Lewis Base-catalyzed Conjugate Reduction and Reductive Aldol Reaction of α,β-Unsaturated Ketones Using Trichlorosilane

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Supplementary Information

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General Methods

Melting points (Mp) are uncorrected. ^{1}H and ^{13}C NMR spectra were measured in $CDCl_3$ with JEOL JNM-ECX400 spectrometer. Tetramethylsilane (TMS) (δ = 0 ppm) and $CDCl_3$ (δ = 77.0 ppm) served as internal standards for ^{1}H and ^{13}C NMR, respectively. Infrared spectra were recorded on JASCO IR Report-100. Mass spectra were measured with JEOL JMS-DX303HF mass spectrometer. Optical rotations were recorded on JASCO P-1010 polarimeter. High-pressure liquid chromatography (HPLC) was performed on JASCO P-980 and UV-1575.

Thin-layer chromatography (TLC) analysis was carried out using Merck silica gel plates. Visualization was accomplished with UV light, phosphomolybdic acid and/or anisaldehyde. Column chromatography was performed using Kanto Chemical Silica Gel 60N (spherical, neutral, 63-210 μm).

All reactions were performed under argon atmosphere using oven- and heating gun-dried glassware equipped with a rubber septum and a magnetic stirring bar.

Solvents and Chemicals

Dry dichloromethane (dehydrated) was purchased from Kanto Chemical and stored over 4Å MS prior to use. All other solvents were purified based on standard procedures.

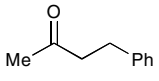
Trichlorosilane was purchased from Tokyo Kasei Kogyo (TCI) and used without further purification. (*S*)-BINAP dioxide (BINAPO) was prepared by oxidation of (*S*)-BINAP with hydrogen peroxide in acetone.[1] All other chemicals were purified based on standard procedures.

Starting materials: (*E*)-4-Phenyl-3-buten-2-one (benzalacetone, **1a**), (*E*)-1,3-diphenyl-2-propen-1-one (chalcone, **1b**), α- and β-ionone (**1e** and **1f**) and (*R*)-(+)-pulegone (**1k**) were obtained commercially and used after purification by standard procedures. (*E*)-1-Phenyl-2-buten-1-one (**1c**) was prepared by oxidation of (*E*)-1-phenyl-2-buten-1-ol with MnO_2. Dibenzylideneacetone (**1d**), 2-benzylidenecyclohexanone (**1i**) and 2,6-dibenzylidenecyclohexanone (**1j**) were prepared by aldol condensation.[2] 2-Methyl-1-phenyl-2-propen-1-one (**1h**) was prepared by Mannich reaction of propiophenone with hexamethylenetetramine and acetic anhydride. (*E*)-1,3-Diphenyl-2-methyl-2-propen-1-one (**1g**) was prepared according to the literatures.[3]

General Procedure for Conjugate Reduction

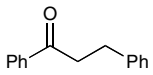
To a solution of a Lewis base [HMPA, $Ph_3P=O$ (20 mol%) or (*S*)-BINAPO (10 mol%)] and an enone (1.0 or 0.5 mmol) in dry dichloromethane (2 or 1 mL) was added dropwise trichlorosilane (1.5~3 M CH_2Cl_2 solution, 2 equiv) at 0 °C. The reaction was monitored by TLC analysis. For the reactions of less reactive enones, the temperature was raised to ambient temperature. After the starting material was consumed or no significant change was observed, the reaction was quenched with sat. aqueous $NaHCO_3$ (3 mL). After addition of ethyl acetate (10 mL), the mixture was stirred for 1 h, filtered through a Celite pad with ethyl acetate and extracted with ethyl acetate (3×). The combined organic layers were washed with brine (1×), dried over anhydrous $MgSO_4$, filtered, evaporated and purified by silica gel column chromatography (hexane/ethyl acetate = 20/1~8/1) to give a reduction product.

4-Phenylbutan-2-one (2a) (Table 1, entry 1)



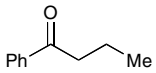
According to the general procedure, the reaction of benzalacetone (**1a**) (145.6 mg) with HMPA and $HSiCl_3$ (0 °C, 30 min) gave the reduction product (127.7 mg, 87% yield). The product was identified in comparison with a commercially available sample.

1,3-Diphenylpropan-1-one (Table 1, entry 2)



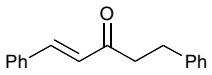
According to the general procedure, the reaction of chalcone (**1b**) (208.3 mg) with HMPA and $HSiCl_3$ (0 °C, 30 min) gave the reduction product (191.7 mg, 91% yield). The product was identified in comparison with a commercially available sample.

1-Phenylbutan-1-one (butyrophenone) (Table 1, entry 3)



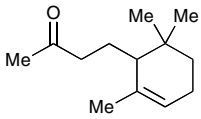
According to the general procedure, the reaction of (*E*)-1-phenyl-2-buten-1-one (**1c**) (74.8 mg) with HMPA and $HSiCl_3$ (0 °C, 30 min) gave the reduction product (54.3 mg, 72% yield). The product was identified in comparison with a commercially available sample.

(*E*)-1,5-Diphenyl-1-penten-3-one (Table 1, entry 4)



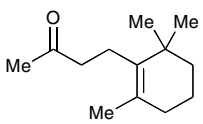
According to the general procedure, the reaction of dibenzylideneacetone (**1d**) (111.7 mg) with HMPA and $HSiCl_3$ (0 °C, 60 min) gave the reduction product (89.7 mg, 80% yield). Spectroscopic data were consistent with the literature data.[4]

4-(2,6,6-Trimethyl-2-cyclohexen-1-yl)butan-2-one (Table 1, entry 5)



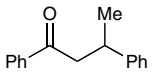
According to the general procedure, the reaction of α-ionone (**1e**) (192.2 mg) with HMPA and $HSiCl_3$ (0 °C, 4 h then rt, 2 h) gave the reduction product (180.7 mg, 93% yield). Spectroscopic data were consistent with the literature data.[4]

4-(2,6,6-Trimethyl-1-cyclohexen-1-yl)butan-2-one (Table 1, entry 6)



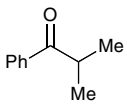
According to the general procedure, the reaction of β-ionone (**1f**) (192.3 mg) with HMPA and $HSiCl_3$ (0 °C, 30 min) gave the reduction product (182.2 mg, 94% yield). Spectroscopic data were consistent with the literature data.[4]

(±)-1,3-Diphenylbutan-1-one (Table 1, entry 7)



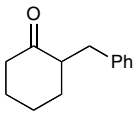
According to the general procedure, the reaction of 1,3-diphenyl-2-methyl-2-propen-1-one (**1g**) (112.0 mg) with HMPA and $HSiCl_3$ (0 °C, 5 h then rt, 19 h) gave the reduction product (83.4 mg, 74% yield). Spectroscopic data were consistent with the literature data.[5]

2-Methyl-1-Phenylpropan-1-one (isobutyrophenone) (Table 1, entry 8)



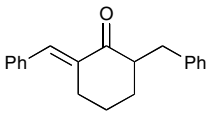
According to the general procedure, the reaction of 2-methyl-1-phenyl-2-propen-1-one (**1h**) (65.8 mg) with HMPA and $HSiCl_3$ (0 °C, 9 h then rt, 17 h) gave the reduction product (39.7 mg, 60% yield). The product was identified in comparison with a commercially available sample.

2-Benzylcyclohexanone (Table 1, entry 9)



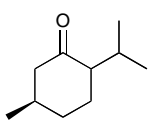
According to the general procedure, the reaction of 2-benzylidenecyclohexanone (**1i**) (92.6 mg) with HMPA and $HSiCl_3$ (0 °C, 30 min) gave the reduction product (83.5 mg, 89% yield). The product was identified in comparison with a commercially available sample.

