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Preparation of Enantiomerically Enriched *P*-Stereogenic Dialkyl-Arylphosphine Oxides via Coordination Mediated Optical Resolution

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1. General methods (instruments)

The (−)-*O*,*O*′-dibenzoyl and(−)-*O*,*O*′-di-*p*-toluoyl-(2*R*,3*R*)-tartaric acids, calcium oxide, cobalt(II) acetate, copper(II) acetate and nickel(II) acetate were purchased from Sigma Aldrich Ltd. The phosphine oxides (**1**-**7**) were synthesized as described in the literature [1-6].

The solvents were purchased from commercial sources, and they were used without further purification.

The ^{31}P, ^{13}C, ^{1}H NMR spectra were taken on a Bruker AV-300 or DRX-500 spectrometer operating at 121.5, 75.5 and 300 or 202.4, 125.7 and 500 MHz, respectively.

The chemical shifts (δ) are given in parts per million (ppm). The chemical shifts (δ) for ^{1}H and ^{13}C in $CDCl_3$ and referenced to 7.26 and 77.16 ppm, respectively. 85% Solution of H_3PO_4 was the external reference for ^{31}P NMR chemical shifts.

Coupling constants are expressed in Hertz (Hz). The following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quadruplet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, dq = doublet of quadruplets.

The exact mass measurements were performed using an Agilent 6230C TOF LCMS System with Agilent Jet Stream source in positive ESI mode (Buffer: ammonium-formate in water / acetonitrile; Drying gas: 325 °C; Capillary: 3000 V; Fragmentor 100 V).

Thin layer chromatography (TLC) was performed on Merck pre-coated Silica gel 60 F_{254} aluminium plates with realization by UV irradiation.

The enantiomeric excess (*ee*) values were determined by chiral HPLC on a Perkin Elmer Series 200 instrument equipped with Phenomenex Lux ® 5μm Cellulose-1 or Phenomenex Lux ® 5μm Cellulose-2 or Phenomenex Lux ® 5μm Amylose-2 column (250 × 4.6 mm ID, a mixture of hexane-ethanol as the eluent with a flow rate of 0.8 mL/min, UV detector α = 254 nm). The exact chromatographic parameters are detailed in Supplementary Table 1.

Optical rotations were determined on a Perkin–Elmer 241 polarimeter.

Supplementary Table 1. HPLC parameters for the *ee* determination of dialkyl-arylphosphine oxides (**1-7**).

R[1], R[2]	Temperature (°C)	Hexane : Ethanol ratio	Retention time 1 (min)	Enantiomer 1	Retention time 2 (min)	Enantiomer 2
Me, Et (**1**)[a]	13	50:50	6.8	(*S*)	8.1	(*R*)
Me, Pr (**2**)[b]	10	90:10	17.9	(*R*)	20.1	(*S*)
Et, Pr (**3**)[b]	10	90:10	13.2	(*R*)	14.7	(*S*)
Me, Bu (**4**)[b]	20	50:50	5.5	(*R*)	6.1	(*S*)
Me, *i*-Pr (**5**)[b]	20	85:15	10.0	(*S*)	11.6	(*R*)
Me, *c*-Hex (**6**)[b]	20	85:15	11.8	(*S*)	13.7	(*R*)
Me, *t*-Bu (**7**)[c]	20	85:15	5.6	(*R*)	6.1	(*S*)

[a] Phenomenex Lux ® 5µm Cellulose-2 column.
[b] Phenomenex Lux ® 5µm Amylose-2 column
[c] Phenomenex Lux ® 5µm Cellulose-1 column

2. Preparation of the metal complexes of (–)-*O*,*O*′-dibenzoyl-(2*R*,3*R*)-tartaric acid

To a solution of 1.0 g (2.66 mmol) of DBTA H_2O in 3.0 mL of ethanol and 0.30 mL of water was added 1.33 mmol of CaO (0.074 g) or MgO (0.053 g) or $Co(OAc)_2$ (0.33 g) or $Cu(OAc)_2$ (0.24 g) or $Ni(OAc)_2$ (0.23 g). The mixture was refluxed until it became clear. The solution was cooled to room temperature, and the solvent was evaporated. The residue was dried *in vacuo* over KOH, to give 0.92 g (92%) of $Ca(H\text{-}DBTA)_2$, 0.87 g (89%) of $Mg(H\text{-}DBTA)_2$, 0.97 g (94%) of $Co(H\text{-}DBTA)_2$, 0.88 g (86%) of $Cu(H\text{-}DBTA)_2$ and 0.92 g (90%) of $Ni(H\text{-}DBTA)_2$.

3. Supplementary Resolution Procedures

3.1. Resolution of ethyl-phenyl-propylphosphine oxide (3) with a resolving agent prepared prior to the resolution (Representative Procedure)

0.096 g (0.13 mmol) of $Ca(H\text{-}DBTA)_2$ [(*R*,*R*)-**8**] prepared as described above was dissolved in 0.29 mL of boiling ethanol, and then a solution of 0.050 g (0.26 mmol) of racemic ethyl-phenyl-propylphosphine oxide (**3**) in 0.29 mL of hot ethyl acetate was added. Colorless crystalline diastereomeric complex of (*S*)-**3**·$Ca(H\text{-}DBTA)_2$ appeared upon cooling the reaction mixture to 25°C. After standing at 25°C for 24 hours, the crystals were filtered, washed with a mixture of 0.10 mL of ethanol and 0.10 mL of ethyl acetate, to give 0.10 g (83%) of (*S*)-**3**·$Ca(H\text{-}DBTA)_2$ with a *de* of 44% (Table 1, Entry 1; Supplementary Table 1, Entry 1).

