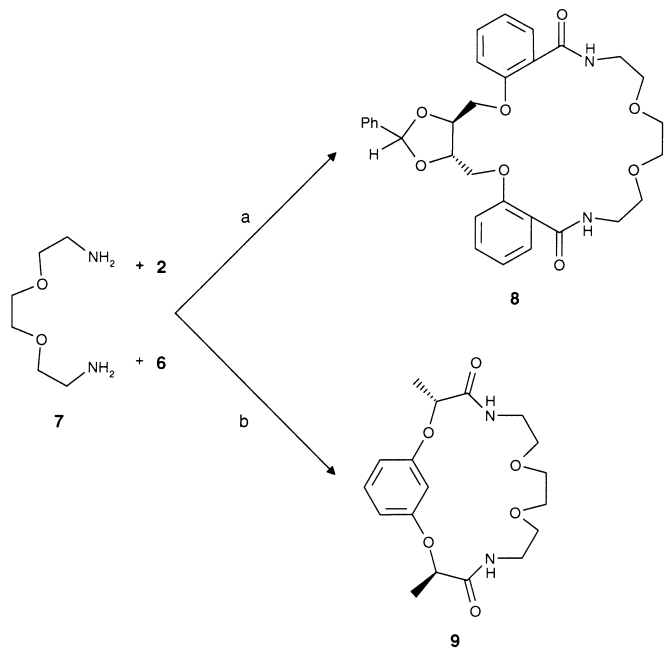
Next we used these three chiral compounds (**2**, **4** and **6**) as substrates in the macrocyclization reaction. The reaction of diester **2** with amine **7** failed under standard conditions. After eight weeks we recovered 100% of ester **2**. In the light of these results, we resolved to modify the reaction conditions using the high-pressure technique.[5b,18] Under high-pressure conditions the synthesis of bisamide **8** was performed in a yield of 39% (Scheme 2).

In the synthesis of macrocycle **9**, we applied conditions that were developed by us for reactions of various di-*tert*-butyl esters with amine **7** affording macrocyclic bisamides.[19] Treatment of ester **6** with amine **7** in the presence of DBU as a transesterification catalyst provided, via the appropriate dimethyl ester, macrocyclic bisamide **9** in a yield of 20% (Scheme 2).[7]

Considering the rigid structure of diamine **4**, we expected that the results of the macrocyclization



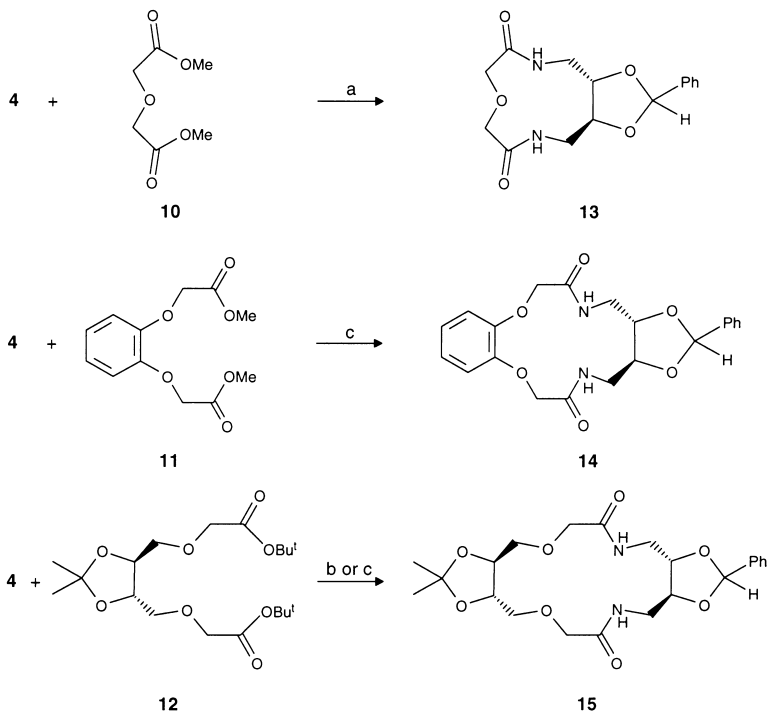
Scheme 1. (a) Lit.[5]; (b) lit.[6]; (c) methyl salicylate, DEAD, PPh_3, THF; (d) phthalimide, DEAD, PPh_3, THF; (e) $H_2N–NH_2 \cdot H_2O$, Pr^iOH, reflux; (f) DEAD, PPh_3, THF



