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Synthesis of Enantiomerically Pure 3-Aryloxy-2-hydroxypropanoic Acids, Intermediate Products in the Synthesis of *cis*-4-Aminochroman-3-ols

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Abstract—Oxidation of accessible (*R*)-3-chloropropane-1,2-diol to (*R*)-3-chloro-2-hydroxypropanoic acid and subsequent reaction of the latter with *ortho*-substituted sodium phenoxide gave a number of enantiomerically pure 3-aryloxy-2-hydroxypropanoic acid which are intermediate products in the synthesis of nonracemic 4-aminochroman-3-ols.

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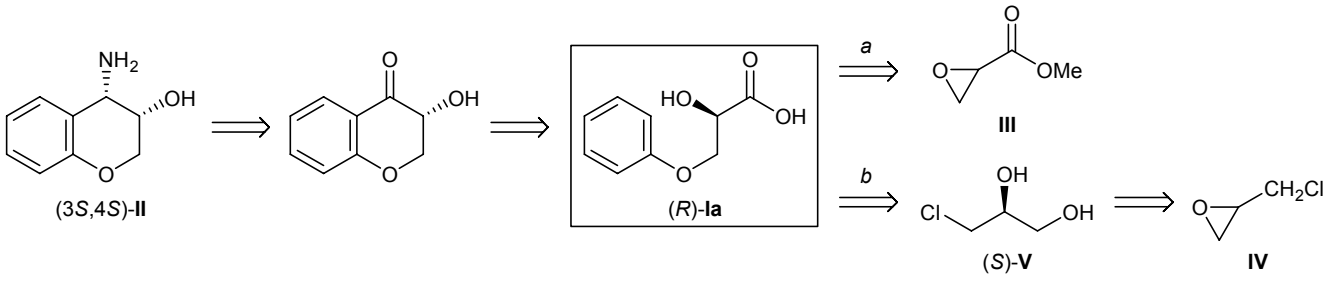
3-Aryloxy-2-hydroxypropanoic acids (**I**) are used as building blocks in the synthesis of biologically active chromanones and aminochromanols **II** [1, 2]. Compounds **II** are structural analogs of *cis*-3-aminoindan-2-ol, intermediate product in the synthesis of the HIV protease inhibitor Indinavir (Crixivan) [3].

Both enantiomers of *cis*-4-aminochroman-3-ol, (*R*,*R*)-**II** and (*S*,*S*)-**II**, have been reported [1]. Retrosynthetic analysis of aminochromanol **II** (Scheme 1) led us to presume methyl oxirane-2-carboxylate (**III**) or 2-chloromethyloxirane (**IV**, epichlorohydrin) as starting compound. Path *a* has been well documented [2, 4]. It includes kinetic resolution of racemic ester **III** via enantioselective opening of the oxirane ring with phenol in the presence of chiral (salen)Co(III) complex, followed by alkaline hydrolysis of the ester group. This approach ensures preparation of key acid (*R*)-**Ia** in a good yield (85–90%) and satisfactory enantiomeric excess (*ee* 95%). When *ortho*-substituted phenols were used for the kinetic resolution, the reaction rate sharply decreased, and the yield and *ee* value of the product were also lower [4].

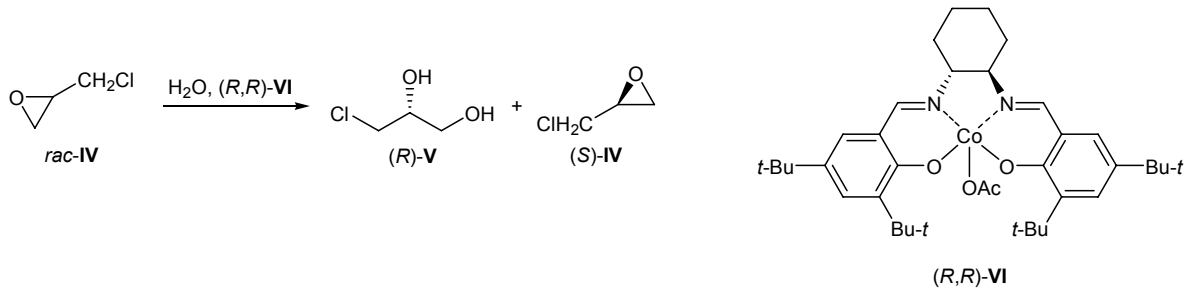
In this paper we describe the synthesis of nonracemic 2-hydroxy-3-(2-R-phenoxy)propanoic acids **Ia**–**Ie** starting from epichlorohydrin (**IV**) (path *b*). Insofar as acid (*R*)-**Ia** has already been described in the literature, we tried to synthesize (*S*)-enantiomers of acid **I**. Kinetically controlled enantioselective hydrolysis of racemic epichlorohydrin (*rac*-**IV**) in the presence of chiral salen cobalt complexes **VI** (Scheme 2) was reported in detail in [5]. The use of (*R*,*R*)-**VI** as catalyst ensured formation of enantiomerically pure epichlorohydrin (*S*)-**IV** and 3-chloropropan-1,2-diol (*R*)-**V**, which can be readily separated by distillation.

