Supplementary Material (ESI) for Chemical Communications

Supporting Information for:

Highly Enantioselective Aza-ene-type Reaction Catalyzed by Chiral *N*,*N*'-Dioxide-Nickel(II) Complex

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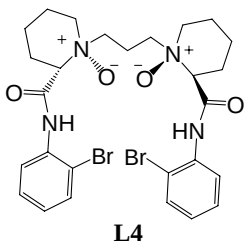
CONTENTS:

(A) General

^{1}H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts are recorded in ppm relative to tetramethylsilane and with the solvent resonance as the internal standard. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, br = broad), coupling constants (Hz), integration. ^{13}C NMR data were collected on commercial instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard. Enantiomer excesses were determined by chiral HPLC analysis on DAICEL CHIRALCEL AD-H/OJ-H/IB/AS-H in comparison with the authentic racemates. Optical rotations were reported as follows: $[\alpha]_D^T$ (c: g/100 mL, in solvent). HRMS was recorded on a commercial apparatus (ESI Source). All the solvents were purified by usual methods before use. Glyoxal derivatives **1a-1r** were synthesized according to the previously reported method.[1] Enamides **2a-2i** and enecarbamates **3a-3d** were prepared in accordance with literature methods.[2]

(B) General procedure for chiral *N,N'*-dioxide preparation

The *N,N'*-dioxide ligands **L1-L5** were synthesized by the same procedure in the literature.[3]



L4

Ligand **L4**: white solid; $[\alpha]^{29}_D$ = - 57.1 (c 0.65 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): 1.41-1.45 (m, 2 H), 1.64-1.70 (m, 2 H), 1.84-1.92 (m, 2 H), 2.00-2.06 (m, 2 H), 2.35-2.56 (m, 4 H), 2.67-2.73 (m, 2 H), 2.87-2.90 (m, 2 H), 3.52-3.60 (m, 8 H), 6.89-6.93 (m, ArH, 2 H), 7.19-7.23 (m, ArH, 2 H), 7.44-7.46 (m, ArH, 2 H),

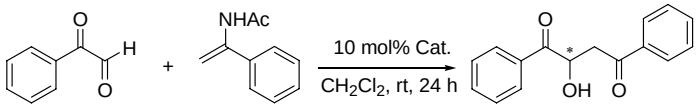
8.12-7.17 (m, ArH, 2 H), 13.40 (s, NH, 2 H), ppm; ^{13}C NMR (100 MHz, $CDCl_3$): 16.3, 20.1, 22.3, 26.4, 64.5, 66.8, 76.1, 114.2, 122.7, 125.2, 127.8, 132.6, 136.5, 167.1 ppm. ESI-HRMS: calcd for $C_{27}H_{34}Br_2N_4O_4$ [M+H$^+$] 637.1020, found 637.1032.

C) General procedure for the enantioselective catalytic aza-ene-type reaction

The mixture of *N*,*N*'-dioxide **L4** (3.2 mg, 0.005 mmol) and $Ni(BF_4)_2{\cdot}6H_2O$ (1.7 mg, 0.005 mmol) in CH_2Cl_2 (1.0 mL) were stirred at 23 ^{o}C for 30 min. Then, phenylglyoxal **1a** (0.10 mmol) and enamide **2a** (0.11 mmoL) were added to the mixture at 23 ^{o}C. After being stirred at 23 ^{o}C in air for 8 h, the reaction mixture was directly purified by column chromatography on silica gel eluted (ether : petroleum ether = 1 : 2) to afford the corresponding product **5a**.

(D) Optimization of conditions

Screening of metals[a]

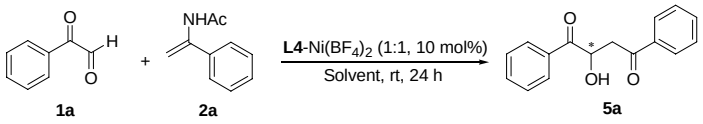


1a **2a** **5a**

Entry	Ligand	Metal	Yield [%][b]	ee [%][c]
1	**L1**	$Cu(AcO)_2$	trace	-
2	**L1**	$Cu(OTf)_2$	47	15
3	**L1**	$Yb(OTf)_2$	58	67
4	**L1**	$Sc(OTf)_3$	63	-7
5	**L1**	$Zn(OTf)_2$	43	4
6	**L1**	$Ni(ClO_4)_2{\cdot}6H_2O$	50	94
7	**L1**	$Ni(BF_4)_2{\cdot}6H_2O$	78	94
8	**L1**	$Ni(acac)_2$	trace	-
9	**L1**	$Ni(AcO)_2$	trace	-
10	**L1**	$NiBr_2$	trace	-
11	-	$Ni(BF_4)_2{\cdot}6H_2O$	56	-

[a] The reaction was carried out with 0.1 mmol phenylglyoxal, 0.11 mmol enamide **2a** in CH_2Cl_2 (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Screening of solvents[a]

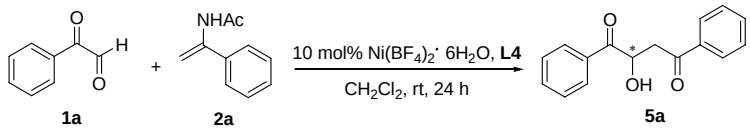


1a **2a** **5a**

Entry	solvent	Yield [%][b]	ee [%][c]
1	THF	trace	-14
2	Et_2O	26	10
3	Toluene	trace	22
4	DMF	trace	-
5	CH_2Cl_2	93	>99
6	CH_2ClCH_2Cl	83	99
7	$CHCl_3$	42	92

[a] The reaction was carried out with 0.1 mmol phenylglyoxal, 0.11 mmol enamide **2a** in solvent (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Optimization of the ratio between ligand and metal[a]

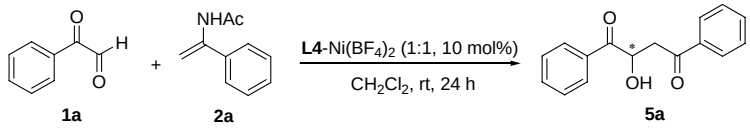


1a **2a** **5a**

Entry	ratio of ligand/metal	Yield [%][b]	ee [%][c]
1	2 : 1	73	75
2	1.5 : 1	82	85
3	1 : 1	93	>99
4	1 : 1.5	93	99
5	1 : 2	92	99
6	1 : 4	87	90

[a] The reaction was carried out with 0.1 mmol phenylglyoxal, 0.11 mmol enamide **2a** in CH_2Cl_2 (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Optimization of the ratio between phenylglyoxal (1a) and enamide (2a)[a]



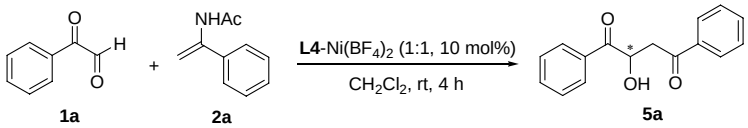
1a **2a** **5a**

Entry	ratio of **1a**/**2a**	Yield [%][b]	ee [%][c]
1	2 : 1	58	98
2	1.1 : 1	63	98
3	1 : 1	90	99
4	1 : 1.1	93	>99

5	1 : 2	92	94

[a] The reaction was carried out in CH_2Cl_2 (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Optimization of additive[a]