Scheme 2. (a) MeOH, RT, 8 kbar; (b) MeOH, DBU, RT

would depend on the length of the diester and its rigidity. Reactions of the diamine with some selected diesters afforded the expected macrocyclic bisamides in moderate to good yields.

Condensation of diamine **4** with ester **10** yielded bisamide **13** in a yield of 32% (Scheme 3). The reaction of diamine **4** with ester **11**, a compound readily available from catechol,[7] provided macrocyclic bisamide **14** in 20% yield (Scheme 3). Finally, we resolved to use chiral diamine **4** and chiral ester **12** for preparation of macrocyclic bisamide **15** possessing stereogenic centers derived from both substrates. We carried out experiments with chiral *tert*-butyl ester **12**, obtained by us previously,[19] under two different conditions, i.e. under high pressure and in the presence of DBU[19] (Scheme 3). Under high-pressure conditions, the yield is five times lower than under DBU conditions (10% and 49%, respectively). This suggests that substrates **4** and **12** are strongly adapted spaciously and that an influence of high pressure on the reaction of diamines with diesters is highly sophisticated.



Scheme 3. (a) MeOH, RT; (b) MeOH, DBU, RT; (c) MeOH, RT, 12 kbar

We proved that the Mitsunobu reaction is very useful in the preparation of chiral α,ω-diesters and α,ω-diamines. Additionally, we showed that these compounds can be transformed into various chiral benzophanes with good yields.

3. Experimental

3.1. General methods

Melting points were taken on a Köfler type (Boetius) hot stage apparatus and are not corrected. Optical rotations were recorded using a JASCO DIP-360 polarimeter with a thermally jacketed 10 cm cell. ^{1}H NMR spectra were recorded with a Varian Gemini (200 MHz) and/or a Bruker AM500 (500 MHz) spectrometer in $CDCl_3$ or DMSO-d_6 using TMS as an internal standard. ^{13}C NMR spectra were also recorded using a Varian Gemini (50 MHz) and/or a Bruker AM500 (125 MHz) spectrometer. All chemical shifts are quoted in parts per million relative to tetramethylsilane (δ, 0.00 ppm), and coupling constants

(*J*) are measured in hertz. High-resolution mass spectrometry (HRMS) experiments were performed on an AMD-604 Intectra instrument using the electron impact (EI) technique. Column chromatography was carried out on silica gel (Kieselgel-60, 200–400 mesh). α,ω-Diamine **7** was purchased from Fluka. Diol **1**[15,16] and esters **10**,[20] **11**[7] and **12**[19] were prepared according to the literature procedures.

*3.2. Methyl 2-[((4S,5S)-5-{[2-(methyloxycarbonyl)phenoxy]methyl}-2-phenyl-1,3-dioxolan-4-ylo) methoxy] benzoate **2***

Diethyl azodicarboxylate (4.5 mL, 29 mmol) was added at 0°C to a solution of diol **1** (3.0 g, 14.3 mmol), methyl salicylate (3.75 mL, 29 mmol) and triphenylphosphine (7.87 g, 30 mmol) in 50 mL anhydrous THF. The mixture was stirred for 48 h at room temperature. Then the solvent was evaporated and the residue was chromatographed on a silica gel column using an *n*-hexane/ethyl acetate system as an eluent to afford diester **2** (1.71 g, 25%) as a thick, colourless oil: $[\alpha]_D^{23}$=+53.3 (c 1.0, $CHCl_3$); δ_H (200 MHz, $CDCl_3$): 7.81 (m, 2H); 7.6–7.3 (m, 7H); 7.1–6.9 (m, 4H); 6.22 (s, 1H); 4.9–4.6 (m, 2H); 4.5–4.3 (m, 4H); 3.86 (s, 3H); 3.78 (s, 3H); δ_C (50 MHz, $CDCl_3$): 166.5; 166.4; 158.0; 137.2; 133.5; 133.4; 131.8; 131.6; 129.5; 128.3; 126.8; 120.8; 120.7; 120.4; 120.3; 113.3; 113.1; 104.6; 77.6; 76.6; 69.0; 68.8; 51.9; 51.8; HRMS calcd for $C_{27}H_{26}O_8$ $(M)^+$ 478.1628, found 478.1625.