In order to obtain hydroxypropanoic acids **I** we made use of the procedure for the oxidation of racemic chloropropanediol *rac*-**V** to racemic 3-chloro-2-hydroxypropanoic acid (*rac*-**VII**) with concentrated nitric

Scheme 1.



Scheme 2.

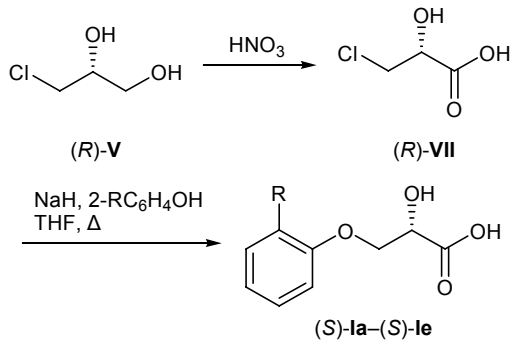


acid, which was originally proposed by Hope and Wälti [6] and optimized by Gerfaud et al. [7]. The same procedure was successfully applied to obtain optically active (*R*)-3-chloro-2-hydroxypropanoic acid (*R*)-(**VII**) [8]. The synthesis of compounds **Ia**–**Ie** is outlined in Scheme 3.

The oxidation of diol (*R*)-**V** was not accompanied by racemization, and the enantiomeric purity of the resulting acid (*R*)-**VII** is directly related to the *ee* value of initial chloropropanediol (*R*)-**V**. The oxidation procedure is also advantageous due to the possibility to improve even moderate enantiomeric purity of (*R*)-**VII** (*ee* ≥80%) to any desired value (*ee* >99%) by simple recrystallization.

Synthesis of enantiomerically pure acids **I** from 3-chloro-2-hydroxypropanoic acids was not reported previously. We carried out reactions of acid (*R*)-**VII** with 2-substituted phenols in water in the presence of excess alkali, as well as in dimethoxyethane and THF with sodium phenoxide prepared preliminarily by treatment of the corresponding phenol with sodium hydride. The best results were obtained by heating the reactants in boiling THF; in this case, the target acids were isolated in 76–80% yield with *ee* ≥99%. The *ee* values were determined by HPLC using chiral stationary phases. Hydroxypropanoic acids (*S*)-**Ia**–(*S*)-**Ie** were analyzed after treatment with diazomethane to convert them into the corresponding methyl esters. The column was initially calibrated against racemic acids *rac*-**Ia**–*rac*-**Ie** prepared according to Scheme 3 from racemic 3-chloropropane-1,2-diol (*rac*-**V**).

Scheme 3.



R = H (**a**), Me (**b**), MeO (**c**), Cl (**d**), Br (**e**).

Important information on the effect of crystallization on the optical activity of a chiral compound can be obtained by comparing melting points of its racemic and enantiomerically enriched samples [9]. Comparison of the melting points of *rac*-**Ia**–*rac*-**Ie** and (*S*)-**Ia**–(*S*)-**Ie** in pairs (see Experimental) revealed considerable differences in these values. Positive difference ΔT = mp(*rac*) – mp(*S*) indicates formation of racemic compounds in crystal. If the melting point of a racemic compound is higher by 20–30°C than that of the pure enantiomer, it is highly probable that this chiral compound crystallizes as an "anti-conglomerate"; enantiomeric purity of such compounds is difficult or almost impossible to improve by recrystallization. High ΔT values were observed for acids **Ia**–**Ic** and **Ie**. Therefore, it is advisable to avoid recrystallization of these acids and select other purification methods, e.g., column chromatography. Specific features of crystallization of the whole series of compounds **Ia**–**Ie** will be the subject of our further studies.

