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Asymmetric Catalysis, Part 91 [1]: Enantioselective Monophenylation of *cis*-1,2-Cyclopentanediol with Triphenylbismuth Diacetate and Chiral Copper(II) Complexes as Catalysts

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Summary. The monophenylation of *cis*-1,2-cyclopentanediol with triphenylbismuth diacetate in the presence of chiral Cu(II) complexes as catalysts gave *cis*-2-hydroxy-1-phenoxy-cyclopentane with enantiomeric excesses up to 38%. The optically active ligands used were triamine derivatives of 2,6-*bis*(aminomethyl)pyridine and diamine derivatives of 2-(aminomethyl)pyridine. Selectivity in the monophenylation occurred only in the presence of the latter as auxiliary ligands.

Keywords. Enantioselective catalysis; Chiral Cu(II) complexes; Monophenylation; Triphenylbismuth diacetate; *cis*-1,2-Cyclopentanediol.

Asymmetrische Katalysen, 91. Mitt. [1]: Enantioselektive Monophenylierung von cis-1,2-Cyclopentandiol mit Triphenylwismutdiacetat und chiralen Kupfer(II)-Komplexen als Katalysatoren

Zusammenfassung. Die Monophenylierung von *cis*-1,2-Cyclopentandiol mit Triphenylwismutdiacetat in Gegenwart chiraler Cu(II)-Komplexe als Katalysatoren ergab *cis*-2-Hydroxy-1-phenoxy-cyclopentan mit Enantiomerenüberschüssen von bis zu 38%. Die eingesetzten optisch aktiven Liganden waren Triamin-Derivate von 2,6-*bis*(Aminomethyl)pyridin und Diamin-Derivate von 2-(Aminomethyl)pyridin. Selektivität bei der Monophenylierung war nur in Gegenwart letzterer als Auxiliar-Liganden zu beobachten.

Introduction

The use of aryl bismuth(V) reagents in the arylation of organic substrates has been increasingly reported, particularly by *Barton et al.* [2-12]. Most of the reactions were carried out catalytically, using traces of copper metal or copper salts.

The phenylation of diols with triphenylbismuth diacetate has been reported by David and Thieffry [13–15], who showed that 1,n-diols (n = 2-6) in refluxing CH₂Cl₂ could be converted into their monophenyl ethers in good yields without any diphenylated byproducts. Here again the catalytic effect of copper has been demonstrated [6], with an increase in the yields already at room temperature, the loss of an induction period, and the possibility of using other solvents.

We are interested in the enantioselective monophenylation of prochiral diol substrates using optically active Cu(II) complexes as catalysts. In previous communications [16–17], we reported relatively high optical inductions in the phenylation of several *meso*-1,2-diols using optically active pyridinyloxazolines as auxiliary ligands.

In this paper, we present new results obtained in the monophenylation of cis-1,2-cyclopentanediol (1), which gave the best optical inductions in our previous studies (Scheme 1), using a series of optically active diamines and triamines for the chiral modification of the Cu(II) catalysts.



Results and Discussion

Two types of optically active ligands were used in this work: a) C_2 -symmetric triamines possessing the general framework of 2,6-bis(amino-methyl)pyridine (Scheme 2).



Scheme 2

- "CH-'nн 9a, R = H 10a, R = H 2-{N-(1*S*)-(1-Cyclohexylethylamino)methyl]py 8a, R₁ = H, R₂ = methyl methylpyrrolidinyl) 2.IN./25./2.M (.).2.(N.Norer **8b.** $R_1 = phenyl, R_2 = H$ 2-[N-(2*S*)-(2-Diphenylhydroxymethylpyrrolidinyl)methyl]pyridine 9b B = methy 10b. R = methyl (-)-2-(N-EphedrinyImethyl)pyridine 2-{[N-(1S)-1-Cyclohexylethyl-N-mgthylamino]methyl)pyridine нŃ -MCH CH₃ 13 2-(45,55)-(4,5-Diphenylimidazolinyl)pyridine 2-[N-(1R)-(1-Phenylethylamino)methyl]pyridine (+)-2-IN-(3-Aminometi unyi)methyl]pyridin
- b) Diamines with the general framework of 2-(aminomethyl)pyridine (Scheme 3).

Scheme 3

Ligands 8-10 were synthesized by reacting the corresponding optically pure amines with 2-(chloromethyl)pyridine (14, Scheme 4). The appropriate references for the syntheses of the other ligands are given in Table 1.