*3.3. 2-({(4S,5S)-5-[(1,3-Dioxo-2,3-dihydro-1H-2-isoindolyl)methyl]-2-phenyl-1,3-dioxolan-4-yl} methyl-1,3-isoindolinodione **3***

Diethyl azodicarboxylate (5.3 mL, 34 mmol) was added at 0°C to a solution of diol **1** (3.0 g, 14.3 mmol), phthalimide (5.0 g, 34 mmol) and triphenylphosphine (9.0 g, 34 mmol) in 100 mL anhydrous THF. The mixture was stirred for 48 h at room temperature, the precipitated product was isolated by filtration and washed with cold THF. After removing the solvent from the fitrate by evaporation, additional product was isolated which had to be recrystallized from methanol. The combined crystals were chromatographed on a silica gel column using an *n*-hexane/ethyl acetate system as an eluent, to afford compound **3** (3.7 g, 61%) as colourless crystals: $[\alpha]_D^{23}$=−46.6 (c 1.0, DMSO); mp 200–201°C; δ_H (500 MHz, $CDCl_3$): 7.79 (m, 4H); 7.68 (m, 4H); 7.48 (m, 2H); 7.4–7.2 (m, 3H); 6.08 (s, 1H); 4.53 (m, 1H); 4.45 (m, 1H); 4.2–3.8 (m, 4H); δ_C (125 MHz, $CDCl_3$): 168.1; 168.0; 136.5; 134.0; 133.9; 131.9; 131.8; 129.4; 128.3; 126.7; 123.4; 103.5; 77.1; 77.0; 40.6; 38.9; HRMS calcd for $C_{27}H_{20}N_2O_6$ $(M)^+$ 468.1321, found 468.1324.

*3.4. [(4S,5S)-5-(Aminomethyl)-2-phenyl-1,3-dioxolan-4-yl]-methanamine **4***

Hydrazine hydrate (3.94 mL, 126 mmol) was added to a stirred solution of compound **3** (3.7 g, 7.9 mmol), and the solution was heated to reflux for eight hours. It was then cooled to room temperature, and the solvent was removed under reduced pressure. The residue was dissolved in NaOH (30 mL, 20%), and the resulting two layers were then extracted with CH_2Cl_2 (3×50 mL). The combined organic solution was then dried (Na_2SO_4). The filtrate was evaporated to leave the diamine as a pale yellow oil. Crude product was then distilled under reduced pressure in a Kugelrohr apparatus (120°C, 0.2 mmHg) to afford the diamine **4** (1.56 g, 95%) as a colourless oil, which has to be stored under argon because CO_2 from the air is strongly absorbed: δ_H (200 MHz, $CDCl_3$): 7.6–7.3 (m, 5H); 5.94 (s, 1H); 4.0–3.9 (m, 2H); 3.1–2.8 (m, 4H); 1.6–1.2 (bs, 4H); δ_C (50 MHz, $CDCl_3$): 137.5; 129.3; 128.2; 126.5; 103.0; 81.6; 80.7; 44.1; 43.9; HRMS calcd for $C_{11}H_{17}N_2O_2$ $(M+H)^+$ 209.1290, found 209.1292.