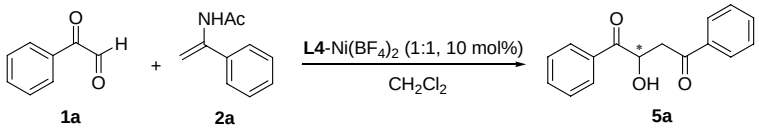


1a **2a** **5a**

Entry	additive	Yield [%][b]	ee [%][c]
1	3Å MS	76	97
2	4Å MS	79	98
3	5Å MS	83	99
4	$^{i}Pr_2NEt$	trace	-
5	phenol	85	96
6	benzoic acid	63	98

[a] The reaction was carried out with 0.1 mmol phenylglyoxal, 0.11 mmol enamide **2a** in CH_2Cl_2 (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Optimization of temperature[a]

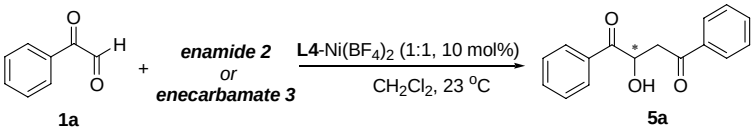


1a **2a** **5a**

Entry	temperature [°C]	time [h]	Yield [%][b]	ee [%][c]
1	-20	64	45	>99
2	0	32	74	>99
3	15	16	92	>99
3	23	12	93	>99
4	30	7	86	97

[a] The reaction was carried out with 0.1 mmol phenylglyoxal, 0.11 mmol enamide **2a** in CH_2Cl_2 (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Optimization of nucleophile[a]



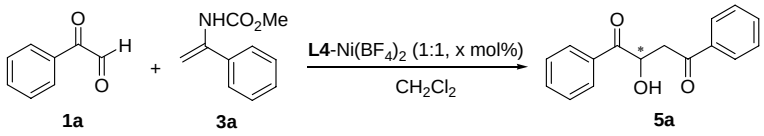
1a **5a**

Entry	Nucleophile	time [h]	Yield [%][b]	ee [%][c]
1	**2a**	12	93	>99
2	**3a**	3	99	>99

3	**3b**	3	92	97
4	**3c**	3	94	98
5	**3d**	3	99	97

[a] The reaction was carried out with 0.1 mmol phenylglyoxal, 0.11 mmol enamide **2** (enecarbamate **3**) in CH_2Cl_2 (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Optimization of catalyst loadin[a]



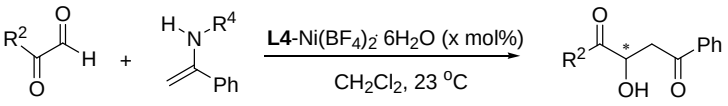
1a **3a** **5a**

Entry	catalyst loading [mol %]	time [h]	Yield [%][b]	ee [%][c]
1	10	3	99	>99
2	5	8	93	99
3	2.5	16	91	99
4	1	32	82	97
5	0.25	48	70	92

[a] The reaction was carried out with 0.1 mmol phenylglyoxal, 0.11 mmol enecarbamate **3a** in CH_2Cl_2 (1.0 mL). [b] Isolated yield. [c] Determined by chiral HPLC.

Full list of substrates

Table 1 Substrate scope for the catalytic enantioselective aza-ene-type reaction with enecarbamate[a]



1 **3** **5**

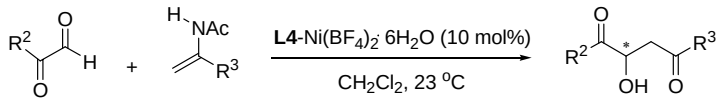
R^2 = Aryl, Alkyl, etc

Entry	R^2	R^4	x [mol %]	Yield [%][b]	ee [%][c]
1	Ph	CO_2Me	5	93	99
2	Ph	CO_2Et	5	92	97
3	Ph	$CO_2{}^{i}Bu$	5	94	98
4	Ph	CO_2Bn	5	99	97
5	2-MeC_6H_4	CO_2Me	5	89	>99
			2.5	83	98
6	3-MeC_6H_4	CO_2Me	5	90	98
			2.5	85	98
7	4-MeC_6H_4	CO_2Me	5	95	99
			2.5	90	99
			1	83	96
8	3-$MeOC_6H_4$	CO_2Me	5	92	99
			2.5	84	98
9	4-$MeOC_6H_4$	CO_2Me	5	99	>99

			2.5	94	99
			1	86	97
10	3,4-$(MeO)_2C_6H_3$	CO_2Me	5	92	>99
11	3-ClC_6H_4	CO_2Me	5	87	99
12	4-ClC_6H_4	CO_2Me	5	92	99
			2.5	85	98
13	3,4-$Cl_2C_6H_3$	CO_2Me	5	85	98
14	4-FC_6H_4	CO_2Me	5	91	99
			2.5	82	98
15	4-BrC_6H_4	CO_2Me	5	82	99
16	3-$NO_2C_6H_4$	CO_2Me	5	80	99
17	4-$NO_2C_6H_4$	CO_2Me	5	72	99
18	2-naphthyl	CO_2Me	5	86	99
19	2-furyl	CO_2Me	5	75	99
20	2-thienyl	CO_2Me	5	86	98
21	*c*-hexyl	CO_2Me	5	70	99
22	OEt	CO_2Me	5	85	98 (*S*)[d]

[a] Unless otherwise noted, the reaction was carried out with 1-5 mol% **L4**-$Ni(BF_4)_2{\cdot}6H_2O$, 0.1 mmol of glyoxal derivative (glyoxylate), and 1.1 equiv of enecarbamate **3** in CH_2Cl_2 (1.0 mL) at 23 °C for 3-18 h. [b] Isolated yield. [c] Determined by chiral HPLC analysis. [d] The absolute configuration of (*S*)-**5s** was determined by comparison with literature data.[2a]

Table 2 Substrate scope for the catalytic enantioselective aza-ene-type reaction with enamide[a]

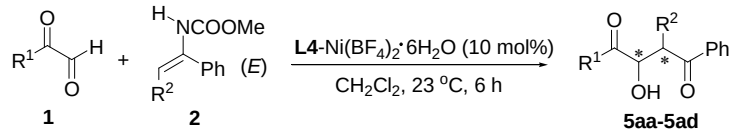


1 **2** **5**

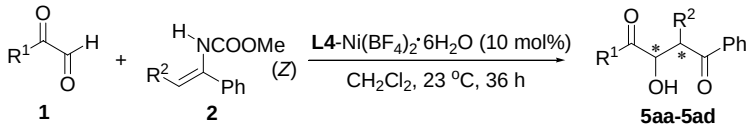
R^2 = Aryl, Alkyl, etc

Entry	R^2	R^3	Product	Yield [%][b]	ee [%][c]
1	Ph	Ph	**5a**	93	>99
2	Ph	2-MeC_6H_4	**5t**	92	>99
3	Ph	3-MeC_6H_4	**5u**	92	>99
4	Ph	4-MeC_6H_4	**5v**	94	>99
5	Ph	4-$MeOC_6H_4$	**5w**	95	99
6	Ph	4-ClC_6H_4	**5x**	87	99
7	Ph	4-FC_6H_4	**5y**	87	99
8	Ph	2-naphthyl	**5z**	91	>99
9	Ph	*c*-hexyl	**-**	N.R.	-
10	Ph	*i*-Pr	**-**	N.R.	-
11	Ph	COOMe	**-**	N.R.	-

[a] Unless otherwise noted, the reaction was carried out with 10 mol% **L4**-$Ni(BF_4)_2{\cdot}6H_2O$, 0.1 mmol of glyoxal derivative, and 1.1 equiv of enamide **2** in CH_2Cl_2 (1.0 mL) at 23 °C for 6-16 h. [b] Isolated yield. [c] Determined by chiral HPLC.



