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Journal of Molecular Catalysis B: Enzymatic 39 (2006) 45–49
JOURNAL OF MOLECULAR CATALYSIS B: ENZYMATIC
www.elsevier.com/locate/molcatb

Lipase-mediated kinetic resolution of racemic and desymmetrization of prochiral organophosphorus P-boranes

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Available online 3 March 2006

Abstract

Enantiomerically enriched alkoxy(hydroxymethyl)phenylphosphine boranes were obtained via a lipase-catalyzed acetylation performed under kinetic resolution conditions. The reaction was slow and proceeded with a rather low enantioselectivity. A lipase-catalyzed acetylation of prochiral bis(2-hydroxyethyl)phenylphosphine borane resulted in its desymmetrization and gave the enantiomerically enriched monoacetyl derivative with ee up to 90%.

Keywords: Lipase; Kinetic resolution; Desymmetrization; Phosphine P-boranes; Chirality

1. Introduction

Chiral, non-racemic organophosphorus compounds containing a stereogenic phosphorus atom play an important role in various areas of current research, such as asymmetric organic synthesis, biochemistry and catalysis. They are used, among others, as chiral auxiliaries in stoichiometric reactions. Trivalent phosphorus compounds, especially tertiary phosphines, are used as chiral ligands in transition metal catalysts. However the synthetic approaches to enantiomerically pure forms of tertiary phosphines are few in number and of limited scope. Recently, there is growing interest in the synthesis and transformations of borane complexes of trivalent phosphorus compounds [1,2]. In contrast to phosphines and other derivatives of tricoordinate phosphorus, which are prone to oxidation and usually difficult to handle, they are stable compounds and can easily be converted into the corresponding P^{III} compounds without racemization. In view of growing tendency towards the use of enzymes and microorganisms for the synthesis of optically active compounds, we decided to apply biocatalytic methods for the synthesis of optically active borane complexes of chiral or prochiral P^{III} compounds. It should be noted that many hydrolytic enzymes proved to be capable of recognizing and stereoselectively binding heteroatom stereogenic centers, among them those located on phosphorus [3]. In our earlier investigations we have applied a variety of hydrolases (PLE, α-chymotrypsin and a number of lipases) in the synthesis of optically active organophosphorus compounds. Thus, we reported a successful PLE-promoted kinetic resolution of racemic P-chiral phosphonylacetates [4,5] and phosphorylacetates [6] and desymmetrization of prochiral phosphinyldiacetates [7]. In another set of experiments we investigated a lipase-promoted kinetic resolution of P-chiral hydroxymethylphosphoryl derivatives and proved that these reactions could be successfully carried out in various media: organic solvents [8,9], ionic liquids [10] and supercritical carbon dioxide, $scCO_2$ [11]. Simultaneously, some other groups also reported the results of their investigations devoted to similar subjects [12–15].

It should also be underlined that our preliminary results on the resolution of P^{III}-boranes were presented in part as posters at two international conferences—ICPC XV in Sendai [16] and recently at BIOTRANS 2005 in Delft [17]. The publication by Shioji et al. describing a closely related lipase-catalyzed optical resolution of P-chiral phosphine boranes, which came out in the meantime [14], prompted us to disclose our own results. Thus, the present paper deals with two subjects: a lipase-mediated kinetic resolution of alkoxy(hydroxymethyl)phenylphosphine boranes and desymmetrization of bis(2-hydroxyethyl)phenylphosphine borane.

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doi:10.1016/j.molcatb.2006.01.025

2. Experimental

2.1. General

NMR spectra were recorded on a Bruker instrument at 200 MHz for ^{1}H and 81 MHz for ^{31}P with $CDCl_3$ or C_6D_6 as solvents. Optical rotations were measured on a Perkin-Elmer 241 MC polarimeter. Column chromatography was carried out using Merck 60 silica gel. TLC was performed on Merck 60 F_{254} silica gel plates. The enzymes were purchased from AMANO or FLUKA.