3.5. *Ethyl (2*S*)-2-(3-{[(1*S*)-1-(ethyloxycarbonyl)ethyl]oxy}phenoxy) propanoate* **6**

Diethyl azodicarboxylate (3.1 mL, 19.7 mmol) was added at 0°C to a solution of 1,3-dihydroxybenzene (1.0 g, 9.1 mmol), ethyl L-lactate (2.1 mL, 18.4 mmol) and triphenylphosphine (5.0 g, 19.1 mmol) in 20 mL anhydrous THF. The mixture was stirred for 48 h at room temperature. Then the solvent was evaporated and the residue was chromatographed on a silica gel column using an *n*-hexane/ethyl acetate system as an eluent, to afford diester **6** (1.48 g, 53%, >97% d.e. from ^{1}H NMR) as a thick, colourless oil: $[\alpha]_D^{23}$=+40.9 (c 1.5, $CHCl_3$); δ_H (200 MHz, $CDCl_3$): 7.2–7.0 (m, 1H); 6.6–6.3 (m, 3H); 4.70 (q, *J*=6.8, 2H); 4.22 (q, *J*=7.1, 4H); 1.59 (d, *J*=6.8, 6H); 1.25 (t, *J*=7.1, 6H); δ_C (50 MHz, $CDCl_3$): 172.0; 158.7; 129.9; 108.0; 103.0; 72.6; 61.2; 18.4; 14.0; HRMS calcd for $C_{16}H_{22}O_6$ $(M)^+$ 310.1416, found 310.1424.

3.6. *General procedures for the synthesis of macrocyclic bisamides*

Method A (standard conditions): An equimolar 0.1 M methanolic solution (1.5 mmol) of α,ω-diamine and dimethyl α,ω-dicarboxylate was left at ambient temperature over a period of 7 days. Then the solvent was evaporated and the residue was chromatographed on a silica gel column using 0–3% mixtures of methanol in chloroform.

Method B (under high pressure): A Teflon ampoule was filled with an equimolar solution of the dimethyl α,ω-dicarboxylate (0.5 mmol) and the appropriate α,ω-diamine (0.5 mmol) in 5 mL of methanol and was placed in a high-pressure vessel filled with ligroine as a transmission medium and compressed (12 kbar) at room temperature for 48 h. After decompression, the reaction mixture was transferred quantitatively to a round-bottomed flask and the solvent was evaporated. The residue was chromatographed on a silica gel column using 0–3% mixtures of methanol in chloroform.

Method C (with DBU): An equimolar 0.1 M methanolic solution (1.5 mmol) of α,ω-diamine and dimethyl α,ω-dicarboxylate containing DBU (20 mol%) was left at ambient temperature over a period of 21 days. Then the solvent was evaporated and the residue was chromatographed on a silica gel column using 0–3% mixtures of methanol in chloroform.

3.7. *(22a*S*,25a*S*)-24-Phenyl-5,6,7,8,10,11,13,14,15,16,22,22a,25,26-tetrahydrodibenzo-[*i,q*][1,3] dioxolo[4,5-*m*][1,4,11,16,7,20]tetraoxadiazacyclodocosine-5,16-dione* **8**

Method B, yield 39%; $[\alpha]_D^{23}$=+3.2 (c 1.0, $CHCl_3$); δ_H (500 MHz, $CDCl_3$): 8.28 (m, 2H); 8.19 (m, 2H); 7.5–7.4 (m, 4H); 7.37 (m, 3H); 7.2–6.9 (m, 4H); 6.16 (s, 1H); 4.73 (m, 2H); 4.40 (m, 4H); 3.7–3.3 (m, 12H); δ_C (125 MHz, $CDCl_3$): 165.0; 164.9; 156.3; 156.2; 136.7; 132.7; 132.3; 132.2; 129.7; 128.4; 126.3; 122.7; 122.4; 122.3; 122.2; 113.2; 113.0; 104.3; 76.9; 76.7; 70.1; 69.9; 69.4; 69.2; 69.1; 69.0; 39.7; 39.3; HRMS calcd for $C_{31}H_{34}N_2O_8$ $(M)^+$ 562.2315, found 562.2310.