R^1 = Ph, R^2 = Me, **5aa,** 88%, anti/syn = 99:1, 98% ee (*anti*)
R^1 = Ph, R^2 = Et, **5ab,** 83%, anti/syn = 99:1, 99% ee (*anti*)
R^1 = OEt, R^2 = Me, **5ac,** 85%, anti/syn = 99:1, >99% ee (*anti*)
R^1 = OEt, R^2 = Et, **5ad,** 82%, anti/syn = 99:1, 99% ee (*anti*)



R^1 = Ph, R^2 = Me, **5aa,** 70%, anti/syn = 5:95, 19% ee (*syn*)
R^1 = Ph, R^2 = Et, **5ab,** 63%, anti/syn = 5:95, 16% ee (*syn*)
R^1 = OEt, R^2 = Me, **5ac,** 73%, anti/syn = 50:50, 55% ee (*syn*), -73% ee (*anti*)
R^1 = OEt, R^2 = Et, **5ad,** 71%, anti/syn = 60:40, 52% ee (*syn*), -74% ee (*anti*)

Scheme 1 Substrate scope for the catalytic enantioselective aza-ene-type reaction with α-substituted enecarbamates

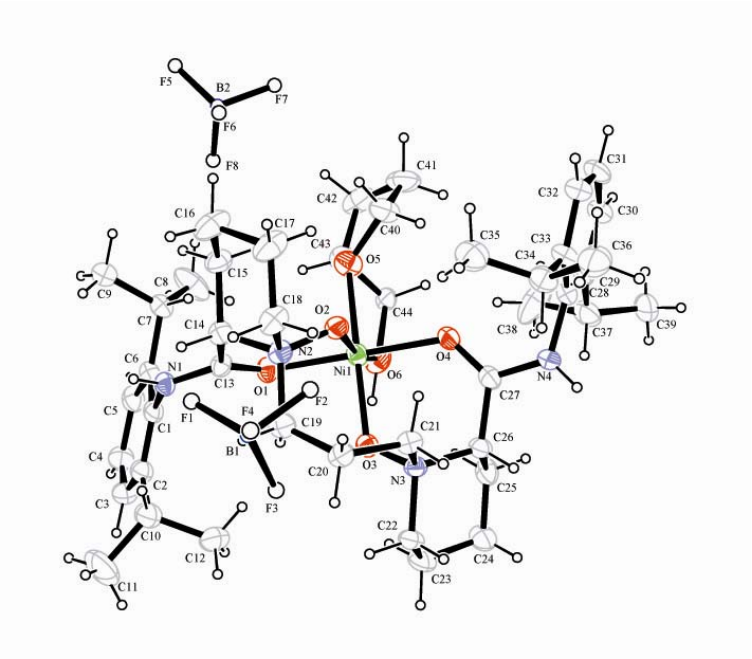


Fig. 1 X-ray crystallographic structure of the **L3**-Ni(II) ·TFM (TFM = Tetrahydro-2-furanmethanol) complex**.**

Single crystals of $C_{44}H_{69}B_2F_8N_4NiO_6$ **L3**-Ni(II) complex were crystallized from mixed solvents of THF and petroleum ether. The thermal ellipsoids' level is 20 % for the above crystal structure. We conjectured that the exogenous ligand (TFM) was come from the unpurified solvent THF.

Crystal data. $C_{44}H_{69}B_2F_8N_4NiO_6$, M = 982.36, orthorhombic, a = 12.784(3), b = 13.268(3), c = 34.336(7) Å, U = 5824(2) Å^3, T = 113 K, space group $P2_12_12_1$ (no. 19), Z = 4, 37884 reflections measured, 11378 unique (R_{int} = 0.0526) which used in all calculations. The final $wR(F_2)$ was 0.2144 (all data). The structure of compound **L3**-Ni(II) has been refined carefully by PLATON/SQUEEZE software. (ref.: A.L. Spek, *Acta Cryst. D,* 2009, **65**, 148-155.）

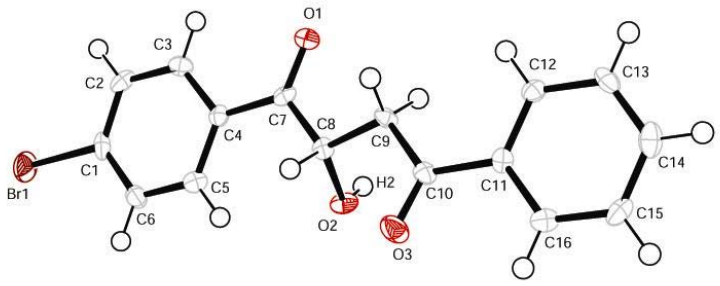


Fig. 2 X-ray crystallographic structure of the product **5l.**

Single crystals of $C_{16}H_{13}BrO_3$ **5l** were crystallized from mixed solvents of THF and petroleum ether. The absolute configuration of C8 is *S*. The thermal ellipsoids' level is 30 % for the above crystal structure.

Crystal data. $C_{16}H_{13}BrO_3$, M = 330.00, orthorhombic, a = 5.5934(11), b = 7.4974(15), c = 31.910(6) Å, U = 1338.2(5) Å^3, T = 113 K, space group $P2_12_12_1$ (no. 19), Z = 4, 10559 reflections measured, 3171 unique (R_{int} = 0.0691) which used in all calculations. The final $wR(F_2)$ was 0.0666 (all data).

Reference(Program)：

(1) Data reduction：

CrystalClear，

Rigaku/MSC(2005), CrystalClear and CrystalStructure, Rigaku/MSC, The Woodlands , Texas, USA

(2) Structure solution：

(a) absorption correction

ABSCOR/multi-scan

Higashi,T. (1995) ABSCOR, Rigaku Corperation, Tokyo, Japan

(b) Structure Solution and refinement

SHELXS-97 and SHELXL-97

Sheldrick,G.M. (1997), SHELXS97 and SHELXL97，University of Göttingen, Germany

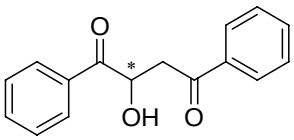
(3) Graphics：

Ortep-3 for Windows 1.076

Farrugia,L.J. (1997) *J. Appl. Cryst.* 30,565

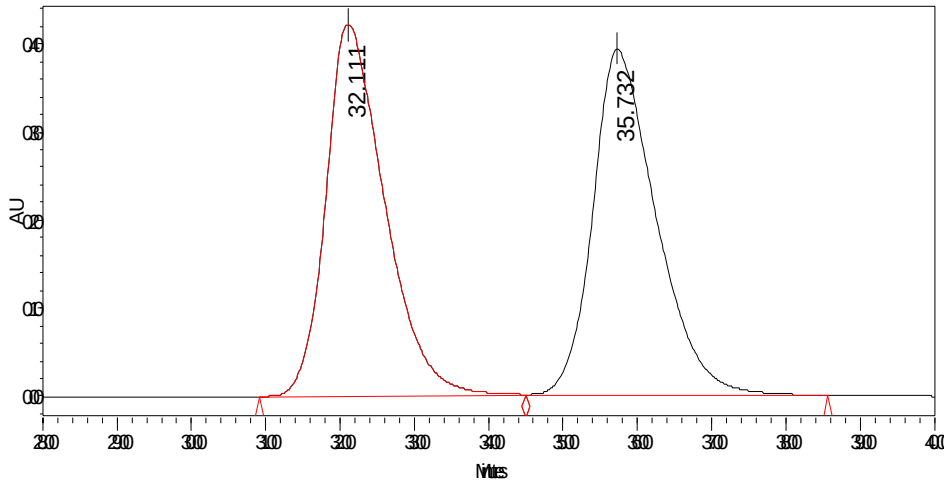
(E) The analytical and spectral characterization data of aza-ene-type reaction products