2-Benzylidene-6-benzylcyclohexanone (Table 1, entry 10)



According to the general procedure, the reaction of 2,6-dibenzylidenecyclohexanone (**1j**) (137.5 mg) with HMPA and $HSiCl_3$ (0 °C, 40 min) gave the reduction product (114.0 mg, 82% yield). Slightly yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.50-1.65 (m, 2H), 1.83-2.00 (m, 2H), 2.58-2.73 (m, 3H), 2.94-3.04 (m, 1H), 3.40 (apparent q, J = 8.9 Hz, 1H), 7.18-7.24 (m, 3H), 7.26-7.36 (m, 3H), 7.36-7.42 (m, 4H), 7.46 (t, J = 2.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.8, 28.4, 29.2, 36.9, 51.2, 126.2, 128.49, 128.58, 129.4, 130.3, 135.4, 135.9, 137.5, 140.3, 203.4; HRMS (FAB): calcd for $C_{20}H_{21}O$ ($M+H^+$) 277.1592, found 277.1590; Elemental Analysis calcd (%) for $C_{20}H_{20}O$: C 86.92, H 7.29; found: C 86.73, H 7.41.

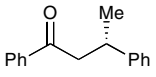
(5*R*)-2-Isopropyl-5-methylcyclohexanone (Table 1, entry 11)



According to the general procedure, the reaction of (*R*)-(+)-pulegone (**1k**) (151.7 mg) with HMPA and $HSiCl_3$ (0 °C, 4 h then rt, 16 h) gave the reduction

product (128.6 mg, 84% yield) as a diastereomeric mixture (*trans*/*cis* = 1.7/1). Spectroscopic data were consistent with a commercially available sample [(–)-menthone] and the literature data.[4]

(*S*)-1,3-Diphenylbutan-1-one (Scheme 2)

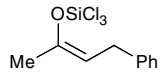


According to the general procedure, the reaction of 2-methyl-1-phenyl-2-propen-1-one (**1g**) (111.8 mg) with (*S*)-BINAPO and $HSiCl_3$ (0 °C, 20 h) gave the reduction product (109.7 mg, 97% yield) with 97% ee (*S*). Spectroscopic data were consistent with the reported data and the absolute configuration was assigned in comparison of the optical rotation with the literature value.[5] Colourless liquid; $[\alpha]^{23}_{D}$ +14.7 (c 1.005, CCl_4) for 97% ee (*S*) [lit.[5] $[\alpha]^{25}_{D}$ –13.5 (c 1.00, CCl_4) for 82% ee (*R*)]; HPLC (a combination of CHIRALPAK AD-H and AS-H, 0.46 cmø × 25 cmL each, hexane/2-propanol = 39/1, flow rate 1.0 mL/min, UV detection at 254 nm) t_R = 14.4 min (*S*), 15.8 min (*R*).

^{1}H NMR Analysis of Reduction of Benzalacetone with Trichlorosilane

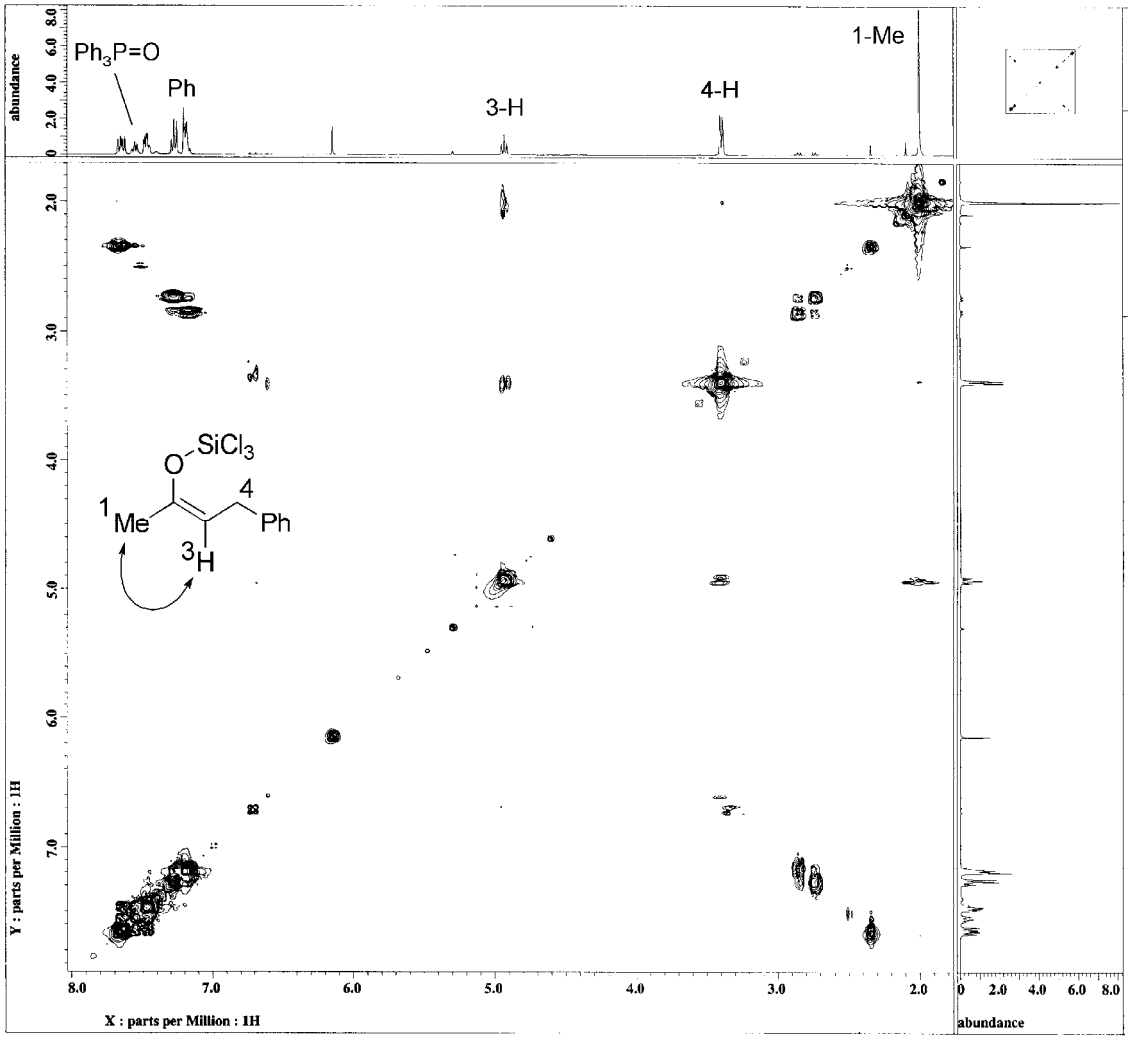
To a solution of benzalacetone (27.5 mg) and Ph_3P=O (10.5 mg) in CD_2Cl_2 (0.6 mL) in a NMR tube capped with a rubber septum was added trichlorosilane (38 μL). Gradual formation of the corresponding (*Z*)-trichlorosilyl enolate was observed by ^{1}H NMR analysis. After 4 h, almost the starting material (>95%) was consumed. The geometry of the enolate was assigned based on a NOESY analysis of the reaction mixture (Chart 1).

(*Z*)-2-(Trichlorosilyloxy)-4-phenyl-2-butene



^{1}H NMR (400 MHz, CD_2Cl_2, internal standard: solvent residual peak = 5.32 ppm) δ 2.35 (s, 3H), 3.41 (d, *J* = 7.3 Hz, 2H), 4.95 (t, *J* = 7.3 Hz, 1H), 7.15-7.24 (m, 3H), 7.24-7.33 (m, 2H).

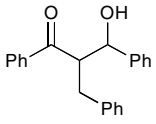
Chart 1. The NOESY spectrum of the trichlorosilyl enolate derived from benzalacetone.