Lipases:

CAL	*Candida antarctica* lipase B (Novozym 435)
AK	Lipase AK (AMANO)
PS	Lipase PS (AMANO)
LPL	Lipoprotein lipase
AH	Lipase AH (AMANO)

2.2. Synthesis of racemic hydroxymethylphosphine boranes **2**—general procedure

A hydroxymethylphenylphosphine oxide **1** (0.018 mol) was dissolved in a solution of BH_3/Me_2S in THF (2 M; 20 mL) and the mixture was stirred overnight at room temperature. To destroy the excess of BH_3 a saturated solution of $NaHCO_3$ was slowly added until no bubbling was observed. The solution was concentrated under vacuum and the aqueous residue was extracted with $CHCl_3$. The combined organic layers were dried over anhydrous magnesium sulfate and, after filtration, the solvents were evaporated. The crude mixture was purified by column chromatography using $CHCl_3$:MeOH in gradient. The yields of the recovered substrates **1**, the desired products **2** and the by-product **3** are shown in Table 1.

2.2.1. Ethoxy(hydroxymethyl)phenylphosphine P-borane ***2b***

Colorless oil, yield: 13%.

^{31}P NMR ($CDCl_3$): $\delta = 108.79$ (q, $J_{P\text{-}B} = 59.55$ Hz);
^{1}H NMR ($CDCl_3$): $\delta = 0.75$ (br q, $J_{B\text{-}H} = 86$ Hz, 3H, BH_3), 1.31 (t, $J = 7.05$, 3H, OCH_2CH_3), 1.85 (br s, 1H, OH), 3.87–4.20 (m, 4H, P-CH_2, OCH_2CH_3), 7.26–7.62 (m, 3H, Ph), 7.74–7.84 (m, 2H, Ph);
MS(CI): *m/z* 197 (*M*-H); HRMS(CI): calc. for $C_9H_{15}BPO_2$ *m/z* 197.090449, found *m/z* 197.09087.

Table 1
Synthesis of racemic hydroxymethylphosphine P-boranes *rac*-**2**

Entry	Substrate		Product yields [%]		
	1	R	**2**	**3**	Recovered **1**
1	**a**	MeO	5	83	12
2	**b**	EtO	13	52	26
3	**c**	*i*-PrO	23	35	34
4	**d**	*t*-Bu	100	–	–

2.2.2. Hydroxymethyl(i-propoxy)phenylphosphine P-borane ***2c***

Colorless oil, yield: 23%.

^{31}P NMR ($CDCl_3$): $\delta = 105.75$ (q, $J_{P\text{-}B} = 58.40$ Hz);
^{1}H NMR ($CDCl_3$): $\delta = 0.75$ (br q, $J_{B\text{-}H} = 88$ Hz, 3H, BH_3), 1.21 (d, $J = 6.15$ Hz, 3H) and 1.37 (d, $J = 6.15$, 3H, $OCH(CH_3)_2$), 1.81 (br s, 1H, OH), 4.04 (d, $J = 1.1$ Hz, 2H, P-CH_2), 4.48–4.65 (m, 1H, $OCH(CH_3)_2$), 7.26–7.60 (m, 3H, Ph), 7.76–7.86 (m, 2H, Ph);
^{13}C NMR ($CDCl_3$): $\delta = 20.39$, 62.6 (d, $J = 53$ Hz), 73.4, 128.5, 128.7, 129, 130.2, 131.0, 131.3, 132.3, 132.4.
MS(CI): *m/z* 211 (*M*-H); HRMS(CI): calc. for $C_{10}H_{17}BPO_2$ *m/z* 211.106125, found *m/z* 211.10567;
Anal. calc. for $C_{10}H_{18}BPO_2$: C 56.65, H 8.56, found C 56.71, H 8.45.

2.2.3. t-Butyl(hydroxymethyl)phenylphosphine P-borane ***2d***

Colorless oil, yield: 100%.