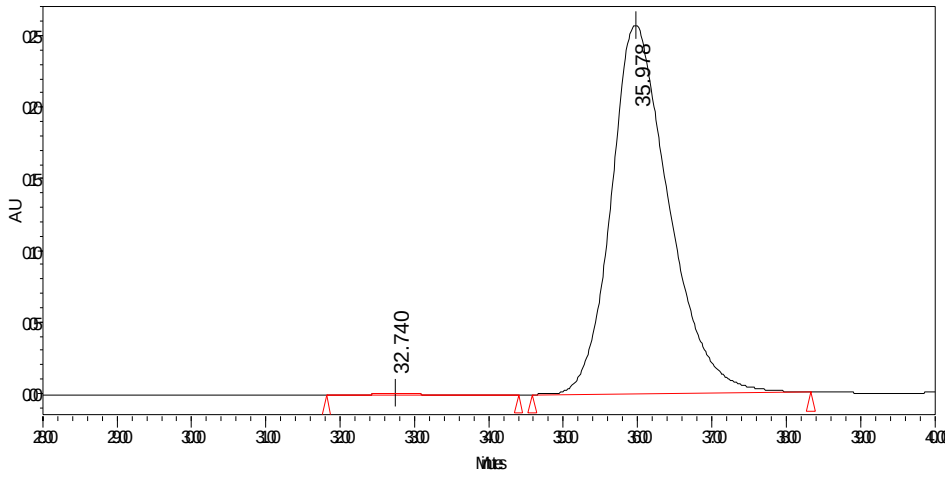
2-hydroxy-1,4-diphenylbutane-1,4-dione (5a)



5a

($C_{16}H_{14}O_3$) a white solid; 93% yield, >99% ee. $[\alpha]_D^{25}$ = +12.5 (*c* 0.440 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 32.7 min (minor) and 36.0 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.37-3.49 (m, 2H), 4.04 (d, *J* = 6.0 Hz, 1H), 5.68-5.73 (m, 1H), 7.46-7.55 (m, 4H), 7.58-7.66 (m, 2H), 7.95-8.01 (m, 4H) ppm.

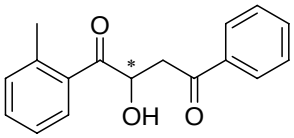




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	32.740	59289	0.42	1452	0.56	31.233	33.750

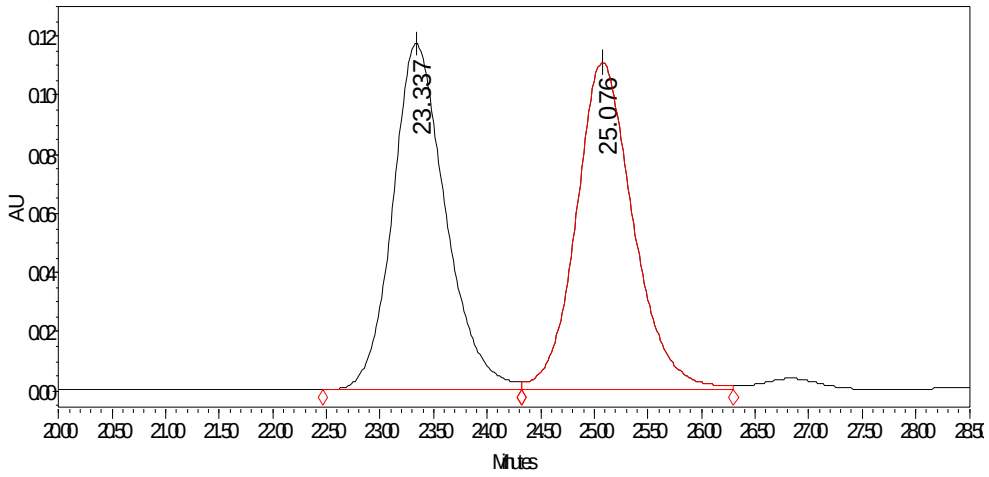
2	35.978	13896192	99.58	257736	99.44	34.583	38.333

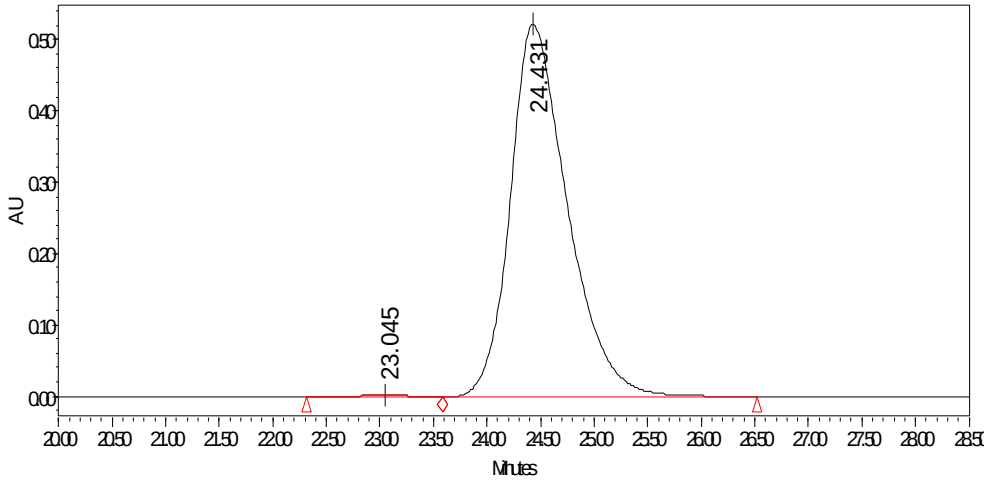
2-hydroxy-4-phenyl-1-o-tolylbutane-1,4-dione (5b)