General Procedure for Reductive Aldol Reaction

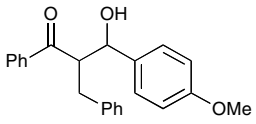
To a solution of a Lewis base [HMPA, Ph_3P=O (20 mol%) or (*S*)-BINAPO (10 mol%)], an enone (0.5 mmol) and an aldehyde (0.6 mmol, 1.2 equiv) in dry dichloromethane (2 mL) was added dropwise trichlorosilane (ca. 3 M CH_2Cl_2 solution, 2 equiv) at the indicated temperature. The reaction was monitored by TLC analysis. After the enone was consumed or no significant change was observed, the reaction was quenched with sat. aqueous $NaHCO_3$ (3 mL). After addition of ethyl acetate (10 mL), the mixture was stirred for 1 h, filtered through a Celite pad with ethyl acetate and extracted with ethyl acetate (3×). The combined organic layers were washed with brine (1×), dried over anhydrous $MgSO_4$, filtered, evaporated and purified by silica gel column chromatography (hexane/ethyl acetate = 20/1~3/1) to give the corresponding aldol product.

2-Benzyl-1,3-diphenyl-3-hydroxypropan-1-one (Table 2, entry 2)



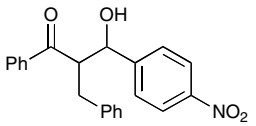
According to the general procedure, the reaction of chalcone (**1b**) (104.2 mg) and benzaldehyde (61 μL) with $Ph_3P{=}O$ and $HSiCl_3$ (0 °C, 4 h) gave the aldol product (123.3 mg, 78% yield) as a diastereomeric mixture (50:50). Spectroscopic data were consistent with the literature data.[6,7]

2-Benzyl-3-hydroxy-3-(4-methoxyphenyl)-1-phenylpropan-1-one (Table 2, entry 3)



According to the general procedure, the reaction of chalcone (**1b**) (104.2 mg) and *p*-anisaldehyde (73 μL) with $Ph_3P{=}O$ and $HSiCl_3$ (0 °C, 4 h) gave the aldol product (120.0 mg, 69% yield) as a diastereomeric mixture (52:48). Colourless viscous oil; IR (film on NaCl, cm^{-1}) 3450, 1670, 1610, 1593, 1578, 1511, 1247, 1175, 1035, 700; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.79 (dd, *J* = 13.3, 5.5 Hz, 0.48H), 2.99 (dd, *J* = 13.3, 8.9 Hz, 0.48H), 3.10 (dd, *J* = 13.6, 3.7 Hz, 0.52H), 3.17 (dd, *J* = 13.6, 10.1 Hz, 0.52H), 3.30 (brs, 0.52H), 3.36 (brs, 0.48H), 3.72 (s, 1.56H), 3.73 (s, 1.44H), 3.98-4.10 (m, 1H), 4.91 (brd, *J* = 6.4 Hz, 0.48H), 5.02 (d, *J* = 5.0 Hz, 0.52H), 6.80 (d, *J* = 9.4 Hz, 1.04H), 6.82 (d, *J* = 9.4 Hz, 0.96H), 6.95-7.43 (m, 10H), 7.49 (d, *J* = 7.3 Hz, 1.04H), 7.66 (d, *J* = 7.3 Hz, 0.96H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 33.9, 36.5, 55.0, 55.1, 55.8, 73.7, 75.3, 113.6, 113.8, 126.00, 126.24, 127.35, 127.40, 128.06, 128.14, 128.19, 128.24, 128.30, 128.86, 128.92, 132.84, 132.89, 133.72, 134.67, 137.37, 138.08, 138.57, 139.26, 158.82, 159.00, 204.6, 205.5; HRMS (FAB): calcd for $C_{23}H_{22}O_3Na$ ($M+Na^+$) 369.1467, found 369.1474.

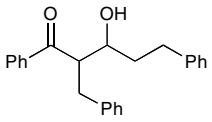
2-Benzyl-3-hydroxy-3-(4-nitrophenyl)-1-phenylpropan-1-one (Table 2, entry 4)



According to the general procedure, the reaction of chalcone (**1b**) (104.6 mg) and *p*-nitrobenzaldehyde (90.9 mg) with $Ph_3P{=}O$ and $HSiCl_3$ (0 °C, 4 h) gave the aldol product (130.7 mg, 72% yield) as a diastereomeric mixture (74:26). Colourless solid; Mp = 122-126 °C; IR (film on NaCl, cm^{-1}) 3460, 1670, 1592, 1516, 1343, 698; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.96 (dd, *J* = 13.8, 4.1 Hz, 0.26H), 3.04 (dd, *J* = 13.5, 7.8 Hz, 0.74H), 3.11 (dd, *J* = 13.5, 7.3 Hz, 0.74H), 3.17 (dd, *J* = 13.8, 10.3 Hz, 0.26H), 3.86 (d, *J* = 1.8 Hz, 0.26H), 4.00-4.12 (m, 1H), 4.38 (d, *J* = 8.2 Hz, 0.74H), 4.99 (dd, *J* = 8.2, 4.6 Hz, 0.74H), 5.20 (brd, *J* = 1.8 Hz, 0.26H), 6.92 (d, *J* = 7.1 Hz, 0.52H), 6.99-7.10 (m, 0.78H), 7.14-7.36 (m, 5.70H), 7.41-7.50 (m, 2.48H), 7.54-7.61 (m, 1.04H), 7.66 (d, *J* = 7.6 Hz, 1.48H), 8.07 (d, *J* = 8.7 Hz, 1.48H), 8.14 (d, *J* = 8.7 Hz, 0.52H); ^{13}C NMR (100

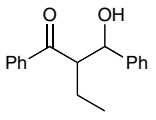
MHz, $CDCl_3$) δ 33.4, 36.4, 53.6, 54.6, 72.9, 73.6, 123.5, 126.3, 126.67, 126.76, 127.09, 128.05, 128.21, 128.32, 128.43, 128.58, 128.64, 128.83, 128.95, 133.51, 133.75, 136.62, 136.72, 137.8, 138.3, 147.03, 147.10, 149.0, 150.3, 204.4, 205.0; HRMS (FAB): calcd for $C_{22}H_{20}O_4$ ($M+H^+$) 362.1392, found 362.1385.

2-Benzyl-1,5-diphenyl-3-hydroxypentan-1-one (Table 2, entry 5)



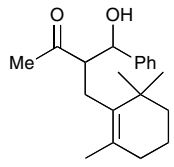
According to the general procedure, the reaction of chalcone (**1b**) (104.1 mg) and hydrocinnamaldehyde (79 μL) with $Ph_3P=O$ and $HSiCl_3$ (rt, 24 h) gave the aldol product (33.1 mg, 19% yield) as a diastereomeric mixture (52:48). Colourless viscous oil; IR (film on NaCl, cm^{-1}) 3450, 1667, 1590, 1575, 1490, 1445, 693; ^1H NMR (400 MHz, $CDCl_3$) less polar isomer (major): δ 1.63-1.82 (m, 2H), 2.54-2.63 (m, 1H), 2.79-2.90 (m, 1H), 3.07 (dd, *J* = 13.8, 6.9 Hz, 1H), 3.11 (dd, *J* = 13.8, 7.8 Hz, 1H), 3.41 (d, *J* = 9.2 Hz, 1H), 3.70-3.84 (m, 2H), 7.05-7.26 (m, 10H), 7.40 (apparent t, *J* = 7.8 Hz, 2H), 7.54 (apparent t, *J* = 7.3 Hz, 1H), 7.79 (apparent d, *J* = 8.0 Hz, 2H); polar isomer (minor): δ 1.76-1.88 (m, 1H), 1.90-2.03 (m, 1H), 2.64-2.74 (m, 1H), 2.76-2.92 (m, 2H), 3.10 (dd, *J* = 13.7, 5.0 Hz, 1H), 3.15 (dd, *J* = 13.7, 9.2 Hz, 1H), 3.78 (ddd, *J* = 9.2, 5.0, 3.7 Hz, 1H), 3.94-4.00 (m, 1H), 7.05-7.21 (m, 8H), 7.24-7.33 (m, 4H), 7.46 (apparent t, *J* = 7.3 Hz, 1H), 7.62 (apparent d, *J* = 7.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) less polar isomer: δ 32.6, 36.5, 38.2, 52.0, 71.9, 125.8, 126.5, 128.23, 128.33, 128.39, 128.52, 128.70, 129.1, 133.6, 137.3, 138.7, 141.8, 206.4; polar isomer: δ 32.4, 33.6, 36.5, 53.2, 71.5, 126.0, 126.2, 128.3, 128.4, 129.0, 133.2, 137.2, 139.4, 141.6, 205.1; HRMS (FAB): calcd for $C_{24}H_{25}O_2$ ($M+H^+$) 345.1855, found 345.1865.