^{31}P NMR ($CDCl_3$): $\delta = 31.32$ (q, $J_{P\text{-}B} = 49.90$ Hz);
^{1}H NMR ($CDCl_3$): $\delta = 0.75$ (br q, $J_{B\text{-}H} = 90$ Hz, 3H, BH_3), 1.17 (d, $J = 13.78$ Hz, 9H, $PC(CH_3)$), 2.10 (br s, 1H, OH), 4.31–4.49 (m, 2H, P-CH_2), 7.26–7.57 (m, 3H, Ph), 7.67–7.76 (m, 2H, Ph);
^{13}C NMR ($CDCl_3$): $\delta = 25.78$, 29.15 (d, $J = 30.15$ Hz), 55.89 (d, $J = 39.11$ Hz), 124.34, 125.32, 128.36, 128.55, 131.50, 131.55, 133.36, 133.51.
MS(CI): *m/z* 209 (*M*-H).

2.3. Synthesis of bis(2-hydroxyethyl)phenylphosphine P-borane ***5***

This compound was synthesized in an analogous way as the phosphine boranes **2**, using 6-molar excess of BH_3/Me_2S at 45 °C for 12 h. After a similar work-up a white precipitate was obtained in 77% yield, mp = 73–75 °C.

^{31}P NMR ($CDCl_3$): $\delta = 8.3$–11.2 (m);
^{1}H NMR ($CDCl_3$): $\delta = 0.75$ (br q, $J_{B\text{-}H} = 88$ Hz, 3H, BH_3), 2.11–2.45 (m, 4H, P-CH_2), 3.72–4.06 (m, 4H, CH_2OH), 7.41–7.60 (m, 3H), 7.68–7.85 (m, 2H);
^{13}C NMR ($CDCl_3$): $\delta = 29.49$ (d, $J = 35.0$ Hz, P-*C*H_2), 57.49 (*C*H_2OH), 127.42, 128.49, 128.89, 129.09, 131.72, 131.92 (Ph).
MS(CI): *m/z* 211 (*M*-H).

2.4. Synthesis of racemic acetoxymethyl(i-propoxy)phenylphosphine P-borane ***6c***

Acetyl chloride (0.061 g, 0.778 mmol) was added dropwise to a solution of **2c** (0.150 g, 0.707 mmol), Et_3N (0.215 g, 2.122 mmol) and DMAP (0.017 g, 0.141 mmol) in CH_2Cl_2 (5 mL). The reaction mixture was stirred at room temperature for 16 h. The solution was washed with 1 M HCl (3× 5 mL) and the combined aqueous phases were extracted with ether (3× 5 mL). The combined organic layers were washed with aqueous

saturated Na_2CO_3 (3 mL) and brine (3 mL) and dried over anhydrous magnesium sulfate. The solvent was evaporated and the crude mixture was purified by column chromatography using chloroform as eluent.

Colorless oil, yield: 56%.

^{31}P NMR ($CDCl_3$): $\delta = 104.87$ (q, $J_{P\text{-}B} = 50.07$ Hz);
^{1}H NMR ($CDCl_3$): $\delta = 0.75$ (br q, $J_{B\text{-}H} = 88$ Hz, 3H, BH_3), 1.21 (d, $J = 6.15$, 3H) and 1.37 (d, $J = 6.15$, 3H, OCH(CH_3)$_2$), 2.04 (s, 3H, CH_3CO), 4.43–4.69 (m, 3H, P-CH_2, OCH(CH$_3$)$_2$), 7.26–7.60 (m, 3H, Ph), 7.76–7.86 (m, 2H, Ph);
^{13}C NMR ($CDCl_3$): $\delta = 20.39$, 24.11 (d, $J = 3.82$ Hz), 62.6 (d, $J = 53$ Hz), 73.4, 128.5, 128.7, 129, 130.2, 131.0, 131.3, 132.3, 132.4, 169.82 (d, $J = 2.64$ Hz).
MS(CI): *m/z* 253 (*M*-H); HRMS(CI): calc. for $C_{12}H_{19}BPO_3$ *m/z* 253.116740, found *m/z* 253.11691;
Anal. calc. for $C_{12}H_{20}BPO_3$: C 56.73, H 7.93, found C 56.71, H 7.85.