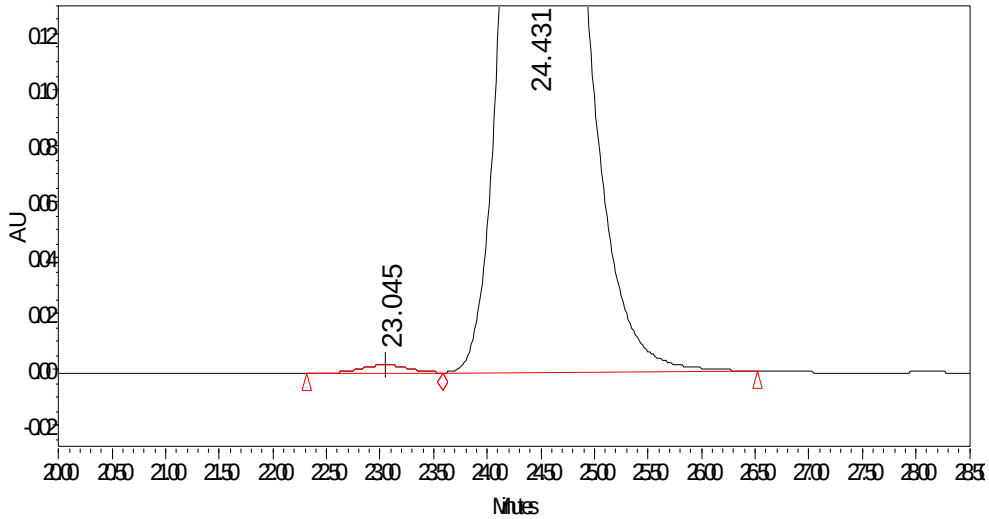


5b

($C_{17}H_{16}O_3$) a colourless viscous liquid; 89% yield, >99% ee. $[\alpha]_D^{25}$ = +9.1 (*c* 0.460 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 23.1min (minor) and 24.4 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 2.42 (s, 3H), 3.37-3.40 (m, 2H), 4.02 (d, *J* = 6.0 Hz, 1H), 5.65-5.69 (m, 1H), 7.36-7.47 (m, 4H), 7.55-7.59 (m, 1H), 7.50-7.80 (m, 2H), 7.93-7.95 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 21.4, 43.7, 70.0, 125.9, 128.3, 128.7, 128.8, 129.2, 133.6, 134.8, 136.7, 138.9, 197.1, 201.0 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_3$ ([M-H^+]) = 267.1021, Found 267.1030.



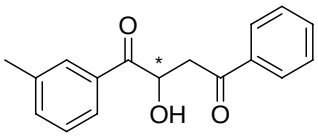




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	23.045	93728	0.48	2974	0.57	22.317	23.583

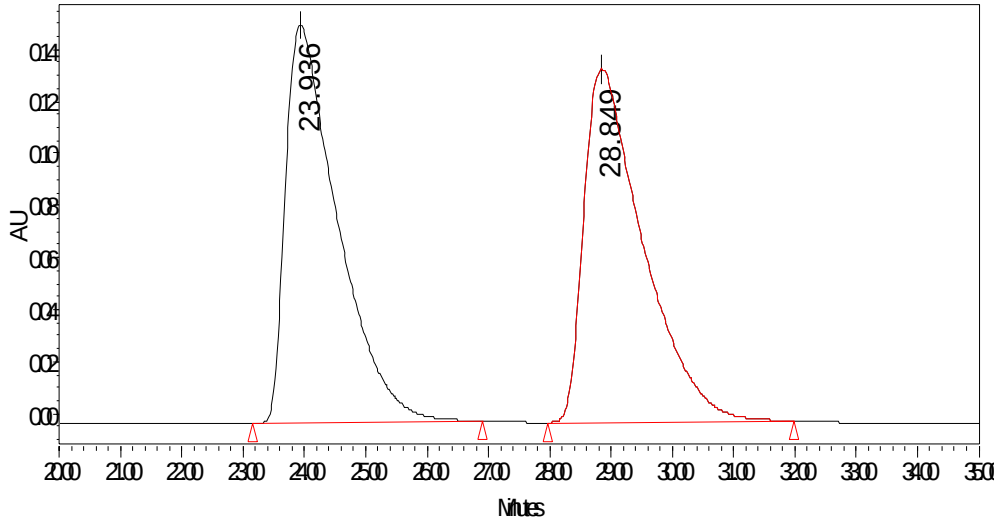
2	24.431	19422004	99.52	523142	99.43	23.583	26.517

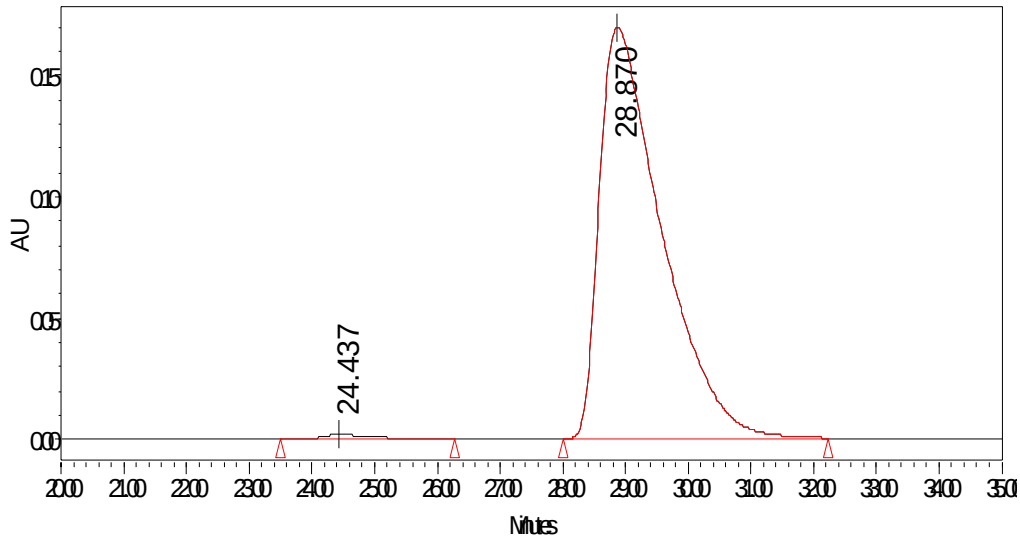
2-hydroxy-4-phenyl-1-m-tolylbutane-1,4-dione (5c)



5c

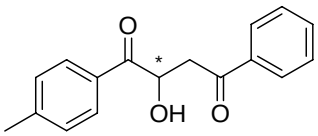
($C_{17}H_{16}O_3$) a colourless viscous liquid; 90% yield, 98% ee. $[\alpha]_D^{25}$ = +13.0 (*c* 0.432 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 24.4 min (minor) and 28.9 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 2.57 (s, 3H), 3.30-3.50 (m, 2H), 4.06 (d, *J* = 8.0 Hz, 1H), 5.45-5.49 (m, 1H), 7.25-7.31 (m, 2H), 7.39-7.45 (m, 3H), 7.54-7.61 (m, 2H), 7.91-7.95 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 20.9, 42.8, 71.2, 125.8, 128.3, 128.4, 128.7, 132.1, 132.3, 133.5, 134.1, 136.6, 139.5, 197.0, 203.9 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_3$ ([M-H$^+$]) = 267.1021, Found 267.1028.