For the monophenylation of cis-1,2-cyclopentanediol (1) with triphenylbismuth diacetate (2), leading to cis-2-hydroxy-1-phenoxy-cyclopentane (3, Scheme 1), the chiral Cu(II) catalysts were synthesized *in situ* from Cu(OAc)₂·H₂O and ligands 4–13. In addition, the copper(II) perchlorate salts of ligands 4 and 6 were used. For further reaction conditions, see Experimental. Results for these monophenylation catalyses are given in Table 1. Yields were determined after distillation of 3, and the optical induction was measured by gas chromatographic analysis of the isopropylisocyanate derivative of 3. For further analytical details, see Experimental.

From the results given in Table 1, it can be seen that the reaction does not occur in the presence of an excess (even slight) of ligands 4 and 8a (relative to the amount of $Cu(OAc)_2 \cdot H_2O$). For ligands 4, the reaction is only catalyzed when they are used as their [Cu(4)Cl]ClO₄ complexes (Table 1, bottom). With other triamine (5–7) and diamine ligands (8b–13), the monophenylation is catalyzed also with an excess of

Ligand	Ligand synthesis	Mol % Cu(OAc) ₂ ·H ₂ O	Mol % ligand	Chemical yield (%)	Selectivity (% ee)
4 a	[18]	3	10	_	
4b	[18]	3	10	_	_
4c	[18]	3	9	-	
	[18]	3	3	10	
5	[19]	3	10	35	_
6	[20]	3	8	60	_
7	[21-22]	3	10	60	-
8a	this study	3	10	-	_
	this study	4	4.5	_	_
8b	this study	3	10	40	_
9a	this study	3	10	35	4(1R,2S)
9b	this study	3	10	10	5 (1 <i>S</i> ,2 <i>R</i>)
10a	this study	3	10	55	38 (1 <i>R</i> ,2 <i>S</i>)
10b	this study	3	10	20	
11	[23]	3	10	40	12 (1 <i>R</i> ,2 <i>S</i>)
12	[23]	3	11	60	6 (1 <i>R</i> ,2 <i>S</i>)
13	[24]	3	9	50	28 (1 <i>S</i> ,2 <i>S</i>)
Ligand	Complex	Complex	Mol %	Chemical	Selectivity
	synthesis		complex	yield (%)	(% ee)
4a	[18]	$[Cu(4a)Cl]ClO_4 \cdot H_2O$	4	30	_
4b	[18]	[Cu(4b)Cl]ClO ₄	3	60	_
4c	[18]	$[Cu(4c)Cl]ClO_4$	3	60	—
6	[20]	$[Cu(6)H_2O](ClO_4)_2$	4	60	_

Table 1. Monophenylation of cis-1,2-cyclopentanediol (1) with Ph₃Bi(OAc)₂ (2)

the ligands. Nevertheless, the yields are always under 70%, which is the yield obtained for $Cu(OAc)_2 \cdot H_2O$ catalysis without any auxiliary ligand [15].

Since ligands 4 and 8a both contain oxygen as a potential ligand, its coordination could explain the fact that Cu(II) no longer catalyzes the reaction. One may well argue that monophenylation occurred with ligands 8b and 9, which also contain an oxygen atom. Nevertheless, the coordination of the oxygen atom to Cu(II) in these cases would be difficult, due to the presence of one or two bulky phenyl groups at the α -carbon with respect to the oxygen atom.

Table 1 shows that selectivities, if observed, occur only with diamine ligands. The 2,6-*bis*(pyridine)triamine derivatives **5** and **7**, which contain twice the chiral information of the 2-pyridine derivatives **11** and **15a** [25] (Scheme 5), were expected to induce much higher enantioselectivities than the latter. However, diamines **11** and **15a** gave optical inductions in the monophenylation of 12% and 50% [17], respectively, whereas only racemic **3** was formed in the presence of the triamines **5** and **7**.

The comparison between the optical inductions obtained with ligands 10a and 11 shows the effect of a small change in the structure of the ligands on selectivity.

Enantioselective Syntheses with Chiral Cu(II) Catalysts



The optical induction of 12% obtained with 11 can be increased up to 38% with 10a, the only difference being a cyclohexyl vs. a phenyl group on the asymmetric carbon atom (Scheme 3). A similar effect was observed with ligands 15a and 15b, which differ by an isopropyl vs. a phenyl group on the asymmetric carbon atom [17]. The optical induction of 31% obtained with 15b increased up to 50% with 15a.