2.5. Kinetic resolution of hydroxymethylphosphine P-boranes **2**—general procedure

A solution of racemic **2** (0.25 mmol), an enzyme (20 mg) and vinyl acetate (0.5 mL) in isopropyl ether (5 mL) was stirred at room temperature. The reaction was monitored by TLC ($CHCl_3$:MeOH 15:1) and was stopped after the time indicated in Table 2. The enzyme was filtered and solvents were evaporated. The mixture of the product and the unreacted substrate was separated by column chromatography using a chloroform:methanol gradient as solvent. The results are shown in Table 2. Analytical data for **2b**, **2c** and **6c** were identical with those for the racemic products.

2.5.1. Acetoxymethyl(ethoxy)phenylphosphine P-borane **6b**

Colorless oil.

^{31}P NMR ($CDCl_3$): $\delta = 107.99$ (q, $J_{P\text{-}B} = 52.83$ Hz);
^{1}H NMR ($CDCl_3$): $\delta = 0.75$ (br q, $J_{B\text{-}H} = 86$ Hz, 3H, BH_3) 1.31 (t, $J = 7.05$, 3H, OCH$_2$CH_3), 2.04 (s, 3H, CH_3CO), 3.91–4.20 (m, 2H, OCH_2CH$_3$), 4.57 (q, 2H, P-CH_2), 7.26–7.62 (m, 3H, Ph), 7.74–7.84 (m, 2H, Ph);
MS(CI): *m/z* 239 (*M*-H); HR MS(CI): calc. for $C_{11}H_{17}BPO_3$ *m/z* 239.101049, found *m/z* 239.10147;
Anal. calc. for $C_{11}H_{18}BPO_3$: C 55.04, H 7.56, found C 55.29, H 7.55.

2.6. Enzymatic hydrolysis of racemic **6c**

A mixture of **6c** (0.250 mmol) and a lipase (ca. 10 mg) in a *i*-Pr$_2$O (5 mL) saturated with a phosphate buffer (pH 7.2) was stirred at room temperature. The reaction was monitored by TLC ($CHCl_3$:MeOH 15:1) and was stopped after several days despite the conversion did not reach 50%. Then, anhydrous magnesium sulfate was added to remove water. The precipitates were filtered off, the solvents were removed under vacuum and the residue was purified by column chromatography using chloroform:methanol in gradient as solvent.

2.7. Enzymatic acetylation of bis(2-hydroxyethyl)phenylphosphine P-borane **5**

A mixture of **5** (0.236 mmol), a lipase (10–20 mg) and vinyl acetate (1 mL) in an appropriate solvent (5 mL; see Table 1) was stirred at room temperature. The reaction was monitored by TLC (AcOEt:hexane 2:1). The average reaction time exceeded 2 or 3 weeks. The enzyme was filtered off and the solvent was removed under vacuum. The residue was purified by preparative TLC (AcOEt:hexane 2:1) to give optically active monoacetyl product 8. The yields, optical rotations and ee values are given in Table 3.

2.7.1. 2-Acetoxyethyl(2-hydroxyethyl)phenylphosphine P-borane **8**

^{31}P NMR ($CDCl_3$): $\delta = 0.75$ (br q, $J_{B\text{-}H} = 88$ Hz, 3H, BH_3);
^{1}H NMR ($CDCl_3$): $\delta = 1.85$ (s, 3H, BH_3), 2.10–2.47 (m, 7H), 3.70–3.98 (m, 2H), 4.05–4.0 (m, 2H), 7.35–7.82 (m, 5H, Ph);
^{13}C NMR ($CDCl_3$): $\delta = 20.59$ (CH_3CO), 25.93 (d, $J = 35.4$ Hz, P-CH_2), 29.34 (d, $J = 35.6$ Hz, P-CH_2), 57.36 (CH_2O), 59.12