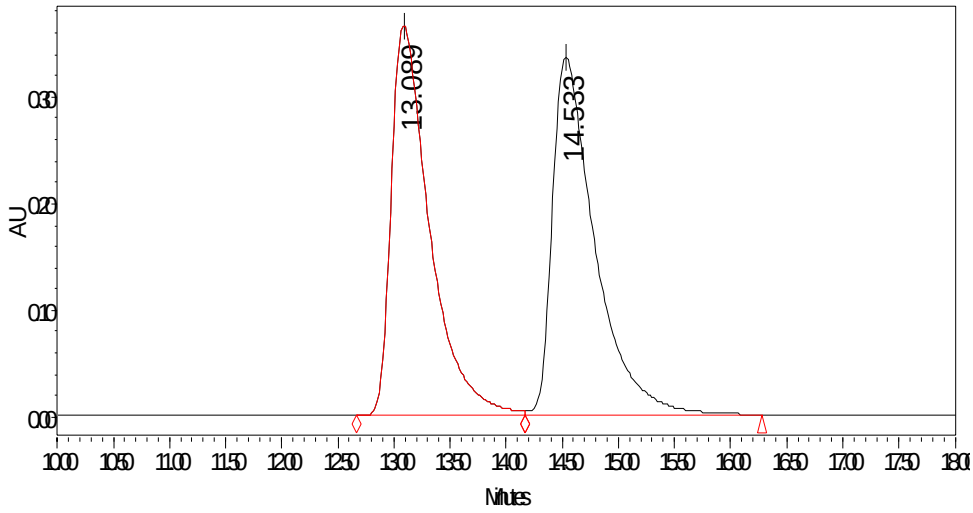
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	24.437	113763	0.96	1942	1.13	23.500	26.283
2	28.870	11688080	99.04	169635	98.87	28.000	32.233

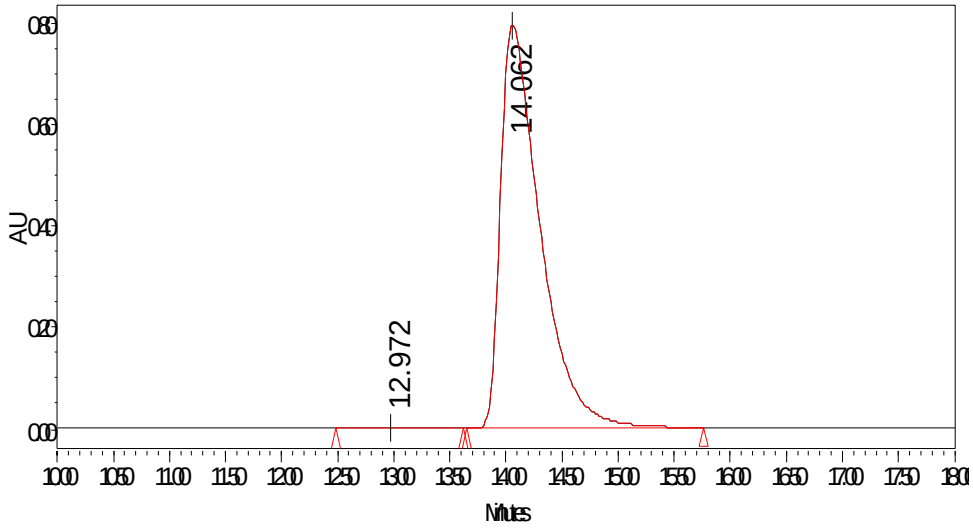
2-hydroxy-4-phenyl-1-p-tolylbutane-1,4-dione (5d)

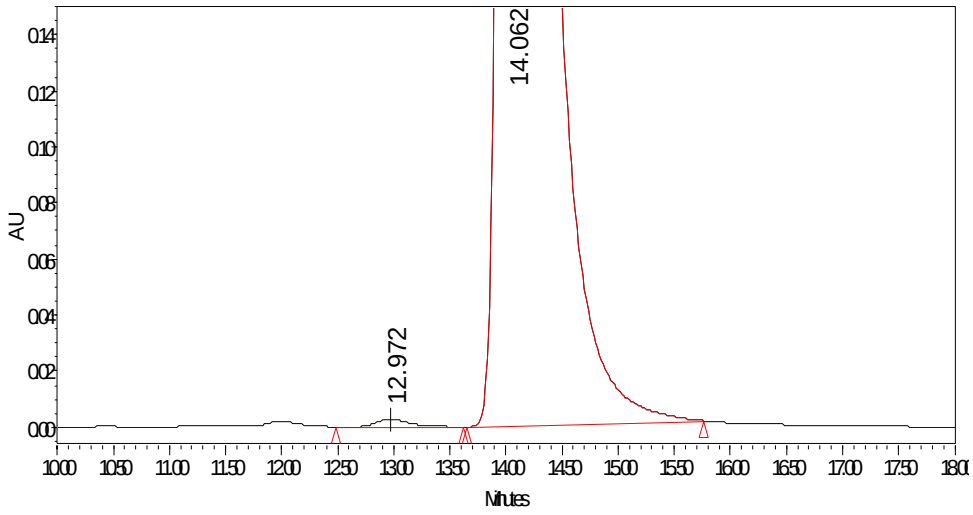


5d

($C_{17}H_{16}O_3$) a white solid; 95% yield, >99% ee. $[\alpha]_D^{25}$ = +5.0 (*c* 0.450 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 13.0 min (minor) and 14.1 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 2.42 (s, 3H), 3.31-3.44 (m, 2H), 4.03 (d, *J* = 6.0 Hz, 1H), 5.65-5.69 (m, 1H), 7.26-7.30 (m, 2H), 7.43-7.47 (m, 2H), 7.55-7.59 (m, 1H), 7.88-7.95 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 21.8, 43.9, 69.9, 128.3, 128.7, 128.9, 129.7, 130.9, 133.6, 136.7, 145.1, 197.1, 200.4 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_3$ ([M-H$^+$]) = 267.1021, Found 267.1026.

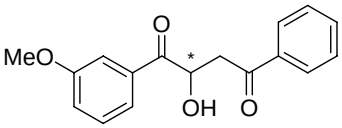

13.089
14.533
AU
Minutes


14.062
12.972
AU
Minutes


14.062
12.972
AU
Minutes

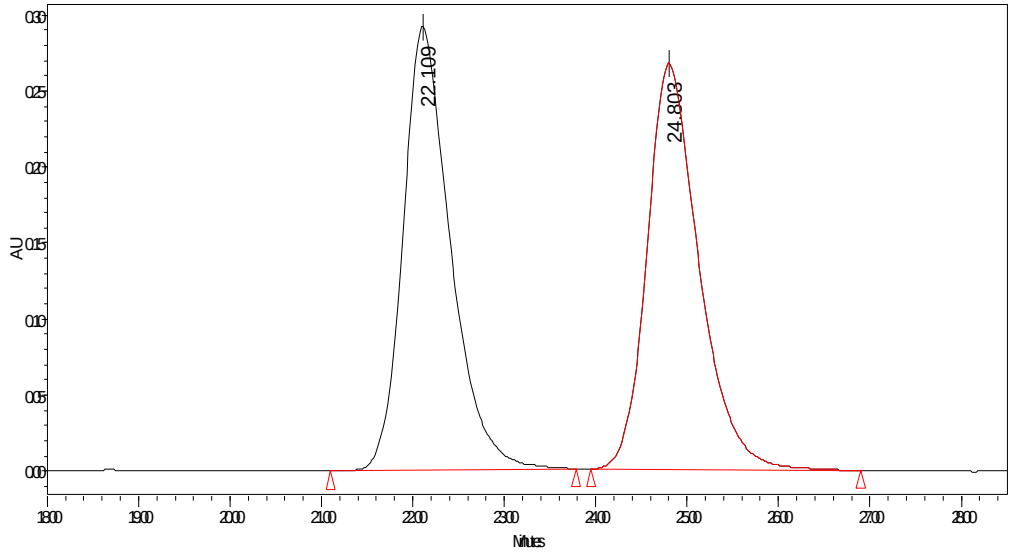
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	12.972	54950	0.28	2726	0.34	12.483	13.617
2	14.062	19629386	99.72	797618	99.66	13.650	15.767