2-Ethyl-1,3-diphenyl-3-hydroxypropan-1-one (Table 2, entry 6)



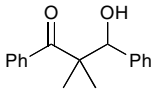
According to the general procedure, the reaction of (*E*)-1-phenyl-2-buten-1-one (**1c**) (73.4 mg) and benzaldehyde (61 μL) with $Ph_3P=O$ and $HSiCl_3$ (0 °C, 4 h) gave the aldol product (88.8 mg, 70% yield) as a diastereomeric mixture (39:61). Spectroscopic data were consistent with the literature data.[7]

4-Hydroxy-3-(2,6,6-trimethyl-1-cyclohexen-1-yl)methyl-4-phenylbutan-2-one (Table 2, entry 7) According to the general procedure, the reaction of β-ionone (**1f**) (81.4 mg) and benzaldehyde (61 μL) with $Ph_3P=O$ and $HSiCl_3$ (0 °C, 5 h)



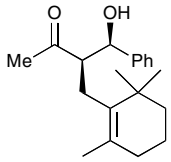
gave the aldol product (82.6 mg, 65% yield) as a diastereomeric mixture (78:22). Spectroscopic data were consistent with the literature data.[7]

2,2-Dimethyl-1,3-diphenyl-3-hydroxypropan-1-one (Table 2, entry 8)



According to the general procedure, the reaction of 2-methyl-1-phenylpropenone (**1h**) (73.5 mg) and benzaldehyde (61 μL) with HMPA and $HSiCl_3$ (rt, 24 h) gave the aldol product (50.2 mg, 39% yield). Spectroscopic data were consistent with the literature data.[8]

(3*R*,4*R*)-4-Hydroxy-3-(2,6,6-trimethyl-1-cyclohexen-1-yl)methyl-4-phenylbutan-2-one

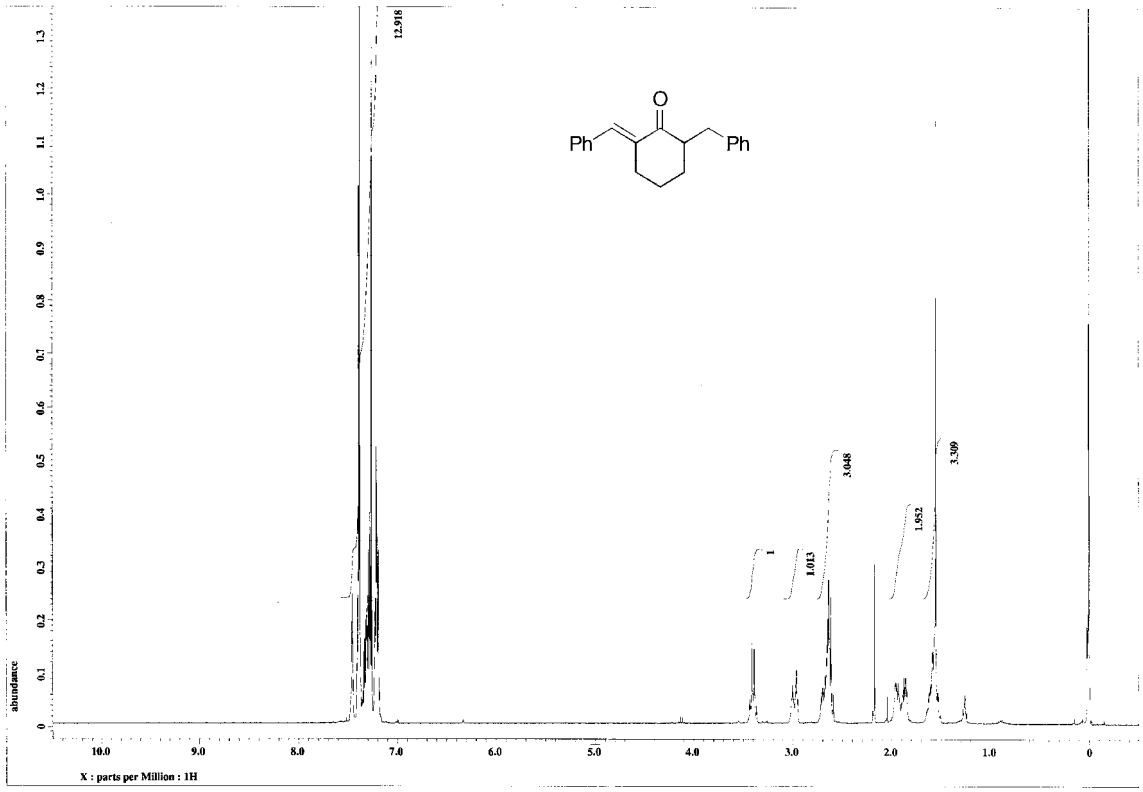


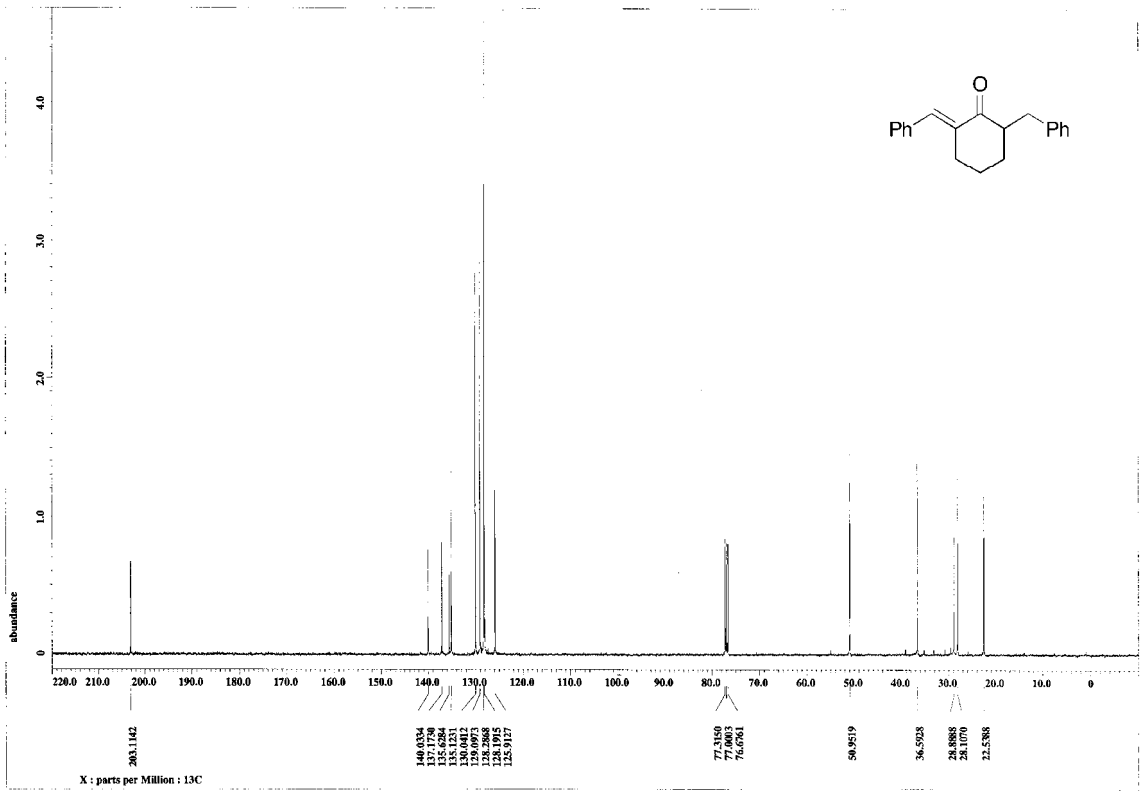
(Scheme 2) According to the general procedure, the reaction of β-ionone (**1f**) (96.5 mg) and benzaldehyde (120 μL, 2 equiv) with (*S*)-BINAPO and $HSiCl_3$ (–78 °C, 21 h) gave the *syn*-aldol product (101.0 mg, 67% yield, 96% ee) and the *anti*-aldol product including impurities (5.3 mg, 0% ee). The diastereomers were separable by column chromatography. The diastereoselectivity was determined by ^{1}H NMR analysis of the crude reaction mixture. Spectroscopic data were consistent with the reported data[6] and the absolute configuration was assigned in comparison of the optical rotation with the literature value.[6]