Resolution of racemic ethyl-phenyl-propylphosphine oxide (**3**) was performed according to this representative procedure when the following resolving agents were used: $Mg(H\text{-}DBTA)_2$, $Co(H\text{-}DBTA)_2$, $Cu(H\text{-}DBTA)_2$ and $Ni(H\text{-}DBTA)_2$. The conditions and the results are shown in Supplementary Table 2.

Supplementary Table 2. Resolution of ethyl-phenyl-propylphosphine oxide (**3**) with the acidic Ca^{2+}, Mg^{2+}, Co^{2+}, Cu^{2+} and Ni^{2+}-salts of *O*,*O*′-dibenzoyl-(2*R*,3*R*)-tartaric acid.

Entry	Resolving agent	Eq.	Solvents[a]	Diastereomeric complex[b]	Yield[c,g] (%)	*ee*[d,g] (%)	S[e,g] (-)	Abs. Config.[f]
1	$Ca(H\text{-}DBTA)_2$	0.5	3×EtOAc/3×EtOH	(**3**)·$Ca(H\text{-}DBTA)_2$	83	44	0.36	(*S*)
2	$Mg(H\text{-}DBTA)_2$	0.5	3×EtOAc/3×EtOH	no complex	-	-	-	-
3	$Cu(H\text{-}DBTA)_2$	0.5	3×EtOAc/3×EtOH	no complex	-	-	-	-
4	$Ni(H\text{-}DBTA)_2$	0.5	3×EtOAc/3×EtOH	no complex	-	-	-	-
5	$Co(H\text{-}DBTA)_2$	0.5	3×EtOAc/3×EtOH	no complex	-	-	-	-

[a]Mixture of solvents for crystallization and recrystallizations [mL of solvent/g of resolving agent].
[b]Ratio of phosphine oxide and resolving agent was determined by ^1H NMR.

[c]Yield of the diastereomer was calculated based on the half of racemic phosphine oxide that is regarded to be 100% for each antipode.
[d]Determined by HPLC using a chiral stationary phase.
[e]Resolving capability, also known as the Fogassy parameter [S (-) = (Yield [%] /100)×(*ee* [%]/100)].
[f]Absolute configuration of phosphine oxides was determined by comparing the specific rotation with the literature data.
[g]Results obtained after the first crystallization are shown. The diastereomeric complexes were not purified.

3.2. Effect of the crystallization time on resolution of ethyl-phenyl-propylphosphine oxide (3) with 1 eq. in situ prepared Ca(H-DBTA)2 [(R,R)-8]

The resolution experiments were performed according to the representative procedure detailed in the manuscript (Section 2.2). The crystallization of the (*S*)-**3**·Ca(H-DBTA)$_2$ diastereomer was changed from 4-72 h. The conditions and results are detailed in Supplementary Table 3.

Supplementary Table 3. Effect of the crystallization time on resolution of ethyl-phenyl-propylphosphine oxide (**3**) with 1 eq. *in situ* prepared Ca(H-DBTA)$_2$ [(*R*,*R*)-**8**].

Entry	Crystallization time	Solvents[a]	Diastereomeric complex[b]	Yield[c,g] (%)	*ee*[d,g] (%)	S[e,g] (-)	Abs. Config.[f]
1	4 h	3×EtOH/3×EtOAc/0.3×H_2O	(**3**)·Ca(H-DBTA)$_2$	79	63	0.50	(*S*)
2	24 h	3×EtOH/3×EtOAc/0.3×H_2O	(**3**)·Ca(H-DBTA)$_2$	79	74	0.58	(*S*)
3	72 h	3×EtOH/3×EtOAc/0.3×H_2O	(**3**)·Ca(H-DBTA)$_2$	76	72	0.55	(*S*)

[a-g]See Supplementary Table 2 for footnotes.

4. Spectroscopic data of the scalemic dialkyl-arylphosphine oxides (1-7) prepared

Ethyl-methyl-phenylphosphine-oxide [(*R*)-**1**] (Table 3, Entry 1)

^{31}P NMR ($CDCl_3$) δ 39.2; ^{1}H NMR ($CDCl_3$) δ 7.70-7.66 (m, 2H, Ar-H), 7.52-7.44 (m, 3H, Ar-H), 1.99-1.82 (m, 2H, P-CH_2), 1.67 (d, J = 12.6, 3H, P-CH_3), 1.10 (dt, J = 7.7 and 17.7, 3H, CH_2-CH_3); ^{13}C NMR ($CDCl_3$) δ 133.3 ($^{1}J_{P\text{-}C}$ = 95.5, C_1), 131.6 ($^{4}J_{P\text{-}C}$ = 2.8, C_4), 130.1 ($^{2}J_{P\text{-}C}$ = 9.1, C_2),* 128.7 ($^{3}J_{P\text{-}C}$ = 11.4, C_3),* 24.7 ($^{1}J_{P\text{-}C}$ = 71.4, P-CH_2), 15.4 ($^{1}J_{P\text{-}C}$ = 69.5, P-CH_3), 5.7 ($^{2}J_{P\text{-}C}$ = 5.1, CH_2-CH_3), * may be reversed; HRMS $[M+H]^{+}_{found}$ = 169.0781, $C_9H_{13}OP$ requires 169.0777; pale-yellow oil; $[\alpha]_{D}^{25}$ = +16.0 (c 1.0, MeOH; ee = 66%) [7].