Experimental

General

Elemental analyses were performed by the Microanalytical Laboratory, University of Regensburg. Optical rotations: Perkin-Elmer 241 polarimeter. ¹H NMR spectra: Bruker ARX 400 or Bruker WM 250. Mass spectra: Varian MAT 311A. Ph₃Bi(OAc)₂ (2) was prepared according to Ref. [17].

Cis-1,2-cyclopentanediol (1)

tert-BuOH (800 ml) was added to a solution of K_2CO_3 (100 g, 0.724 mol) and $K_3[Fe(CN)_6]$ (250 g, 0.759 mol) in H_2O (800 ml). K_2OsO_4 (189 mg, 0.513 mmol) and cinchonine chlorobenzoate (1.14 g, 2.71 mmol) were added to the vigorously stirred mixture. After 30 min cyclopentene (17.0 g, 0.250 mol) was added and the mixture was stirred at room temperature for 20 h. Then, Na₂SO₃ (150 g, 1.19 mol) was added and the mixture was stirred at room temperature for 2 h. tert-BuOH was removed and H_2O was added to dissolve the precipitated salts. The aqueous solution was extracted with ethyl acetate and, after drying over K_2CO_3 , the solvent was evaporated, giving 18.4 g of a yellow liquid. Distillation under vacuum yielded 16.5 g (0.16 mol) of 1 (65%) as a colorless liquid.

¹H NMR (400 MHz, CDCl₃, *TMS*): 1.41–1.53 (m, 1H); 1.58–1.70 (m, 2H); 1.75–1.88 (m, 3H); 3.27 (s, 2H); 3.99 (m, 2H). MS: 102 (M)⁺.

2-[N-(2S)-2-(Methoxymethylpyrrolidinyl)methyl]pyridine (8a)

A solution of the hydrochloride of 2-(chloromethyl)pyridine (14, 1.46 g, 8.88 mmol) in freshly distilled MeOH (30 ml) was added dropwise to a solution of (S)-2-(methoxymethyl)pyrrolidine (0.922 g, 8.00 mmol) in MeOH (30 ml). The mixture was heated under reflux and kept weakly alkaline using a 10% KOH solution in MeOH. When the pink color remained (phenolphthalein as indicator), the mixture was cooled and the solvent was evaporated. The residue was suspended in H₂O (100 ml) and extracted with 3 portions of CH₂Cl₂ (100 ml). The organic phases were combined, dried over MgSO₄ and the solvent was removed to give 1.92 g of a brown liquid. Bulb-to-bulb distillation (oven temp. = 100 °C, $p \approx 0.05$ mm Hg) yielded 1.30 g (6.30 mmol) of ligand **8a** (71%) as a colorless liquid.

Anal.: calc. for $C_{12}H_{18}N_2O$ (206.3): C 69.87, H 8.79, N 13.58; found: C 69.79, H 8.75, N 13.65. $[\alpha]_{589}^{25} = +80 (c = 1, CHCl_3)$. ¹H NMR (400 MHz, CDCl₃ *TMS*): 1.61–1.99 (m, 4H); 2.32 (m, 1H); 2.81 (m, 1H); 2.99 (m, 1H); 3.31–3.45 (m, 2H); 3.33 (s, 3H); 3.61 (d, J = 13.8 Hz, 1H); 4.21 (d, J = 13.8 Hz, 1H); 7.14 (m, 1H); 7.43 (d, J = 7.8 Hz, 1H); 7.64 (m, 1H); 8.54 (d, J = 4.7 Hz, 1H). MS: 207 (MH)⁺.

2-[N-(2S)-(2-Diphenylhydroxymethylpyrrolidinyl)methyl]pyridine (8b)

Ligand **8b** was synthesized as described for **8a** from 1.27 g (7.74 mmol) of **14** and 1.50 g (5.92 mmol) of (*S*)- α , α -diphenyl-*L*-prolinol. After the extraction and the removal of the solvent, 2.40 g of a brown solid was obtained. Crystallization of this solid from a CH₂Cl₂/petrol ether mixture yielded 1.81 g (5.26 mmol) of **8b** (88%) as a fine white powder.