To conclude, it is reasonable to synthesize enantiomerically pure 3-(aryloxy)-2-hydroxypropanoic acids (*S*)-**Ia**–(*S*)-**Ie** via enantioselective kinetic resolution at the stage of preparation of pure enantiomer **V**. This approach is more advantageous than enantioselective opening of the oxirane ring in **III** with phenols, for it allows introduction of substituents into the *ortho* position of the benzene ring and ensures high enantiomeric purity of target hydroxypropanoic acids **I** (*ee* >99%) by means of enantiomeric enrichment of (*R*)-3-chloro-2-hydroxypropanoic acid (*R*)-(**VII**) by crystallization.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance-400 spectrometer at 399.9 and 100.6 MHz, respectively; the chemical shifts were

measured relative to the solvent signals. The IR spectra were obtained in KBr on a Bruker Tensor 27 spectrometer. The optical rotations were measured on a Perkin Elmer 341 polarimeter. The melting points were determined on a Boetius hot stage and were not corrected. The elemental compositions were determined using a EuroVector EA3000 CHN analyzer. Thin-layer chromatography was performed on Silufol UV-254 plates using chloroform–methanol–acetic acid (10 : 1 : 1) as eluent; spots were developed under UV light or by treatment with iodine vapor. GLC analyses were carried out on an Agilent 7890A chromatograph equipped with a flame ionization detector (Supelco BETA DEX^{TM} 225 capillary column, 30 m × 0.25 mm × 0.25 μm; carrier gas helium, flow rate 3 mL/min; oven temperature 150°C). HPLC analyses were obtained on a Shimadzu LC-20AD chromatograph equipped with RID-10A and SPD-20A detectors [Chiralpak AD and Chiralcel OD columns, 0.46 × 25 cm (Daicel); eluent hexane–propan-2-ol, 1 : 1 (A) or 9 : 1 (B), flow rate 1 mL/min]. For the determination of enantiomeric compositions, the columns were calibrated against the corresponding racemic hydroxypropanoic acids which were preliminarily converted into methyl esters by treatment with diazomethane. For this purpose, racemic acid, 10 mmol, was dissolved in aqueous methanol (1 : 10), a solution of diazomethane in diethyl ether was added until the mixture turned light yellow, the solvent was removed under reduced pressure, and the residue was dissolved in 5 mL of propan-2-ol and analyzed by HPLC or GLC.

Racemic epichlorohydrin (**IV**) and 3-chloropropane-1,2-diol (**V**), substituted phenols, and sodium hydride were commercial products. Enantiomerically pure 3-chloropropane-1,2-diol (*R*)-(**V**) was synthesized by hydrolytic kinetic resolution of *rac*-epichlorohydrin according to Jakobsen [5]. (*R*)-**V**: $[\alpha]_D^{20} = -6.3$ (*c* = 5, H_2O), *ee* 95% (Chiralpack AD, eluent B; t_{min} 12.2, t_{maj} 13.1 min).

3-Chloro-2-hydroxypropanoic acid (VII) [6, 8]**.** Racemic or scalemic 3-chloropropane-1,2-diol (**V**), 10.0 g (90.5 mmol), was dissolved at 0°C in 31 mL of 63% nitric acid. (*CAUTION!* Taking into account hazards in handling large amounts of concentrated nitric acid, it is strongly recommended to follow the instructions given in [7] for enlarged syntheses). The solution was heated to 70°C, and vigorous reaction started with evolution of a brown gas. The flask was withdrawn from the heating bath until vigorous reaction ceased (~15 min), and the mixture was then heated for 5–6 h at 100°C, cooled to room temperature, and neutralized by adding in portions 7 g of $NaHCO_3$. The product was extracted into diethyl ether (8 × 100 mL), the extract was dried over $MgSO_4$, most part of the solvent was removed by distillation on a water bath, and the residue was concentrated under reduced pressure at a bath temperature of 60°C. Crude product **VII** gradually crystallized on storage. It was filtered off and washed with cold carbon tetrachloride. Yield 70–80%. An analytical sample was obtained by recrystallization from chloroform.

***rac*-3-Chloro-2-hydroxypropanoic acid (*rac*-VII).** Yield 5 g (45%), mp 78–80°C (from $CHCl_3$); published data [6]: mp 78–80°C. R_f 0.21. IR spectrum, ν, cm^{-1}: 3390 (OH), 2982–2505 (COOH), 1742 (C=O), 679 (C–Cl).