Methyl-phenyl-propylphosphine oxide [(*S*)-**2**] (Table 3, Entry 2)

^{31}P NMR ($CDCl_3$) δ 37.4; ^{1}H NMR ($CDCl_3$) δ 7.70-7.66 (m, 2H, Ar-H), 7.52-7.44 (m, 3H, Ar-H), 1.96-1.80 (m, 2H, P-CH_2), 1.68-1.45 (m, 5H, P-CH_3 and P-CH_2-CH_2), 0.98-0.95 (m, 3H, CH_2-CH_3); ^{13}C NMR ($CDCl_3$) δ 133.8 ($^{1}J_{P\text{-}C}$ = 95.3, C_1), 131.6 ($^{4}J_{P\text{-}C}$ = 2.8, C_4), 130.1 ($^{2}J_{P\text{-}C}$ = 9.2, C_2),* 15.5 ($^{2}J_{P\text{-}C}$ = 3.9, P-CH_2-CH_2), 128.7 ($^{3}J_{P\text{-}C}$ = 11.3, C_3),* 34.0 ($^{1}J_{P\text{-}C}$ = 70.2, P-CH_2), 16.1 ($^{1}J_{P\text{-}C}$ = 69.3, P-CH_3), 15.7 ($^{3}J_{P\text{-}C}$ = 15.4, CH_2-CH_3), * may be reversed; HRMS $[M+H]^{+}_{found}$ = 183.0932, $C_{10}H_{15}OP$ requires 183.0933; clear oil; $[\alpha]_{D}^{25}$ = -3.9 (c 3.6, $CHCl_3$; ee = 37%) [1].

Ethyl-phenyl-propylphosphine oxide [(*S*)-**3**] (Scheme 3 / III)

^{31}P NMR ($CDCl_3$) δ 41.7; ^{1}H NMR ($CDCl_3$) δ 7.58-7.51 (m, 2H, Ar-H), 7.35-7.31 (m, 3H, Ar-H), 1.90-1.63 (m, 4H, 2xP-CH_2), 1.56-1.28 (m, 2H, P-CH_2-CH_2), 1.01-0.89 (m, 3H, CH_2-CH_2-CH_3), 0.82 (dt, J = 7.4, 3.8 Hz, 3H, P-CH_2-CH_3); ^{13}C NMR ($CDCl_3$) δ 132.1 ($^{1}J_{P\text{-}C}$ = 91.6, C_1), 131.2 ($^{4}J_{P\text{-}C}$ = 2.6, C_4), 130.2 ($^{2}J_{P\text{-}C}$ = 8.7, C_2),* 128.4 ($^{3}J_{P\text{-}C}$ = 11.0, C_3),* 31.4 ($^{1}J_{P\text{-}C}$ = 68.3, P-CH_2-CH_2), 22.6 ($^{1}J_{P\text{-}C}$ = 69.2, P-CH_2-CH_3), 15.5 ($^{3}J_{P\text{-}C}$ = 14.7, CH_2-CH_2-CH_3), 15.0 ($^{2}J_{P\text{-}C}$ = 4.1, P-CH_2-CH_2), 5.3 ($^{2}J_{P\text{-}C}$ = 5.1, P-CH_2-CH_3), * may be reversed; HRMS $[M+H]^{+}_{found}$ =197.1098, $C_{11}H_{17}OP$ requires 197.1090; pale-yellow oil; $[\alpha]_{D}^{25}$ = +6.2 (c 1.0, $CHCl_3$; ee = 94%) [1].

Butyl-methyl-phenylphosphine oxide [(*S*)-**4**] (Table 3, Entry 3)

^{31}P NMR ($CDCl_3$) δ 37.7; ^{1}H NMR ($CDCl_3$) δ 7.70-7.66 (m, 2H, Ar-H), 7.52-7.44 (m, 3H, Ar-H), 1.97-1.80 (m, 2H, P-CH_2), 1.67 (d, J = 12.7, 3H, P-CH_3), 1.63-1.31 (m, 4H, P-CH_2-CH_2-CH_2), 0.85 (t, J = 7.3, 3H, CH_2-CH_3); ^{13}C NMR ($CDCl_3$) δ 133.8 ($^{1}J_{P\text{-}C}$ = 95.5, C_1), 131.6 ($^{4}J_{P\text{-}C}$ = 2.7, C_4), 130.1 ($^{2}J_{P\text{-}C}$ = 9.2, C_2)*, 128.7 ($^{3}J_{P\text{-}C}$ = 11.4, C_3),* 31.6 ($^{1}J_{P\text{-}C}$ = 70.5, P-CH_2), 24.1 ($^{3}J_{P\text{-}C}$ = 15.0, P-CH_2-CH_2-CH_2), 23.8 ($^{2}J_{P\text{-}C}$ = 4.1, P-CH_2-CH_2), 16.1 ($^{1}J_{P\text{-}C}$ = 69.5, P-CH_3), 13.6 (CH_2-CH_3), * may be reversed; HRMS $[M+H]^{+}_{found}$ =197.1093, $C_{11}H_{17}OP$ requires 197.1090; clear oil; $[\alpha]_{D}^{25}$ = - 16.3 (c 0.5, $CHCl_3$; ee = 96%) [8].