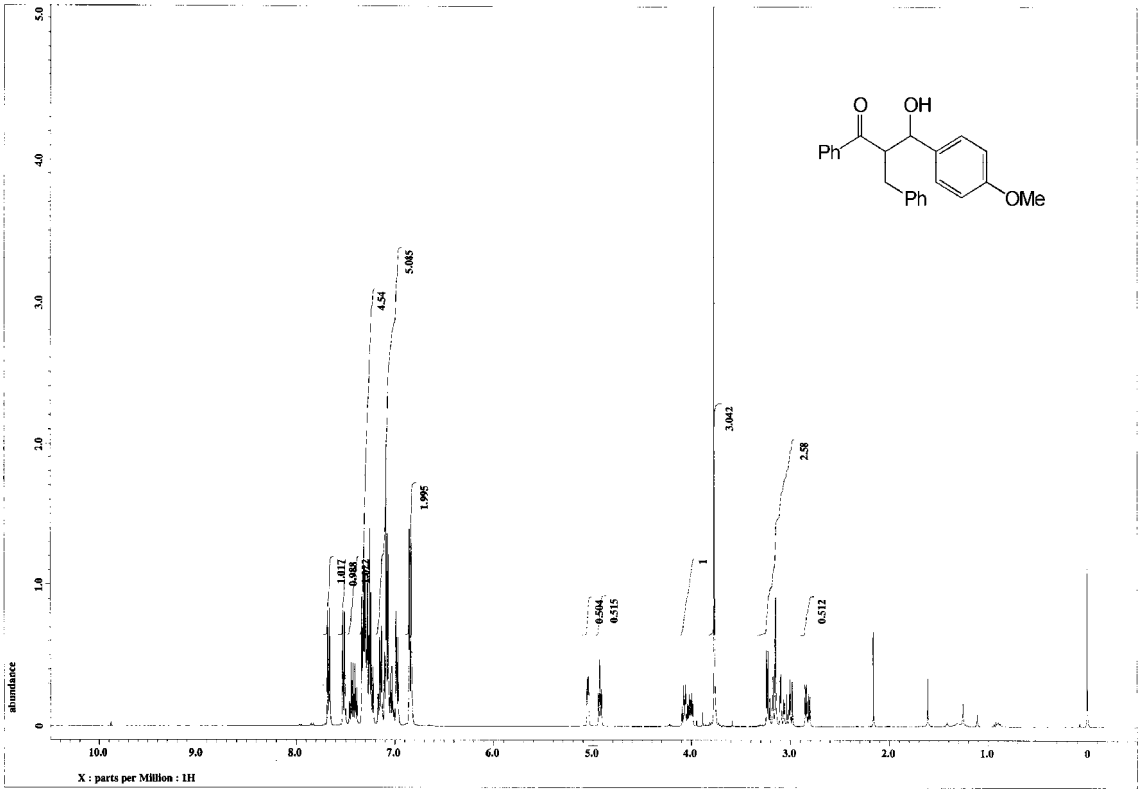
Syn-isomer: Colourless solid; $[\alpha]^{20}_D$ +56.9 (c 1.01, $CHCl_3$) for 58% ee (3*R*,4*R*) [lit[6] $[\alpha]^{20}_D$ – 57.7 (c 1.0, $CHCl_3$) for 75% ee (3*S*,4*S*)]; HPLC (a combination of two CHIRALPAK AD-H, 0.46 cmø × 25 cmL each, hexane/2-propanol = 29/1, flow rate 1.0 mL/min, UV detection at 254 nm) t_R = 17.8 min (3*S*,4*S*), 21.3 min (3*R*,4*R*).

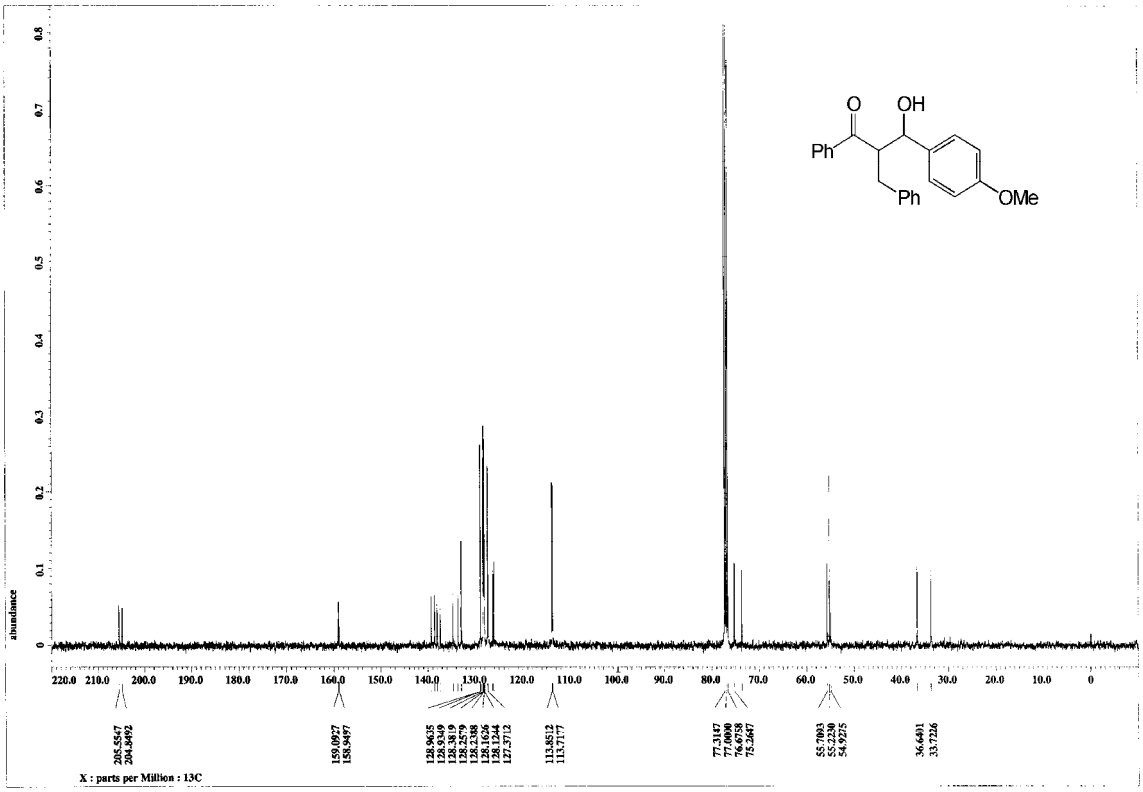
Anti-isomer: HPLC (a combination of two CHIRALPAK AD-H, 0.46 cmø × 25 cmL each, hexane/2-propanol = 29/1, flow rate 1.0 mL/min, UV detection at 254 nm) t_R = 18.4 min, 20.0 min.