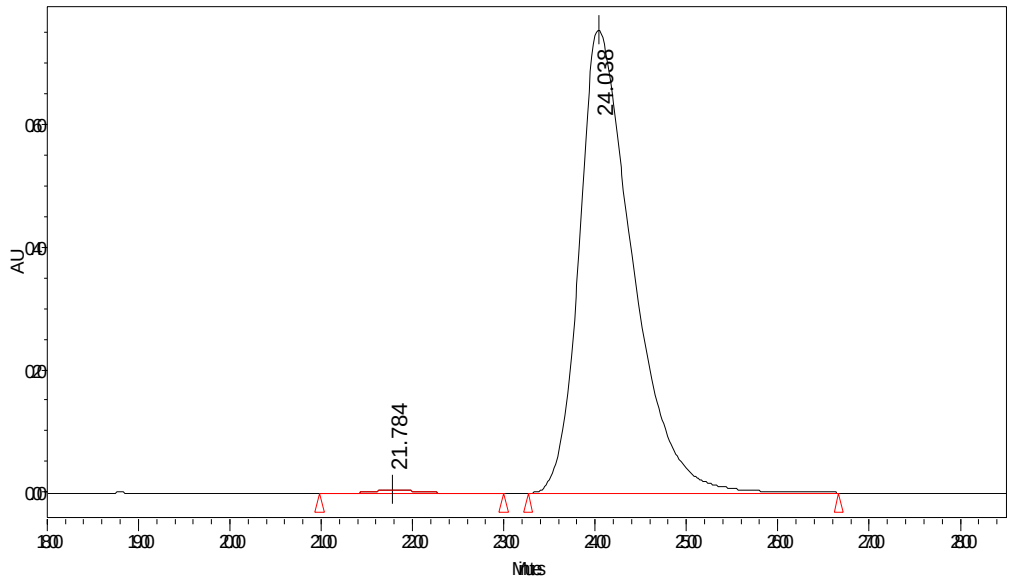
2-hydroxy-1-(3-methoxyphenyl)-4-phenylbutane-1,4-dione (5e)



5e

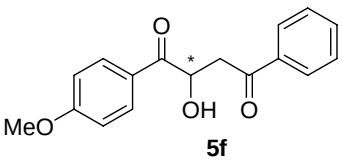
($C_{17}H_{16}O_4$) a colourless viscous liquid; 92% yield, 99% ee. $[\alpha]_D^{25}$ = +26.1 (*c* 0.430 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 21.8 min (minor) and 24.0 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.40-3.44 (m, 2H), 3.86 (s, 3H), 4.01 (d, *J* = 6.0 Hz, 1H), 5.63-5.68 (m, 1H), 7.14-7.17 (m, 1H), 7.38-7.47 (m, 3H), 7.52-7.59 (m, 3H), 7.91-7.94 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.7, 55.5, 70.2, 113.0, 120.5, 121.1, 128.3, 128.7, 130.0, 133.6, 134.9, 136.6, 160.0, 197.1, 200.6 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_4$ ([M+H$^+$]) = 285.1121, Found 285.1128.





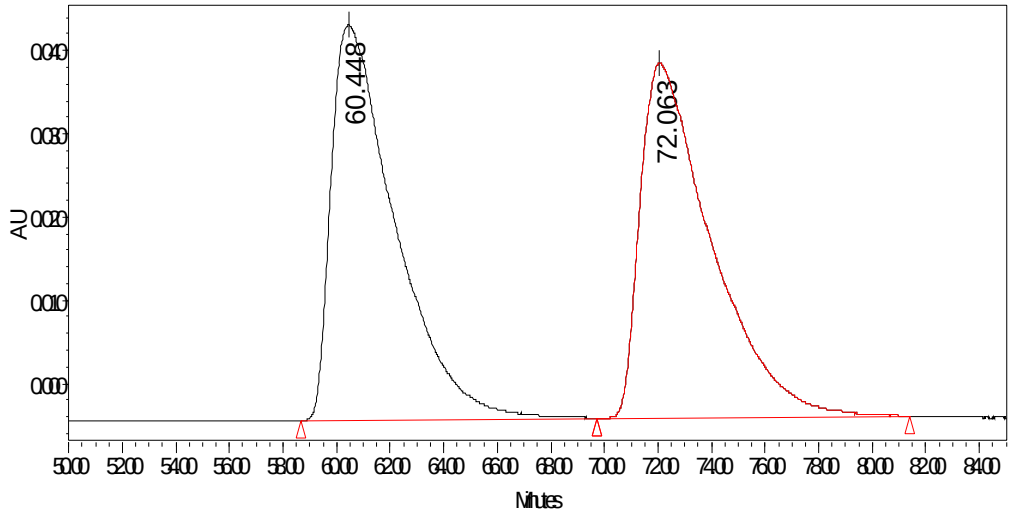
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	21.784	207584	0.67	5783	0.76	20.983	23.067
2	24.038	30720259	99.33	756626	99.24	23.083	26.817

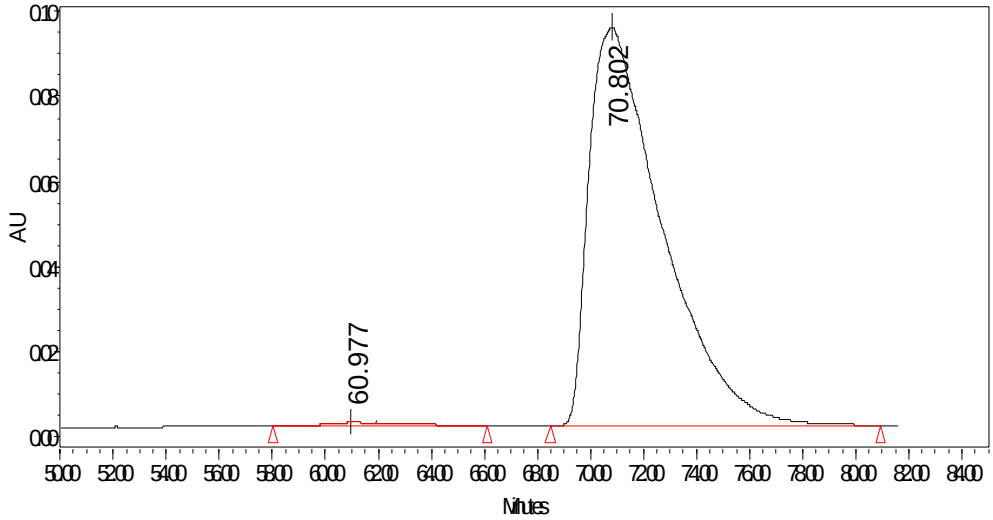
2-hydroxy-1-(4-methoxyphenyl)-4-phenylbutane-1,4-dione (5f)



5f

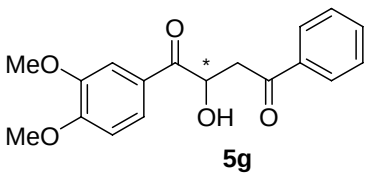
($C_{17}H_{16}O_4$) a white solid; 99% yield, 99% ee. $[\alpha]_D^{25}$ = +4.2 (*c* 0.480 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 61.0 min (minor) and 70.8 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.27-3.44 (m, 2H), 3.88 (s, 3H), 4.05 (d, *J* = 6.4 Hz, 1H), 5.64-5.68 (m, 1H), 6.95-6.99 (m, 2H), 7.44-7.47 (m, 2H), 7.55-7.59 (m, 1H), 7.94-8.01 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 44.1, 55.6, 69.7, 114.2, 126.2, 128.4, 128.7, 131.2, 133.5, 136.7, 164.2, 197.3, 199.0 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_4$ ([M+H$^+$]) = 285.1121, Found 285.1125.