Mp = 118 °C. Anal.: calc. for C₂₃H₂₄N₂O (344.5): C 80.20, H 7.02, N 8.13; found: C 80.10, H 6.94, N 8.10. $[\alpha]_{589}^{25} = +73$ (c = 1, CHCl₃). ¹H NMR (250 MHz, CDCl₃, *TMS*): 1.60–1.70 (m, 2H); 1.72–1.80 (m, 1H); 1.93–2.02 (m, 1H); 2.48–2.55 (m, 1H); 2.95–3.00 (m, 1H); 3.33 (d, J = 13.8 Hz, 1H); 3.37 (d, J = 13.8 Hz, 1H); 4.08 (dd, $J_1 = 9.2$ Hz, $J_2 = 4.9$ Hz, 1H); 4.98 (s, broad, 1H); 7.05–7.31 (m, 8H); 7.55–7.60 (m, 3H); 7.66–7.69 (m, 2H); 8.41–8.43 (m, 1H). MS: 267 (M – 77)⁺.

(-)-2-(N-Norephedrinylmethyl)pyridine (**9a**)

Ligand **9a** was synthesized as described for **8a** from 1.30 g (7.93 mmol) of **14** and 1.79 g (9.53 mmol) of (–)-norephedrine hydrochloride. After the extraction, the solvent was evaporated to give 1.96 g of a brown oil. Bulb-to-bulb distillation yielded two fractions. The first fraction (oven temp. = $150 \,^{\circ}$ C, $p \approx 10 \,\text{mm Hg}$) contained 0.47 g of a mixture of (–)-norephedrine and 2-(chloromethyl)pyridine; the second (oven temp. = $230 \,^{\circ}$ C, $p \approx 0.1 \,\text{mm Hg}$) 0.98 g (4.04 mmol) of **9a** (51%) as a brown oil.

Anal.: calc. for $C_{15}H_{18}N_2O$ (242.3): C 74.35, H 7.49, N 11.56; found: C 71.53, H 7.51, N 10.93. $[\alpha]_{589}^{25} = -72$ ($c \doteq 1$, CHCl₃). ¹H NMR (250 MHz, CDCl₃, *TMS*): 0.99 (d, J = 6.5 Hz, 3H); 2.87 (m, 1H); 3.89 (d, J = 14.3 Hz, 1H); 4.08 (d, J = 14.3 Hz, 1H); 4.36 (d, J = 8.3 Hz, 1H); 4.60 (broad, 2H); 7.16 (m, 1H); 7.22–7.35 (m, 6H); 7.63 (m, 1H); 8.49 (m, 1H). MS: 224 (M - H₂O)⁺⁺.

(-)-2-(N-Ephedrinylmethyl)pyridine (9b)

Ligand **9b** was synthesized as described for **8a** from 1.30 g (7.93 mmol) of **14** and 1.57 g (9.51 mmol) of (–)-ephedrine. After the extraction, the solvent was evaporated to give 2.07 g of a brown oil. Bulb-to-bulb distillation yielded three fractions. The first fraction (oven temp. = $100 \,^{\circ}$ C, $p \approx 0.1 \,\text{mm Hg}$) contained 0.40 g of a mixture of (–)-ephedrine and 2-(chloromethyl)pyridine, the second (oven temp. = $150 \,^{\circ}$ C, $p \approx 0.1 \,\text{mm Hg}$) 0.61 g (3.70 mmol) of solid (–)-ephedrine, and the third (oven temp. = $230 \,^{\circ}$ C, $p \approx 0.1 \,\text{mm Hg}$) 0.80 g (3.13 mmol) of **9b** (40%) as a brown oil.

Anal.: calc. for $C_{16}H_{20}N_2O$ (256.4): C 74.97, H 7.86, N 10.93; found: C 74.43, H 8.13, N 10.56. [α]²⁵₅₈₉ = -45 (c = 1, CHCl₃). ¹H NMR (250 MHz, CDCl₃, *TMS*): 1.01 (d, J = 6.8 Hz, 3H); 1.26 (s, 1H); 2.96 (m, 1H); 3.74 (d, J = 14.7 Hz, 1H); 3.90 (d, J = 14.7 Hz, 1H); 4.25 (broad, 1H); 4.90 (d, J = 4.3 Hz, 1H); 7.11–7.37 (m, 6H); 7.59 (m, 1H); 8.49 (m, 1H). MS: 238 (M – H₂O)⁺.

2-[N-(1S)-(1-Cyclohexylethylamino)methyl]pyridine (10a)

Ligand 10a was synthesized as described for 8a from 1.30 g (7.93 mmol) of 14 and 1.21 g (9.53 mmol) of (S)-(+)-1-cyclohexylethylamine. After the extraction, the solvent was removed to give 1.67 g of a brown oil. Bulb-to-bulb distillation yielded two fractions. The first fraction (oven temp. = $150 \,^{\circ}$ C, $p \approx 10 \,\text{mm}$ Hg) contained 0.19 g (1.50 mmol) of (S)-(+)-1-cyclohexylethylamine, the second (oven temp. = $150 \,^{\circ}$ C, $p \approx 0.1 \,\text{mm}$ Hg) 1.00 g (4.59 mmol) of 10a (58%) as a pale yellow liquid.