Table 2
Kinetic resolution of hydroxymethylphosphine P-boranes **2**

Entry	**2**	Time (days)	Lipase	Solvent	Recovered alcohol **2**				Acetate **6**			
					Yield[a] (%)	$[\alpha]_D$ ($CHCl_3$)	ee (%)	Absolute configuration	Yield[a] (%)	$[\alpha]_D$ ($CHCl_3$)	ee (%)	Absolute configuration
1	**b**	10	CAL	*i*-Pr$_2$O	30	18.9	20[b]	n.d.	38	+16.9	–	n.d.
2	**c**	15	AK	*i*-Pr$_2$O	51	−9.1	14	*S*	21	+11.8	31	*R*
3	**c**	19	PS	*i*-Pr$_2$O	55	+2.0	3	*R*	15	−8.3	22	*S*
4	**c**	41	LPL	*i*-Pr$_2$O	19	−4.4	7	*S*	15	+2.6	7	*R*
5	**c**	6	CAL	*i*-Pr$_2$O	43	−7.9	12	*S*	39	+6.9	18	*R*
6	**c**	47	AH	*i*-Pr$_2$O	39	−1.3	2	*S*	12	+1.3	4	*R*
7	**c**	0.9	CAL	*c*-C$_6$H$_{12}$	31	−24.1	37	*S*	49	+11.0	29	*R*
8	**c**	0.25	CAL	*c*-C$_6$H$_{12}$[c]	28	−20.5	32	*S*	48	+8.4	22	*R*
9	**c**	0.9	PS	*c*-C$_6$H$_{12}$	69	−0.6	1	*S*	5	+2.6	7	*R*

[a] Isolated yields.
[b] Determined by NMR using (+)-(*S*) Mosher's chloride.
[c] Molecular sieves 3 Å (20 mg) were added.

Table 3
Desymmetrization of bis(2-hydroxyethyl)phenylphosphine P-borane **5**

Entry	Time (days)	Solvent	Acetate **8**		
			Yield (%)	$[\alpha]_D$ (MeOH)	ee[a](%)
1	14	$CHCl_3$	43	−10.0	90
2	21	*i*-Pr_2O	42	+1.0	10

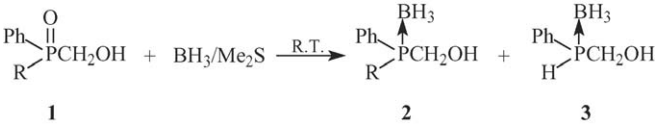
[a] Determined by NMR using (+)-(*R*) *t*-butylphenylphosphinothioic acid as chiral solvating agent.

(CH_2O), 126.93, 128.00, 128.90, 129.09, 131.72, 131.91 (Ph), 170.63 (CH_3COO).
MS (CI): *m/z* 253 (*M*-H).

3. Results and discussion

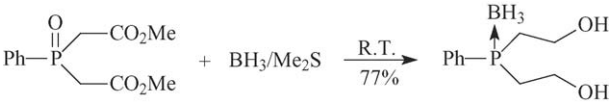
3.1. Synthesis of substrates

Alkoxy(hydroxymethyl)phenylphosphine boranes **2** were obtained by reduction of the corresponding phosphine oxides **1** with borane-dimethyl sulfide complex (Eq. (1), Table 1). The reaction proceeded smoothly, but an unwanted by-product, a secondary phosphine borane **3**, was always formed. It was a product of a subsequent reduction of the alkoxy group and its content in the reaction mixture was higher in the case of lower alkyl substituents. Therefore, the reaction had to be stopped at a relatively low conversion. However, even such an approach proved impractical to obtain the methoxy analogue **2a**. An analogous reduction which was performed for comparison using *t*-butyl(hydroxymethyl)phenylphosphine oxide **1d** gave the expected P-borane derivative **2d** in 100% yield.



1 2 3 (1)

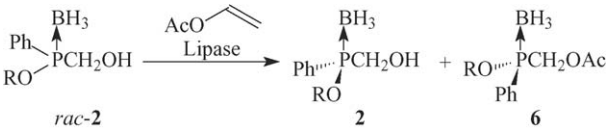
Bis(hydroxyethyl)phenylphosphine borane **5** was synthesized in a similar reaction, starting from the phosphine oxide **4**, described earlier [18] (Eq. (2)).