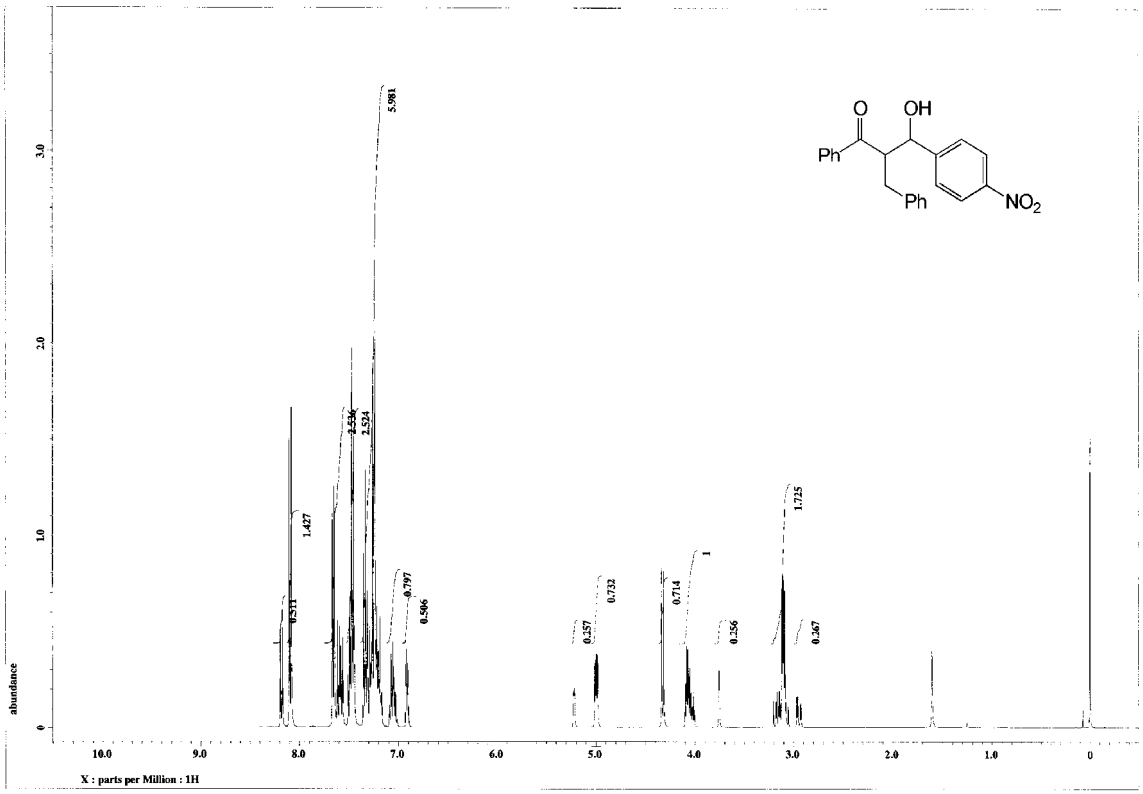
^{1}H and ^{13}C NMR Spectra of All New Compounds