Methyl-phenyl-*i*-propylphosphine oxide [(*R*)-**5**] (Table 3, Entry 5)

^{31}P NMR ($CDCl_3$) δ 43.5; ^{1}H NMR ($CDCl_3$) δ 7.73 – 7.68 (m, 2H, Ar-H), 7.54 – 7.47 (m, 3H, Ar-H), 2.09 – 1.97 (m, 1H, C*H*-(CH_3)$_2$), 1.70 (d, J = 12.4, 3H, P-CH_3), [1.24 – 1.18 (m, 3H) and 1.10 – 1.05 (m, 3H)] CH-(CH_3)$_2$; ^{13}C NMR ($CDCl_3$) δ 132.3 ($^{1}J_{P\text{-}C}$ = 92.7, C_1), 131.3 ($^{4}J_{P\text{-}C}$ = 2.8, C_4), 130.2 ($^{2}J_{P\text{-}C}$ = 8.7, C_2), 128.3 ($^{3}J_{P\text{-}C}$ = 11.0, C_3), 29.3 ($^{1}J_{P\text{-}C}$ = 71.5, *C*H-(CH_3)$_2$), [15.2 ($^{2}J_{P\text{-}C}$ = 2.4 Hz) and 15.0 ($^{2}J_{P\text{-}C}$ = 2.4)] CH-(*C*H_3)$_2$, 12.7 ($^{1}J_{P\text{-}C}$ = 67.8, P-CH_3); HRMS $[M+H]^{+}_{found}$ = 183.0936, $C_{10}H_{16}PO$ requires 183.0841; clear oil; $[\alpha]_{D}^{25}$ = +11.3 (c 0.7, MeOH; ee = 53%) [9].

c-Hexyl-methyl-phenylphosphine oxide (**6**)

^{31}P NMR ($CDCl_3$) δ 41.0; ^{1}H NMR ($CDCl_3$) δ 7.69-7.64 (m, 2H, Ar-H), 7.52-7.44 (m, 3H, Ar-H), 1.96-1.91 (m, 1H, P-CH, 1 x *c*Hex-H), 1.84-1.61 (m, 7H, 4x *c*Hex-H, P-CH_3), 1.40-1.14 (m, 6H, 6 x *c*Hex-H); ^{13}C NMR ($CDCl_3$) δ 132.7 ($^{1}J_{P\text{-}C}$ = 92.6, C_1), 131.5 ($^{4}J_{P\text{-}C}$ = 2.6, C_4), 130.5 ($^{2}J_{P\text{-}C}$ = 8.6, C_2),* 128.5 ($^{3}J_{P\text{-}C}$ = 11.0, C_3),* 39.7 ($^{1}J_{P\text{-}C}$ = 71.8, C_1'), [26.3 ($^{3}J_{P\text{-}C}$ = 3.6) and 26.2 ($^{3}J_{P\text{-}C}$ = 3.6) 2 x C_3'], 25.7 (C_4'), 25.1 - 25.0 (m, 2 x C_2'), 12.9 ($^{1}J_{P\text{-}C}$ = 67.8, P-CH_3), * may be reversed; HRMS $[M+H]^{+}_{found}$ =223.1244, $C_{13}H_{20}OP$ requires 223.1254; white solid; mp. 98-99°C.

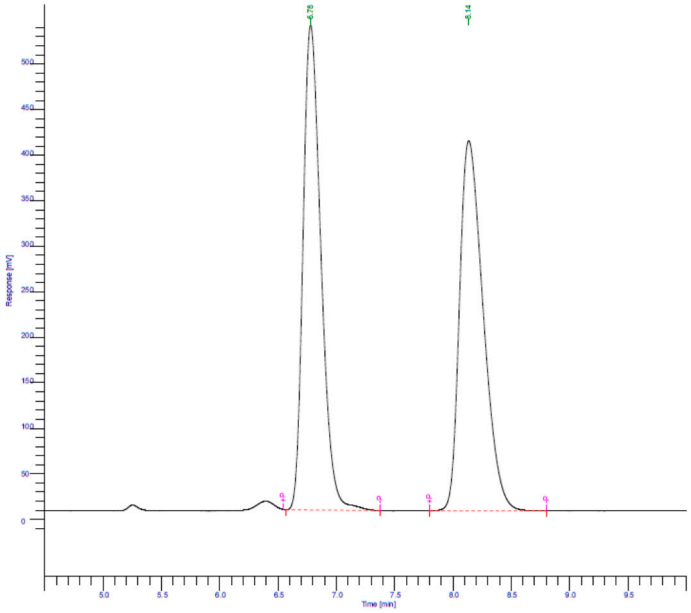
t-Butyl-methyl-phenylphosphine oxide [(*R*)-**7**] (Table 3, Entry 7)

^{31}P NMR ($CDCl_3$) δ 47.8; ^{1}H NMR ($CDCl_3$) δ 7.71-7.67 (m, 2H, Ar-H), 7.52-7.43 (m, 3H, Ar-H), 1.70 (d, J = 12.1 Hz, 3H, P-CH_3), 1.10 (d, J = 14.8 Hz, 9H, C(CH_3)$_3$); ^{13}C NMR ($CDCl_3$) δ 131.6 ($^{2}J_{P\text{-}C}$ = 8.2, C_2), 131.6 ($^{4}J_{P\text{-}C}$ = 2.6, C_4), 131.6 ($^{1}J_{P\text{-}C}$ = 90.0, C_1), 128.3 ($^{3}J_{P\text{-}C}$ = 10.9, C_3), 32.6 ($^{1}J_{P\text{-}C}$ = 70.9, P*C*(CH_3)$_3$), 24.3 (C(*C*H_3)$_3$), 10.3 ($^{1}J_{P\text{-}C}$ = 66.0, P-CH_3); HRMS $[M+H]^{+}_{found}$ = 197.1096, $C_{11}H_{17}OP$ requires 197.1095; white solid; mp. 97-98°C; $[\alpha]_{D}^{25}$ = +0.8 (c 1.2, MeOH; ee = 3%) [10].