Anal.: calc. for $C_{14}H_{22}N_2$ (218.3): C 77.01, H 10.16, N 12.83; found: C 75.82, H 10.10, N 12.58. $[\alpha]_{589}^{25} = +22$ (c = 1, CHCl₃). ¹H NMR (400 MHz, CDCl₃, *TMS*): 0.98–1.29 (m, 5H); 1.05 (d, J = 6.4 Hz, 3H); 1.43 (m, 1H); 1.64–1.77 (m, 5H); 2.14 (s, broad, 1H); 2.51 (m, 1H); 3.84 (d, J = 14.0 Hz, 1H); 3.96 (d, J = 14.0 Hz, 1H); 7.14 (m, 1H); 7.32 (m, 1H); 7.62 (m, 1H); 8.54 (m, 1H). MS: 217 (M - H)⁺.

2-{[N-(1S)-1-Cyclohexylethyl-N-methylamino]methyl}pyridine (10b)

Ligand **10b** was synthesized as described for **8a** from 1.51 g (9.21 mmol) of **14** and 1.00 g (7.09 mmol) of (*S*)-(+)-N-1-cyclohexylethyl-N-methylamine. After the extraction, the solvent was removed to give 1.13 g of a brown oil. Bulb-to-bulb distillation (oven temp. = 150-200 °C, $p \approx 0.1$ mm Hg) yielded 1.05 g (4.52 mmol) of **10b** (64%) as a pale yellow oil.

Anal.: calc. for $C_{15}H_{24}N_2$ (232.4): C 77.53, H 10.41, N 12.06; found: C 77.07, H 10.27, N 11.99. $[\alpha]_{589}^{25} = +31$ (c = 0.5, CHCl₃). ¹H NMR (250 MHz, CDCl₃, *TMS*): 0.73–0.93 (m, 2H); 0.97 (d, J = 6.5 Hz, 3H); 1.01–1.40 (m, 4H); 1.56–1.76 (m, 4H); 2.15 (s, 3H); 2.17 (m, 1H); 2.28–2.41 (m, 1H); 3.60 (d, J = 14.8 Hz, 1H); 3.77 (d, J = 14.8 Hz, 1H); 7.11 (m, 1H); 7.53 (d, J = 7.6 Hz, 1H); 7.63 (m, 1H); 8.49 (m, 1H). MS: 231 (M – H)⁺.

Catalytic reactions

0.3 mmol of the ligand, 0.1 mmol of $Cu(OAc)_2 \cdot H_2O$, and 2.6 mmol of cis-1,2-cyclopentanediol (1) were dissolved in dried CH_2Cl_2 (50 ml). For the reactions with [Cu(4)Cl]ClO₄ and [Cu(6)H_2O](ClO₄)₂, 0.1 mmol of the complex was used instead of $Cu(OAc)_2 \cdot H_2O$ and the ligand. The solution was degassed by bubbling with N₂ for 15 min and 2.6 mmol of Ph₃Bi(OAc)₂ (2) was added to start the reaction. The solution was stirred at room temperature for 15 h and was then put onto a silica gel column (3.5 × 8 cm) which was eluted with CH_2Cl_2 . The first 120 ml of eluent were discarded; the next 350 ml of eluent contained cis-2-hydroxy-1-phenoxy-cyclopentane (3). Cu(II) and unreacted 1 remained on the column. After removal of the solvent, the yellow residue was distilled (bulb-to-bulb, oven temp. = 120 °C, $p \approx 0.1 \text{ mm Hg}$) to give 3 as a colorless liquid.

Gas chromatographic analyses

The derivatization of *cis*-2-hydroxy-1-phenoxy-cyclopentane (**3**) was performed according to Ref. [17]. Separation of the derivatized enantiomers of **3** was performed by capillary gas chromatography using a 25 m Chirasil-L-val column (0.25 mm internal diameter, 0.12 µm film). Conditions: $T_{column} = 150 \,^{\circ}\text{C}$, $T_{injector} = 270 \,^{\circ}\text{C}$, $T_{detector} = 240 \,^{\circ}\text{C}$, pressure = 0.9 bar H₂. Typical retention times were 19.6 min and 20.7 min for the (1*S*,2*R*)- and (1*R*,2*S*)-enantiomers, respectively.

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