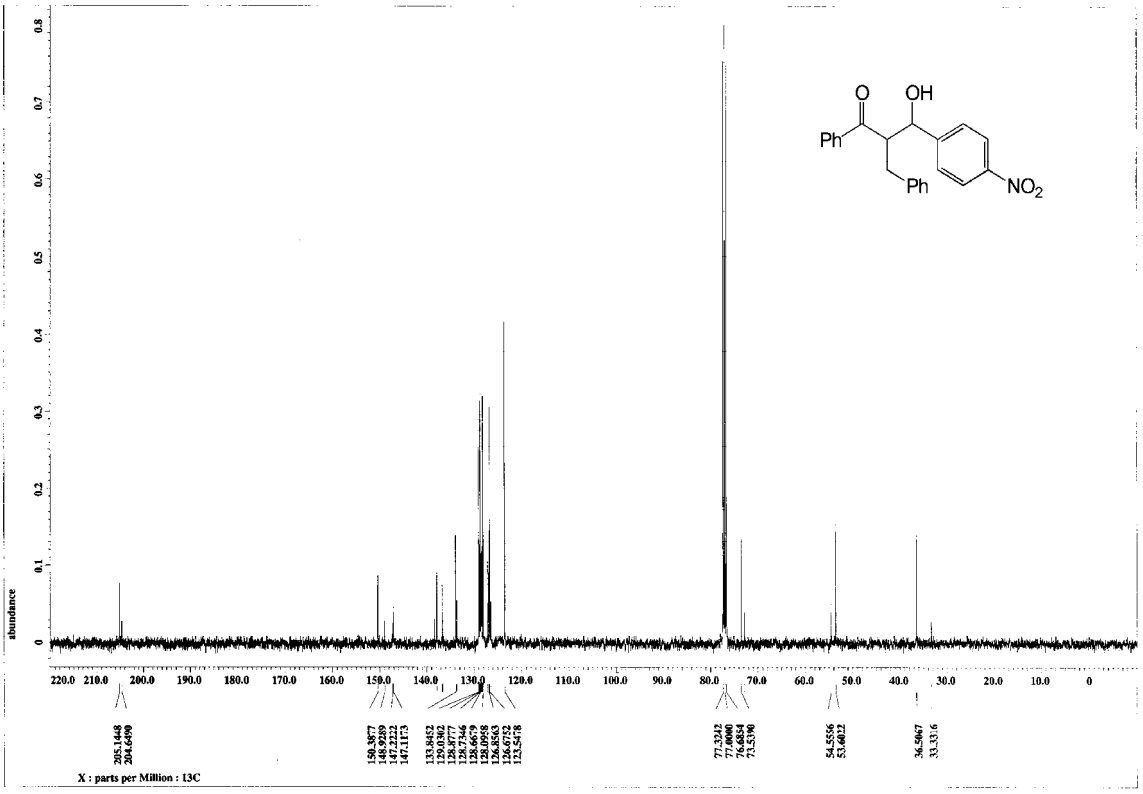


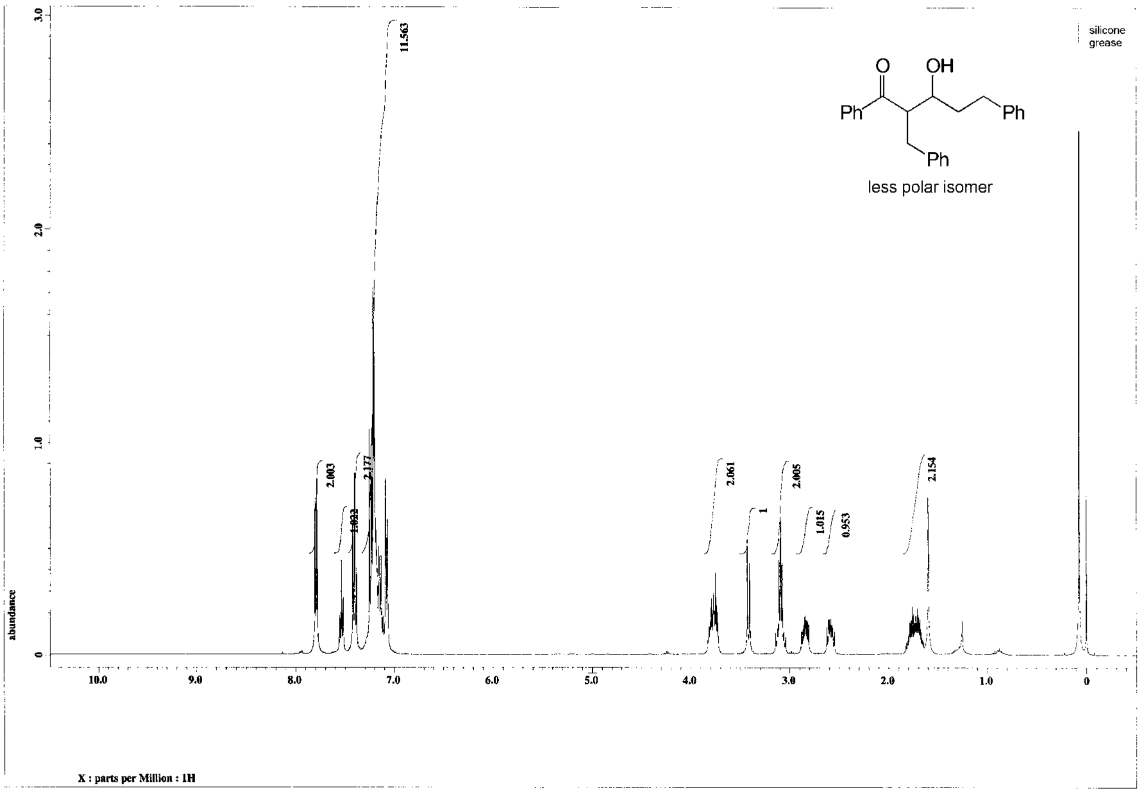


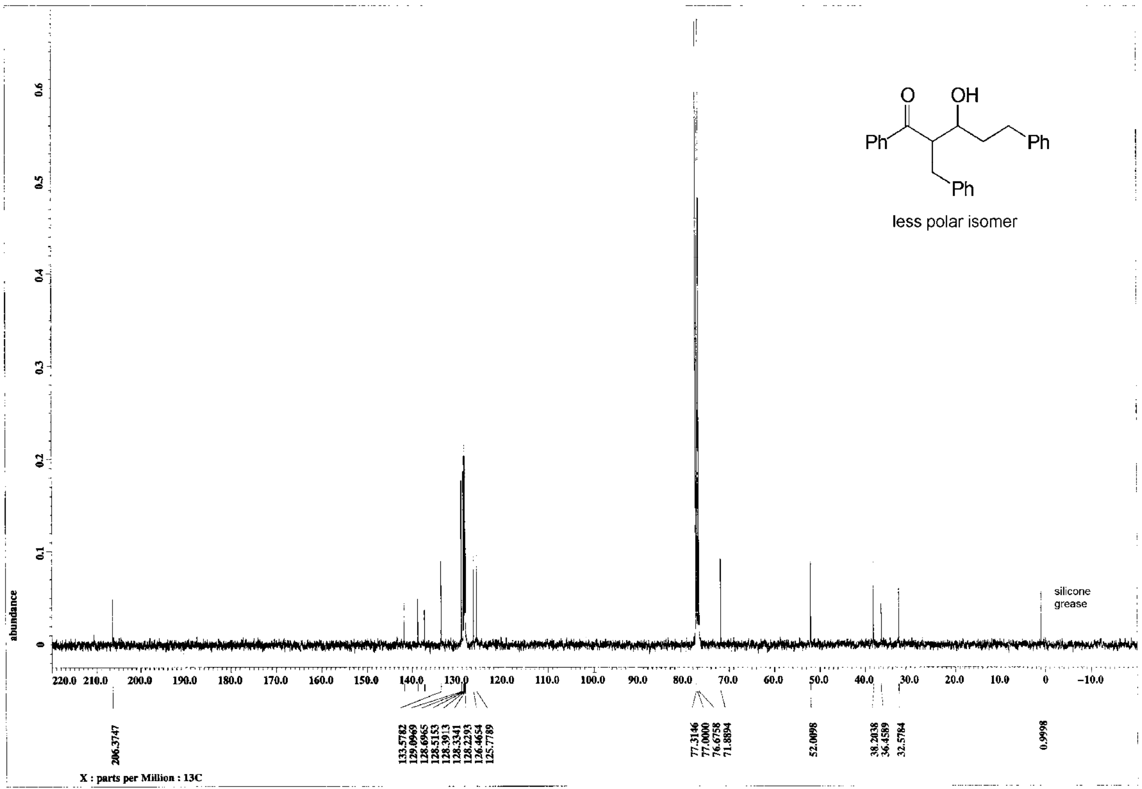

O
OH
Ph
Ph
OMe
5.045
4.54
3.042
2.58
1.995
1.017
0.988
0.972
0.504
0.515
1
0.512
abundance
X : parts per Million : 1H

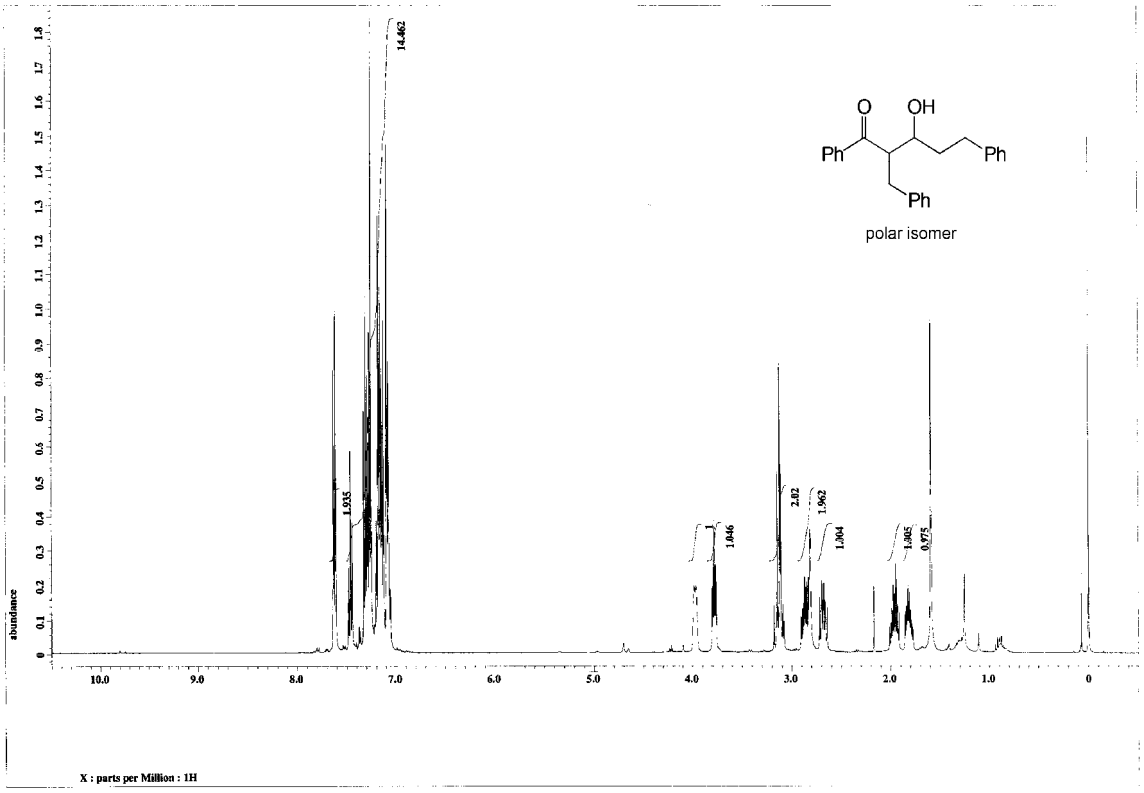

O
OH
Ph
Ph
OMe
abundance
X : parts per Million : 13C


O
OH
Ph
Ph
NO2
5.981
1.427
2.536
2.524
0.511
0.797
0.506
0.357
0.732
0.714
1
0.256
1.725
0.367
abundance
X : parts per Million : 1H


O
OH
Ph
Ph
NO2
abundance
X : parts per Million : 13C


silicone grease
less polar isomer
X : parts per Million : 1H
abundance


less polar isomer
silicone grease
206.3747
133.5782
129.0969
128.6965
128.5153
128.3913
128.3341
128.2293
126.4654
125.7789
77.3146
77.0000
76.6758
71.8894
52.0098
38.2038
36.4589
32.5784
0.9998
X : parts per Million : 13C
abundance