5. HPLC traces of the optically active dialkyl-arylphosphine oxides (1-7)

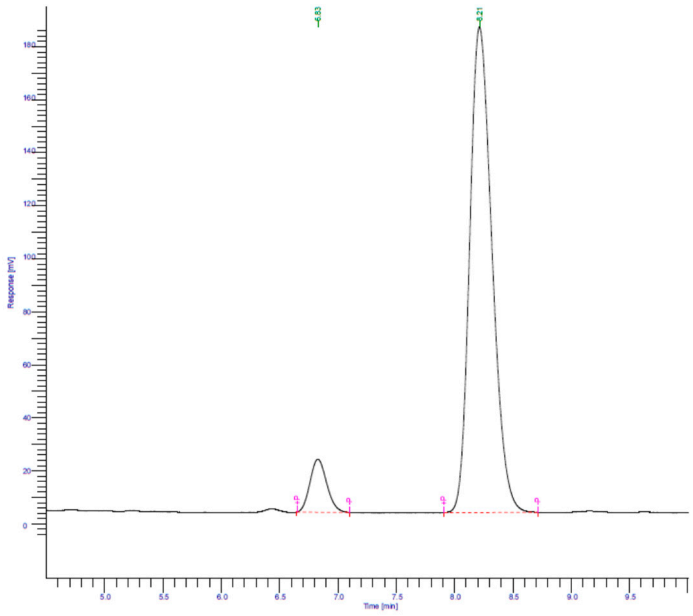
Ethyl-methyl-phenylphosphine-oxide (**1**)
Racemic **1**

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	6.780	5687421.61	532456.73	49.84	49.84	*BB	10.6815
2	8.136	5723967.31	406408.84	50.16	50.16	*BB	14.0843
		11411388.92	938865.57	100.00	100.00		



(*R*)-**1** (Table 3, Entry 1/IV.)

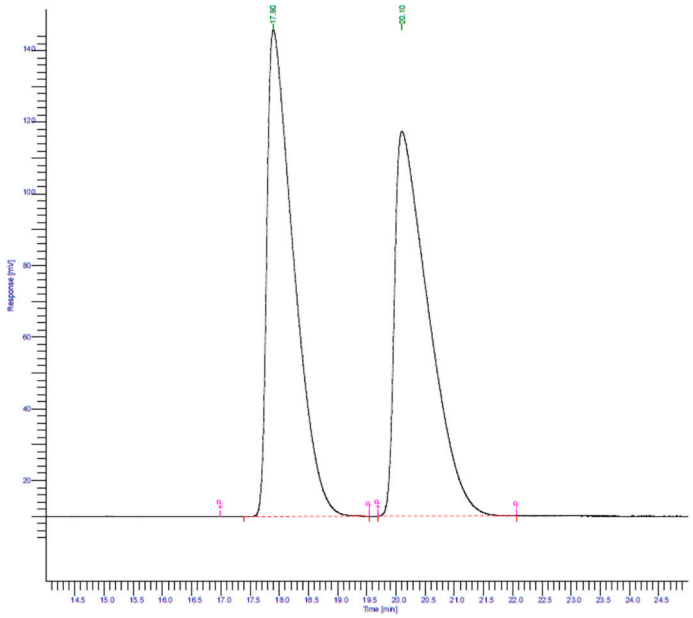
Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	6.828	200604.16	20042.55	7.61	7.61	*BB	10.0089
2	8.214	2434661.25	183115.18	92.39	92.39	*BB	13.2958
		2635265.41	203157.73	100.00	100.00		



Methyl-phenyl-propylphosphine oxide (**2**)

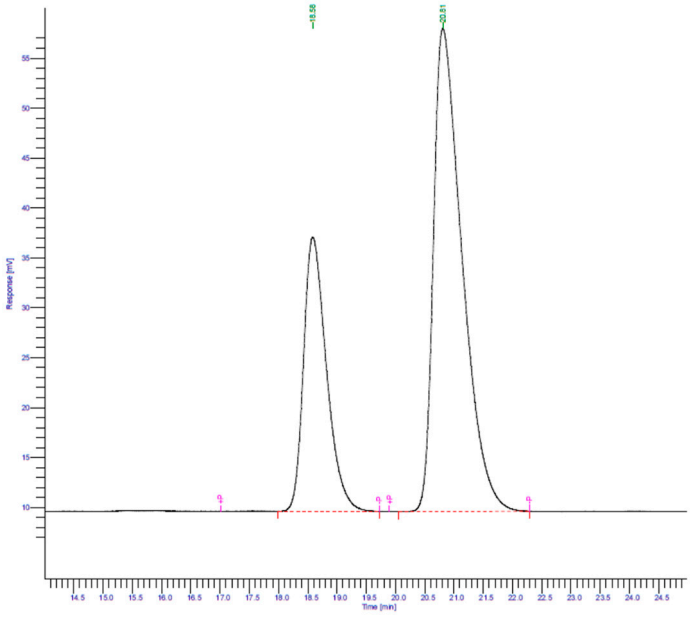
Racemic **2**

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	17.900	4320311.83	135839.87	49.97	49.97	*BB	31.8044
2	20.103	4325913.28	107450.66	50.03	50.03	*BB	40.2595
		8646225.11	243290.53	100.00	100.00		