(*R*)-3-Chloro-2-hydroxypropanoic acid (*R*)-(VII). Yield 5.6 g (50%), mp 89–91°C (from $CHCl_3$), $[\alpha]_D^{20}$ = +3.9 (*c* = 9, H_2O); published data [8]: mp 91.5–93°C, $[\alpha]_D^{20}$ = +3.95 (*c* = 9, H_2O); R_f 0.21; *ee* 99.9% (for methyl 3-chloro-2-hydroxypropanoate, GLC: t_{maj} 15.7, t_{min} 16.1 min). IR spectrum, ν, cm^{-1}: 3450 (OH), 2973–2604 (COOH), 1726 (C=O), 682 (C–Cl).

Acids Ia–Ie (*general procedure***).** Racemic or enantiomerically pure 3-chloro-2-hydroxypropanoic acid *rac*-**VII** or (*R*)-**VII**, 1.12 g (9 mmol), was dissolved in 10 mL of anhydrous THF, 0.36 g (9 mmol) of sodium hydride (a 60% suspension in mineral oil) was added under stirring at 0°C, and a solution of 10.8 mmol of the corresponding substituted sodium phenoxide [prepared from 0.43 g (10.8 mmol) of sodium hydride (60% suspension in mineral oil) and 10.8 mmol of phenol in 10 mL of THF] was added over a period of 0.5 h. The mixture spontaneously warmed up to room temperature under stirring and was heated for ~4 h under reflux. The solvent was removed, the residue was treated with 40 mL of water and 40 mL of diethyl ether, and the aqueous phase was separated, extracted with diethyl ether (2 × 20 mL) to remove neutral substances, acidified with 1 M H_2SO_4, and extracted with ethyl acetate (3 × 40 mL). The extract was dried over $MgSO_4$, and the solvent was distilled off under reduced pressure. Racemic acids were purified by recrystallization. Scalemic products were purified by flash chromatography on silica gel using chloroform–methanol–acetic acid (10 : 1 : 1) as eluent.

***rac*-2-Hydroxy-3-phenoxypropanoic acid (*rac*-Ia).** Yield 80%, mp 157–159°C (from benzene); published data [10]: mp 156°C; R_f 0.26. IR spectrum, ν, cm^{-1}: 3359 (OH), 2946–2546 (COOH), 1713 (C=O), 1599 (C=C_{arom}). ^{1}H NMR spectrum (DMSO-d_6), δ,

ppm: 3.99 d.d.d (1H, OCH_2, J = 5.6, 9.8, 1.4 Hz), 4.10 m (1H, C**H**OH, J = 2.6, 5.6 Hz), 4.16 d.d (1H, OCH_2, J = 2.6, 9.8 Hz), 4.47 br.s (2H, OH), 6.89–6.92 m (3H, *o*-H, *p*-H), 7.23–7.28 m (2H, *m*-H). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 70.9 (CHO), 71.9 (CH_2O), 115.4 (C^o), 121.3 (C^p), 130.3 (C^m), 159.6 (C^i), 174.3 (COOH). Found, %: C 59.58; H 5.32. $C_9H_{10}O_4$. Calculated, %: C 59.34; H 5.53.

(*S*)-2-Hydroxy-3-phenoxypropanoic acid (*S*)-(Ia). Yield 80%, mp 132–134°C, R_f 0.26, $[\alpha]_D^{20}$ = +25.6 (c = 1.2, MeOH); *ee* 99.7% [methyl ester; HPLC, Chiralcel OD, eluent A; t_{maj} 6.0, t_{min} 12.0 min]. IR spectrum, ν, cm^{-1}: 3524 (OH), 2964–2506 (COOH), 1737, 1707 (C=O), 1595 (C=C_{arom}). ^1H NMR spectrum (CD_3OD), δ, ppm: 4.16 d.d (1H, OCH_2, J = 5.6, 9.8 Hz), 4.26 d.d (1H, OCH_2, J = 2.6, 9.8 Hz), 4.38 d.d (1H, C**H**OH, J = 2.6, 5.6 Hz), 6.86–6.98 m (3H, H_{arom}), 7.26–7.29 m (2H, H_{arom}). Found, %: C 59.19; H 5.43. $C_9H_{10}O_4$. Calculated, %: C 59.34; H 5.53.