O
OH
Ph
Ph
Ph
polar isomer
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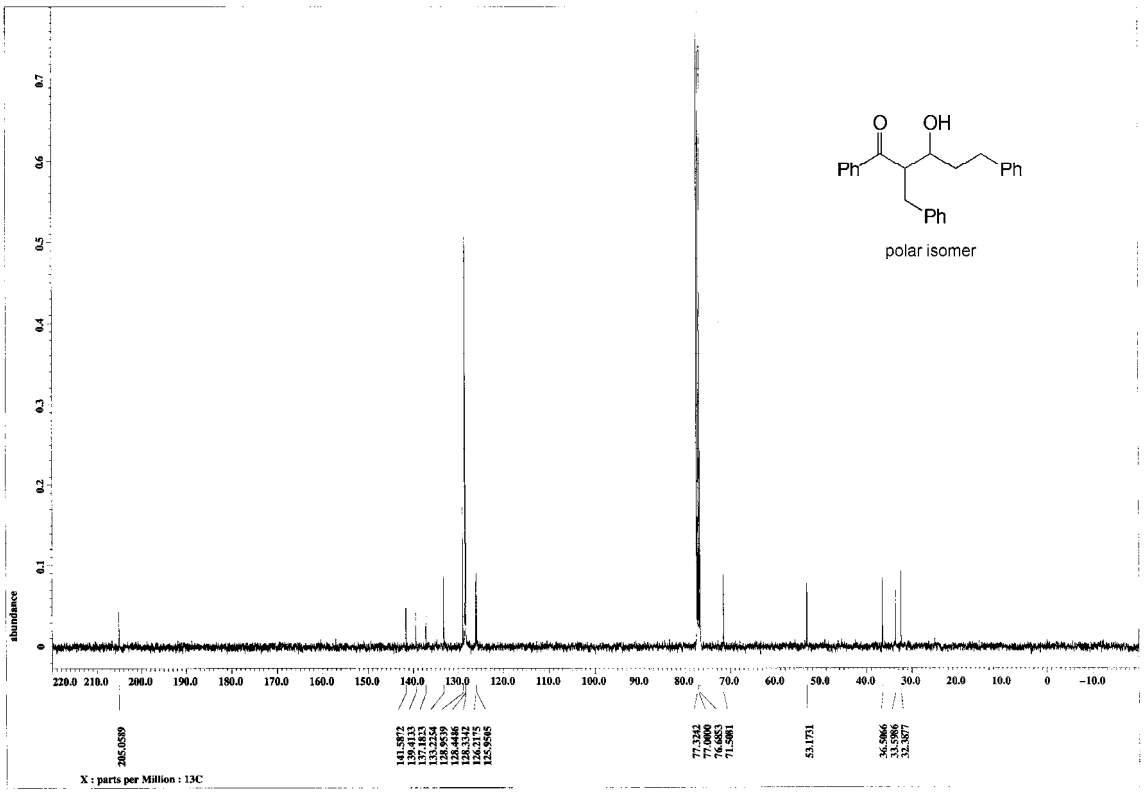
1.046
2.02
1.962
1.004
1.005
0.975
abundance
X : parts per Million : 1H


O
OH
Ph
Ph
Ph
polar isomer
abundance
205.0589
141.5872
139.4133
137.1823
133.2434
128.5853
128.4486
128.3342
126.2175
125.5985
77.3242
77.0000
76.6853
71.5861
53.1731
36.5866
32.3877
X : parts per Million : 13C

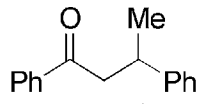
HPLC Traces of the Optically Active Products

D-2500 00/00/00 08:23

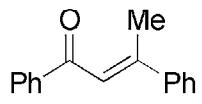
METHOD: TAG: 8 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC
1	7.38	19875	1.084	BB
2	13.94	82447	4.497	BB
3	15.38	81621	4.452	BB
4	18.42	1649472	89.967	BB
TOTAL		1833415	100.000	
PEAK REJ :		0		

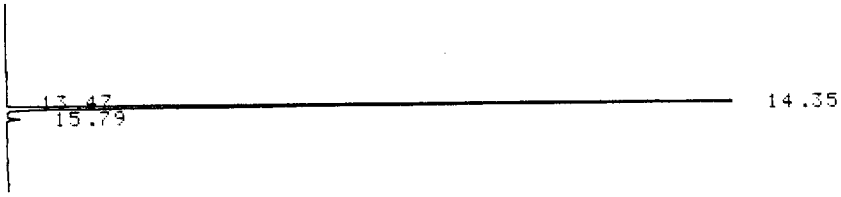


racemate



starting material

CH. 1 C.S 1.25 ATT 8 OFFS 0 00/00/00 01:12



D-2500 00/00/00 01:12

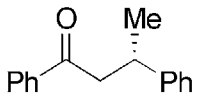
METHOD: TAG: 1 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

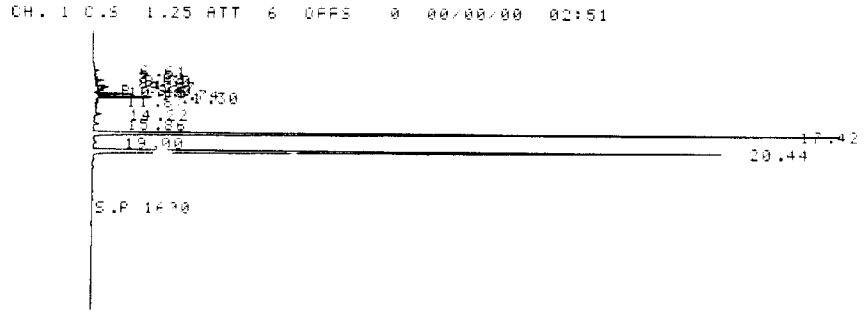
NO.	RT	AREA	CONC	BC
1	13.47	833	0.027	BB
2	14.35	3043437	98.386	BV
3	15.79	49104	1.587	TBB
TOTAL		3093374	100.000	
PEAK REJ :		0		

(*S*)

(*R*)



(*S*)-isomer

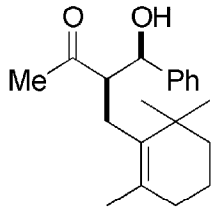


D-2500 00/00/00 02:51

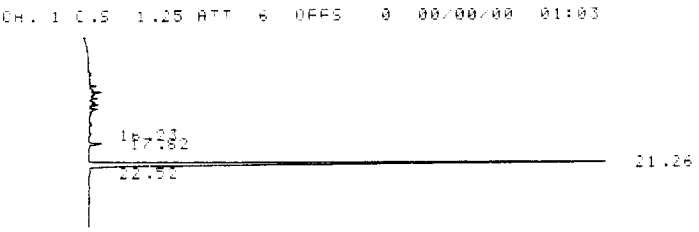
METHOD: TAG: 5 CH: 1

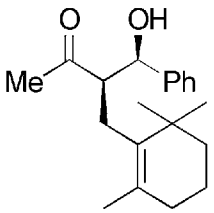
FILE: 1 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC
1	6.61	2922	0.138	BB
2	7.88	1844	0.087	BB
3	8.30	5509	0.261	BV
4	8.56	1327	0.063	VB
5	9.37	26183	1.239	BV
6	10.15	4054	0.192	VB
7	10.74	37688	1.783	BV
8	11.30	49413	2.338	VV
9	11.87	5022	0.238	TBB
10	14.22	5796	0.274	BB
11	15.86	6102	0.289	BB
12	17.42	978900	46.317	BB
13	19.00	3892	0.184	BB
14	20.44	984818	46.597	BB
TOTAL		2113470	100.000	
PEAK REJ :		0		



racemate





(3*R*,4*R*)-isomer

D-2500 00/00/00 01:03

METHOD: TAG: 2 CH: 1

FILE: 1 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC	
2	17.82	19406	2.051	BB	(*3S,4S*)
3	21.26	926804	97.949	BV	(*3R,4R*)
TOTAL		946210	100.000		
PEAK REJ :		3000			

References

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