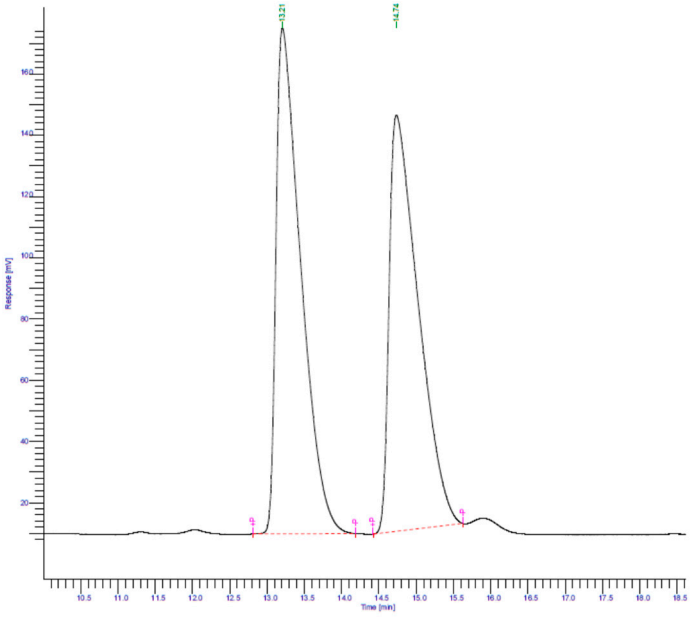
(*S*)-**2** (Table 3, Entry 2/I.)

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	18.585	748807.08	27482.70	31.24	31.24	*BB	27.2465
2	20.809	1648108.25	48385.81	68.76	68.76	*BB	34.0618
		2396915.33	75868.51	100.00	100.00		



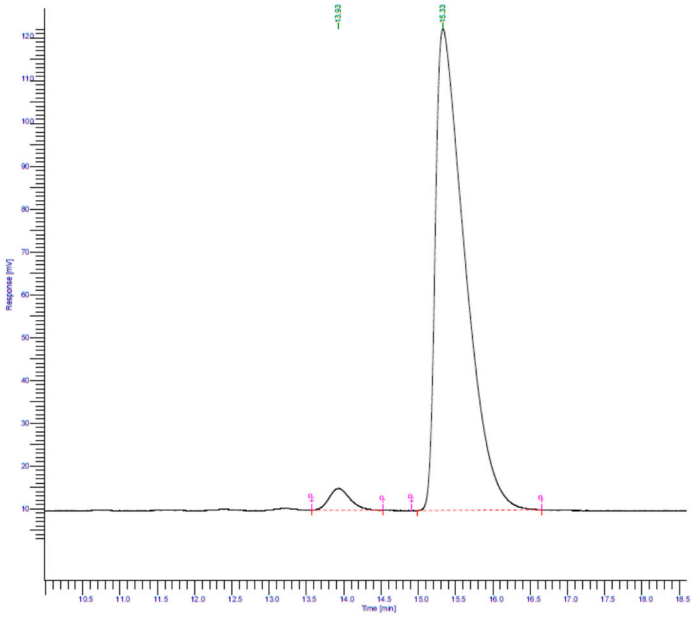
Ethyl-phenyl-propylphosphine oxide (**3**)
Racemic **3**

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	13.206	3814394.65	165165.68	50.91	50.91	*BB	23.0944
2	14.735	3677759.16	135937.08	49.09	49.09	*BB	27.0549
		7492153.81	301102.76	100.00	100.00		



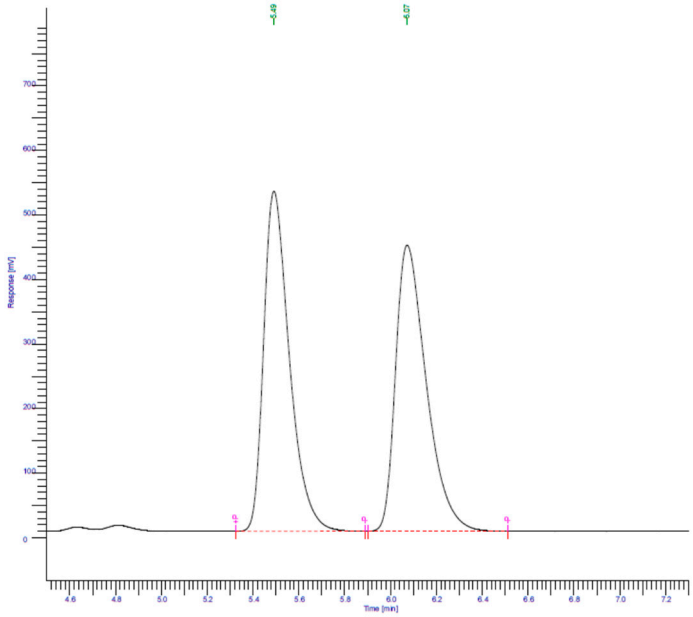
(*S*)-**3** (Scheme 3 / III)

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	13.929	100720.12	5101.22	3.16	3.16	*BB	19.7443
2	15.330	3088413.76	112419.65	96.84	96.84	*BB	27.4722
		3189133.88	117520.87	100.00	100.00		