4 5 (2)

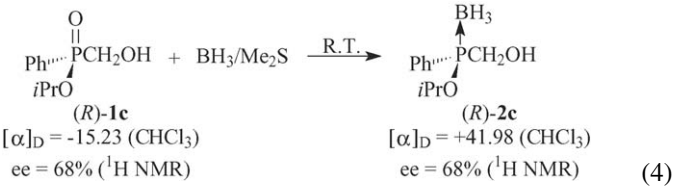
3.2. Kinetic resolution of 2

To achieve kinetic resolution of **2,** we screened several lipases for their enantioselective acetylation (Eq. (3)). All the reactions were carried out in organic solvents as reaction media. The results are collected in Table 2.

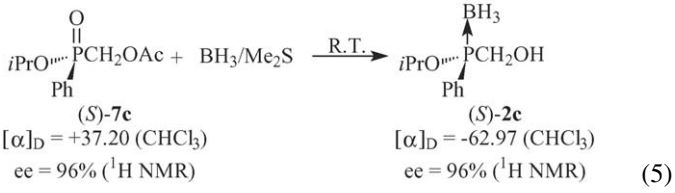


rac-**2** **2** **6** (3)

The absolute configuration and enantiomeric excesses of the recovered **2c** and the acetate **6c** were determined by comparing their $[\alpha]_D$ values with those of the P-boranes obtained by the BH_3/Me_2S reduction of the corresponding P=O derivatives **1c** and **7c**, whose absolute configurations and ee's were known [8,9] (Eqs. (4) and (5)). The reduction was assumed to proceed with retention of configuration at phosphorus [19].



(4)



(5)

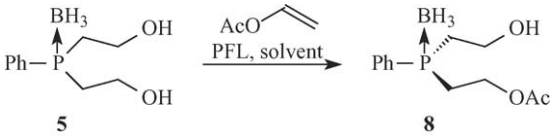
Inspection of the data shown in Table 2 reveals that the lipase-promoted acetylation of **2**, performed in diisopropyl ether was very slow and proceeded with a rather low stereoselectivity. However, the less polar cyclohexane proved to substantially increase the reaction rate, particularly in the presence of molecular sieves, although the stereoselectivity of the transformation remained on the same level (Table 2, entries 7–9) Interestingly, lipase PS was found to exhibit the opposite stereochemical preference comparing with all other lipases applied (Table 2, entry 3). Surprisingly, this was true for the reaction carried out in diisopropyl ether only (cf. Table 2, entry 9). At the moment, it is not possible to provide any reasonable explanation of this observation.

Having obtained these rather disappointing results, we decided to check whether a reverse reaction, i.e. enzyme promoted hydrolysis of the racemic acetates **6**, would be more enantioselective. To this end, **6c** was subjected to hydrolysis in the presence of two lipases—PS and AK. However, this reaction turned out to be even slower than the acetylation described above and to give the products with lower ee, and in the case of **6d** not to proceed at all.

3.3. Desymmetrization of 5

Desymmetrization of the prochiral diol **5** was attempted by a similar procedure, using vinyl acetate as an acetylating agent and several lipases, of which only *Pseudomonas fluorescens* lipase proved efficient (Eq. (6)). It should be stressed that the use of various solvents led to opposite enantiomers of the product **8** and also substantially influenced the stereoselectivity of the process (Table 3). This might be due to a different polarity of the solvents used—the less polar chloroform promotes higher stereoselectivity than diisopropyl ether. A similar relationship

was also observed earlier [14].



5 8 (6)

4. Conclusions

In contrast to P-chiral hydroxymethylphosphine oxides, their P-borane analogues turned out to be rather poor substrates for lipase-catalyzed transformations. The reactions were usually slow and proceeded with low stereoselectivity. Any attempt to explain the difference may be at the moment speculative only. It seems reasonable to assume that the main reason may be a different size and electronic character of the P=O and the P–BH_3 bonds. Our future investigations will be aimed at the optimization of the yields and stereoselectivity by changing the media and conditions of the reaction as well as at the molecular modeling which should allow the location of the substrates in the lipases active sites to be determined.

Acknowledgment

Financial support from the Ministry of Science and Information Society Technologies, Grant No. 3 T09A 166 27, is gratefully acknowledged.

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