***rac*-2-Hydroxy-3-(2-methylphenoxy)propanoic acid (*rac*-Ib).** Yield 75%, mp 153–154°C (first from H_2O and then from CH_2Cl_2); published data [11]: mp 146–146.5°C; R_f 0.33. IR spectrum, ν, cm^{-1}: 3423 (OH), 2943–2645 (COOH), 1725 (C=O), 1602, 1590 (C=C_{arom}). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.13 s (3H, CH_3), 4.04 d.d (1H, OCH_2, J = 5.4, 9.9 Hz), 4.16 d.d (1H, OCH_2, J = 3.2, 9.9 Hz), 4.21 d.d (1H, C**H**OH, J = 3.2, 5.4 Hz), 5.35 br.s (2H, OH), 6.82 t (1H, 5′-H, J = 7.3 Hz), 6.89 d (1H, 6′-H, J = 8.2 Hz), 7.09–7.13 m (2H, 3′-H, 4′-H). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 15.8 (CH_3), 69.8 (CHO), 70.9 (CH_2O), 111.6 ($C^{6'}$), 120.2 ($C^{4'}$), 126.0 ($C^{2'}$), 126.8 ($C^{5'}$), 130.3 ($C^{3'}$), 156.7 ($C^{1'}$), 173.5 (COOH). Found, %: C 60.91; H 5.96. $C_{10}H_{12}O_4$. Calculated, %: C 61.22; H 6.16.

(*S*)-2-Hydroxy-3-(2-methylphenoxy)propanoic acid (*S*)-(Ib). Yield 74%, mp 122–124°C, R_f 0.34, $[\alpha]_D^{20}$ = +26.8 (c = 0.7, MeOH); *ee* 99.8% [methyl ester; HPLC, Chiralcel OD, eluent A; t_{maj} 6.9, t_{min} 11.2 min]. IR spectrum, ν, cm^{-1}: 3566, 3478, 3333 (OH), 2926–2592 (COOH), 1735, 1715 (C=O), 1602, 1590 (C=C_{arom}). The NMR spectra were identical to those given above for acid *rac*-**Ib**. Found, %: C 61.15; H 6.10. $C_{10}H_{12}O_4$. Calculated, %: C 61.22; H 6.16.

***rac*-2-Hydroxy-3-(2-methoxyphenoxy)propanoic acid (*rac*-Ic).** Yield 74%, mp 98–100°C, R_f 0.19. IR spectrum, ν, cm^{-1}: 3419 (OH), 2933–2551 (COOH), 1723 (C=O), 1588 (C=C_{arom}). ^1H NMR spectrum (CD_3OD), δ, ppm: 3.85 s (3H, CH_3), 4.11–4.17 m (1H, CH_2O), 4.24–4.31 m (2H, C**H**OH, CH_2O), 6.85–7.02 m (4H, H_{arom}). ^{13}C NMR spectrum (CD_3OD), δ_C, ppm: 56.7 (CH_3), 71.3 (CHO), 72.9 (CH_2O), 114.0 ($C^{3'}$), 116.1 ($C^{6'}$), 122.3 ($C^{4'}$), 123.2 ($C^{5'}$), 149.6 ($C^{1'}$), 151.1 ($C^{2'}$), 175.0 (COOH). Found, %: C 56.73; H 5.54. $C_{10}H_{12}O_5$. Calculated, %: C 56.60; H 5.70.

(*S*)-2-Hydroxy-3-(2-methoxyphenoxy)propanoic acid (*S*)-(Ic). Yield 74%, mp 70–72°C, R_f 0.19, $[\alpha]_D^{20}$ = +16.9 (c = 0.9, MeOH); *ee* 99.8% [methyl ester; HPLC, Chiralcel OD, eluent A; t_{maj} 7.8, t_{min} 22.9 min]. IR spectrum, ν, cm^{-1}: 3485, 3403, 3263 (OH), 2938–2551 (COOH), 1739, 1715 (C=O), 1593 (C=C_{arom}). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 3.86 s (3H, CH_3), 4.29 d.d (1H, OCH_2, J = 4.0, 10.1 Hz), 4.36 d.d (1H, OCH_2, J = 4.8, 10.1 Hz), 4.54 d.d (1H, C**H**OH, J = 4.0, 4.8 Hz), 6.60 br.s (2H, OH), 6.88– 6.94 m (2H, H_{arom}), 6.95–7.02 m (2H, H_{arom}). Found, %: C 55.96; H 5.78. $C_{10}H_{12}O_5$. Calculated, %: C 56.60; H 5.70.