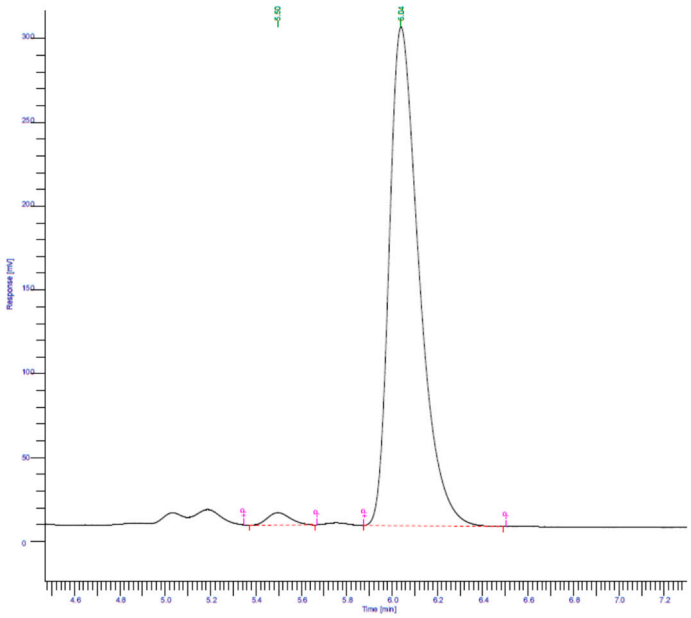
Butyl-methyl-phenylphosphine oxide (**4**)
Racemic **4**

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	5.491	4106275.88	526898.40	49.93	49.93	*BB	7.7933
2	6.073	4117175.98	443175.86	50.07	50.07	*BB	9.2902
		8223451.86	970074.26	100.00	100.00		



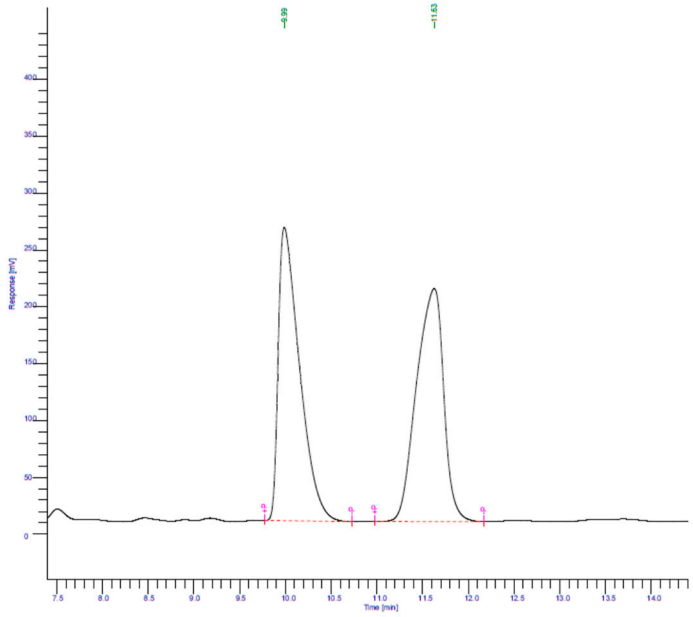
(*S*)-**4** (Table 3, Entry 3/II.)

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	5.496	51806.15	7459.33	1.87	1.87	BB	6.9451
2	6.040	2720302.83	297593.19	98.13	98.13	BB	9.1410
		2772108.97	305052.52	100.00	100.00		



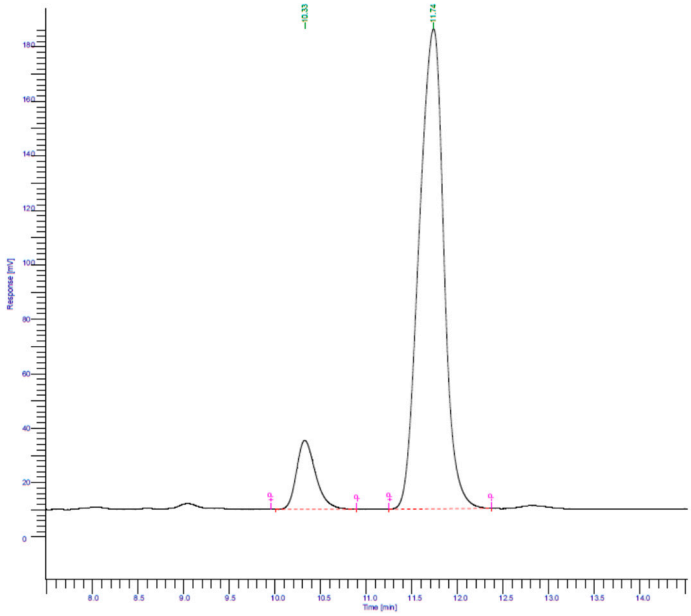
Methyl-phenyl-*i*-propylphosphine oxide (**5**)
Racemic **5**

Peak #	Time [min]	Area [μV·s]	Height [μV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	9.988	4163314.61	258140.63	49.62	49.62	*BB	16.1281
2	11.627	4227673.00	204964.52	50.38	50.38	*BB	20.6264
		8390987.61	463105.15	100.00	100.00		



(*R*)-**5** (Table 3, Entry 5/IV.)