3.8. *(3*S*,16*S*)-3,16-Dimethyl-2,8,11,17-tetraoxa-5,14-diazabicyclo[16.3.1]docosa-1(21),18(22),19-triene-4,15-dione* **9**

Method C, yield 20%; δ_H (500 MHz, $CDCl_3$): 7.15 (m, 1H); 6.7–6.5 (m, 2H); 6.6–6.4 (m, 3H); 4.58 (q, *J*=6.8, 2H); 3.9–3.0 (m, 12H); 1.60 (d, *J*=6.8, 6H); δ_C (125 MHz, $CDCl_3$): 172.2; 158.5; 130.1; 107.9; 101.9; 75.1; 70.7; 69.7; 38.9; 19.2; HRMS calcd for $C_{18}H_{26}N_2O_6$ $(M)^+$ 366.1791, found 366.1792.

*3.9. (3a*S*,12a*S*)-2-Phenylperhydro[1,3]dioxolo[4,5*f*][1,4,9]-oxadiazacycloundecane-6,10-dione* ***13***

Method A, yield 32%; $[\alpha]_D^{23}$=+65.0 (c 1.0, DMSO); mp 283–285°C; δ_H (500 MHz, DMSO-d_6): 7.95 (t, *J*=6.2, 1H); 7.87 (t, *J*=6.0, 1H); 7.5–7.3 (m, 5H); 5.86 (s, 1H); 4.0–3.9 (m, 6H); 3.6–3.2 (m, 4H); δ_C (125 MHz, DMSO-d_6): 169.3; 169.2; 136.9; 129.4; 128.2; 126.7; 101.9; 79.1; 77.6; 73.3; 73.2; 40.6; 40.2; HRMS calcd for $C_{15}H_{17}N_2O_5$ $(M-H)^+$ 305.1137, found 305.1136.

*3.10. (3a*S*,17a*S*)-2-Phenyl-3a,4,5,6,7,7,14,15,16,17,17*a*-decahydrobenzo[*b*][1,3]-dioxolo[4,5-*i*][1,4,7,12]dioxadiazacyclotetradecane-6,15-dione* ***14***

Method A, yield 20%; $[\alpha]_D^{23}$=+18.9 (c 1.0, DMSO); mp 294–296°C; δ_H (200 MHz, DMSO-d_6): 8.15 (bt, *J*=5.9, 1H); 8.06 (bt, *J*=5.8, 1H); 7.6–7.3 (m, 5H); 7.2–7.0 (m, 4H); 5.95 (s, 1H); 4.6–4.3 (m, 4H); 4.3–4.0 (m, 2H); 3.7–3.2 (m, 4H); δ_C (50 MHz, DMSO-d_6): 168.2; 168.1; 148.6; 136.9; 129.5; 128.3; 126.8; 123.6; 123.4; 118.5; 118.1; 102.1; 79.2; 78.3; 70.7; 70.4; 41.3; 40.1; HRMS calcd for $C_{21}H_{22}N_2O_6$ $(M)^+$ 398.1472, found 398.1472.

*3.11. (3a*S*,9a*S*,12a*S*,18a*S*)-2-Phenyl-11,11-dimethylperhydrodi[1,3]-dioxolo[4,5-*f*:4,5-*n*][1,12,4,9]-dioxa-diazacyclohexadecane-7,15-dione* ***15***

Method B, yield 10%; method C, yield 49%; $[\alpha]_D^{23}$=+42.4 (c 1.05, DMSO); mp 239–240°C; δ_H (500 MHz, DMSO-d_6): 7.93–7.83 (q, *J*=6.2, 2H); 7.5–7.3 (m, 5H); 5.93 (s, 1H); 4.14 (m, 1H); 4.1–3.8 (m, 8H); 3.60 (m, 4H); 3.48 (m, 1H); 3.36 (m, 1H); 3.20 (m, 1H); 1.31 (s, 6H); δ_C (125 MHz, DMSO-d_6): 169.3; 169.2; 138.0; 129.3; 128.3; 126.6; 108.7; 102.4; 78.6; 78.5; 76.2; 71.3; 71.2; 69.8; 69.7; 40.9; 39.1; 26.9; HRMS calcd for $C_{22}H_{30}N_2O_8$ $(M)^+$ 450.2002, found 450.2000.

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