***rac*-3-(2-Chlorophenoxy)-2-hydroxypropanoic acid (*rac*-Id).** Yield 74%, mp 136–137°C (from EtOAc), R_f 0.28. IR spectrum, ν, cm^{-1}: 3385 (OH), 2968–2572 (COOH), 1763, 1721 (C=O), 1590 (C=C_{arom}). ^1H NMR spectrum (CD_3OD), δ, ppm: 4.30 d.d (1H, OCH_2, J = 5.0, 10.0 Hz), 4.36 d.d (1H, OCH_2, J = 3.1, 10.0 Hz), 4.55 d.d (1H, C**H**OH, J = 3.1, 5.0 Hz), 6.95 d.d.d (1H, 4′-H, J = 1.3, 7.9, 9.0 Hz), 7.11 d.d (1H, 6′-H, J = 1.3, 8.2 Hz), 7.26 d.d.d (1H, 5′-H, J = 1.6, 8.2, 9.0 Hz), 7.36 d.d (1H, 3′-H, J = 1.6, 7.9 Hz). ^{13}C NMR spectrum (CD_3OD), δ_C, ppm: 70.7 (CHO), 72.0 (CH_2O), 114.4 ($C^{6'}$), 121.5 ($C^{4'}$), 122.9 ($C^{2'}$), 127.6 ($C^{5'}$), 129.8 ($C^{3'}$), 154.6 ($C^{1'}$), 175.0 (COOH). Found, %: C 49.68; H 4.38. $C_9H_9ClO_4$. Calculated, %: C 49.90; H 4.19.

(*S*)-3-(2-Chlorophenoxy)-2-hydroxypropanoic acid (*S*)-(Id). Yield 75%, mp 135–137°C, R_f 0.29, $[\alpha]_D^{20}$ = +14.6 (c = 1, MeOH); *ee* 99.8% [methyl ester; HPLC, Chiralcel OD, eluent A; t_{maj} 6.5, t_{min} 9.0 min]. IR spectrum, ν, cm^{-1}: 3363 (OH), 2984–2553 (COOH), 1705 (C=O), 1588 (C=C_{arom}). The NMR spectra were identical to those given above for *rac*-**Id**. Found, %: C 49.61; H 3.71. $C_9H_9ClO_4$. Calculated, %: C 49.90; H 4.19.

***rac*-3-(2-Bromophenoxy)-2-hydroxypropanoic acid (*rac*-Ie).** Yield 76%, mp 147–149°C (from hexane–EtOAc), R_f 0.32. IR spectrum, ν, cm^{-1}: 3422 (OH), 2963–2583 (COOH), 1726 (C=O), 1584 (C=C_{arom}). ^1H NMR spectrum (CD_3OD), δ, ppm: 4.27 d.d (1H, OCH_2, J = 4.7, 9.9 Hz), 4.32 d.d (1H, OCH_2, J = 3.6, 9.9 Hz), 4.56 d.d (1H, C**H**OH, J = 3.6, 4.7 Hz), 6.86 d.d.d (1H, 4′-H, J = 1.1, 7.9, 8.1 Hz), 7.03 d.d (1H, 6′-H, J = 1.1, 8.2 Hz), 7.28 d.d.d (1H, 5′-H, J = 1.4, 8.1, 8.2 Hz), 7.50 d.d (1H, 3′-H, J = 1.4,

7.9 Hz). ^{13}C NMR spectrum (CD_3OD), δ_C, ppm: 71.1 (CHO), 72.4 (CH_2O), 113.1 ($C^{2'}$), 115.3 ($C^{6'}$), 123.6 ($C^{4'}$), 129.7 ($C^{5'}$), 134.3 ($C^{3'}$), 156.3 ($C^{1'}$), 173.7 (COOH). Found, %: C 40.76; H 3.12. $C_9H_9BrO_4$. Calculated, %: C 41.41; H 3.47.

(*S*)-3-(2-Bromophenoxy)-2-hydroxypropanoic acid (*S*)-(Ie). Yield 78%, mp 127–129°C, R_f 0.32, $[\alpha]_D^{20}$ = +14.7 (*c* = 1.1, MeOH); *ee* 99.4% [methyl ester; HPLC, Chiralcel OD, eluent A; t_{maj} 6.9, t_{min} 9.3 min]. IR spectrum, ν, cm^{-1}: 3357, 3266 (OH), 2983–2548 (COOH), 1727 (C=O), 1587 (C=C_{arom}). The NMR spectra were identical to those given above for *rac*-**Ie**. Found, %: C 41.22; H 3.41. $C_9H_9BrO_4$. Calculated, %: C 41.41; H 3.47.

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