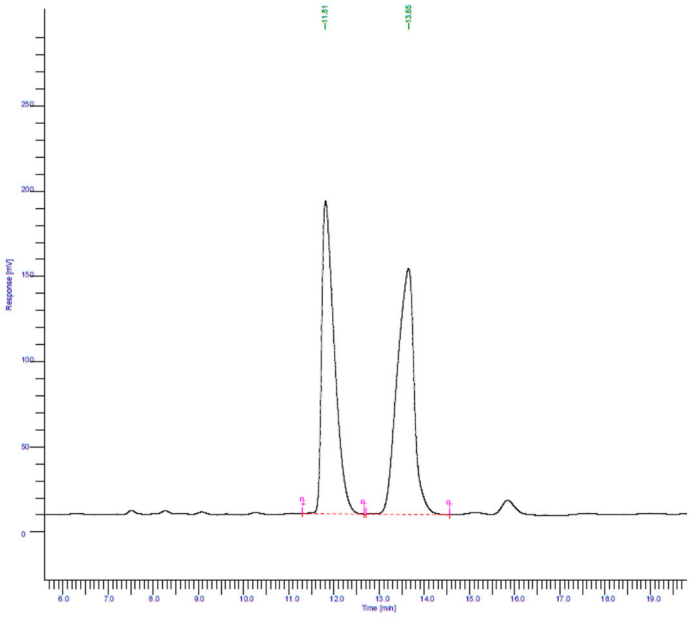
Peak #	Time [min]	Area [μV·s]	Height [μV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	10.325	367862.18	25329.82	10.00	10.00	*BB	14.5229
2	11.737	3308930.46	176312.82	90.00	90.00	*BB	18.7674
		3676792.64	201642.64	100.00	100.00		



c-Hexyl-methyl-phenylphosphine oxide (**6**)

Racemic **6**

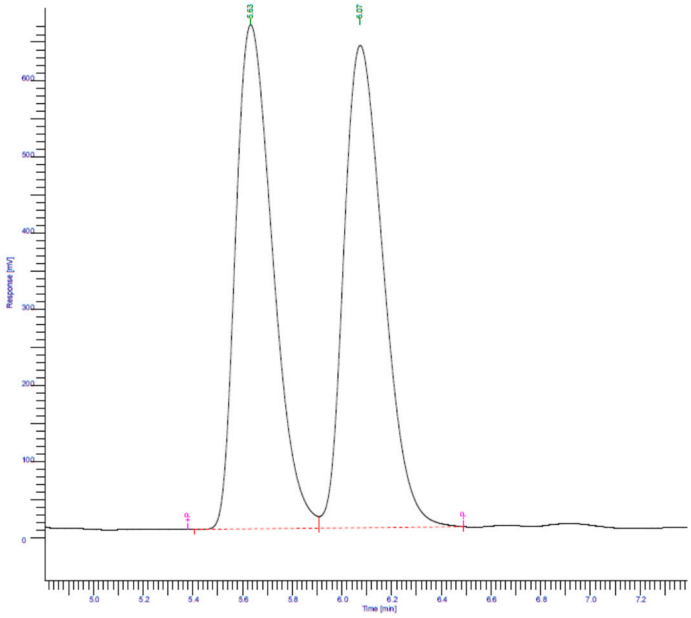
Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	11.813	3566011.68	183656.27	48.64	48.64	*BB	19.4168
2	13.652	3765983.84	144353.58	51.36	51.36	*BB	26.0886
		7331995.52	328009.85	100.00	100.00		



t-Butyl-methyl-phenylphosphine oxide (**7**)

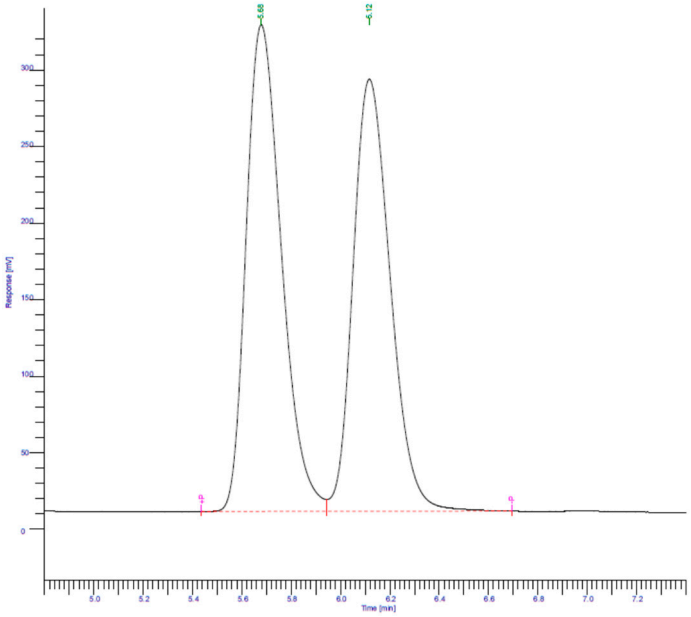
Racemic **7**

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	5.633	6848811.22	660630.98	49.51	49.51	BV	10.3671
2	6.074	6984842.84	632080.89	50.49	50.49	*VB	11.0506
		13833654.06	1.29e+06	100.00	100.00		



(*R*)-**7** (Table 3, Entry 7/II.)

Peak #	Time [min]	Area [µV·s]	Height [µV]	Area [%]	Norm. Area [%]	BL	Area/Height [s]
1	5.679	3148903.11	317733.79	51.59	51.59	BV	9.9105
2	6.117	2954401.44	282136.40	48.41	48.41	*VB	10.4715
		6103304.55	599870.19	100.00	100.00		



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