	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	60.977	130851	0.72	742	0.78	58.033	66.117
2	70.802	18063773	99.28	94039	99.22	68.483	80.933

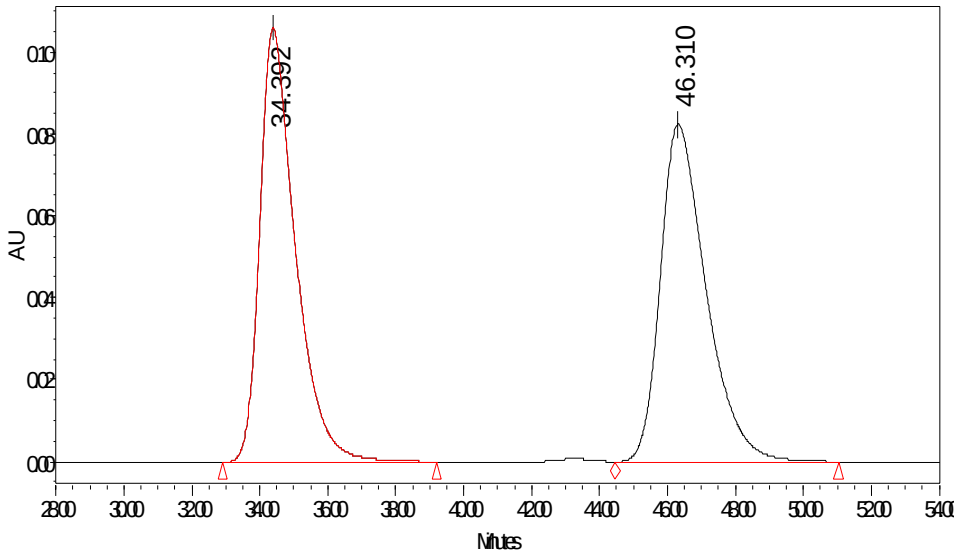
2-hydroxy-1-(3,4-dimethoxyphenyl)-4-phenylbutane-1,4-dione (5g)

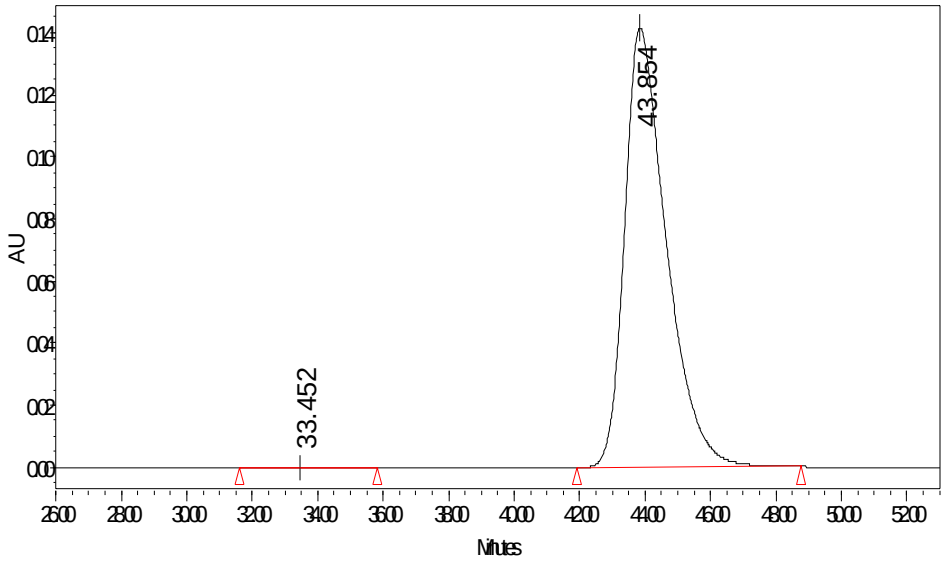


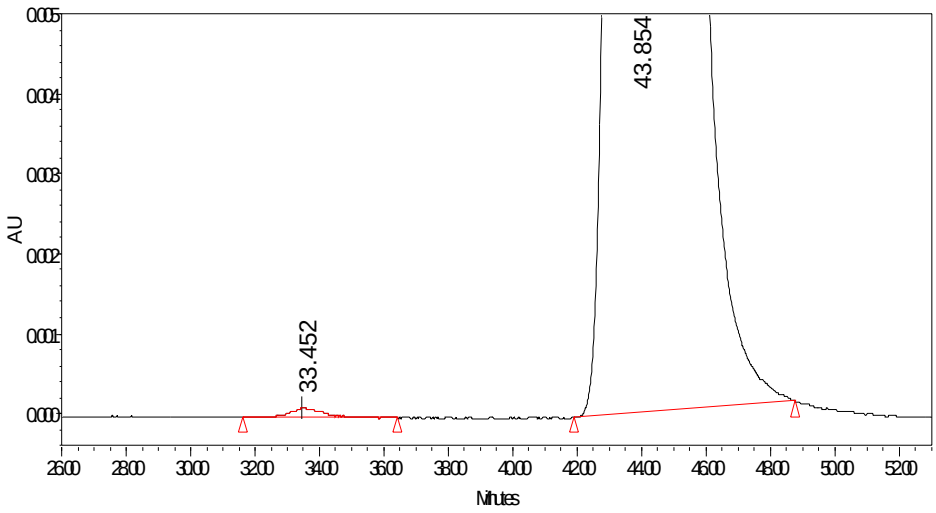
5g

($C_{18}H_{18}O_5$) a white solid; 92% yield, >99% ee. $[\alpha]_D^{25}$ = +29.1 (*c* 0.460 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 25/85, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 33.5 min

(minor) and 43.9 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.30-3.46 (m, 2H), 3.94(s, 6H), 4.03 (d, *J* = 5.2 Hz, 1H), 5.69 (s, 1H), 6.89-6.92 (m, 1H), 7.44-7.48 (m, 2H), 7.55-7.64 (m, 3H), 7.93-7.96 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 44.3, 56.1, 56.2, 69.6, 110.3, 110.8 123.5, 126.3, 128.3, 128.7, 133.6, 136.7, 149.4, 154.1, 197.4, 199.1 ppm; HRMS (ESI-TOF) calcd for $C_{18}H_{18}O_5$ ([M+H$^+$]) = 315.1227, Found 315.1237.

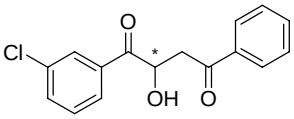






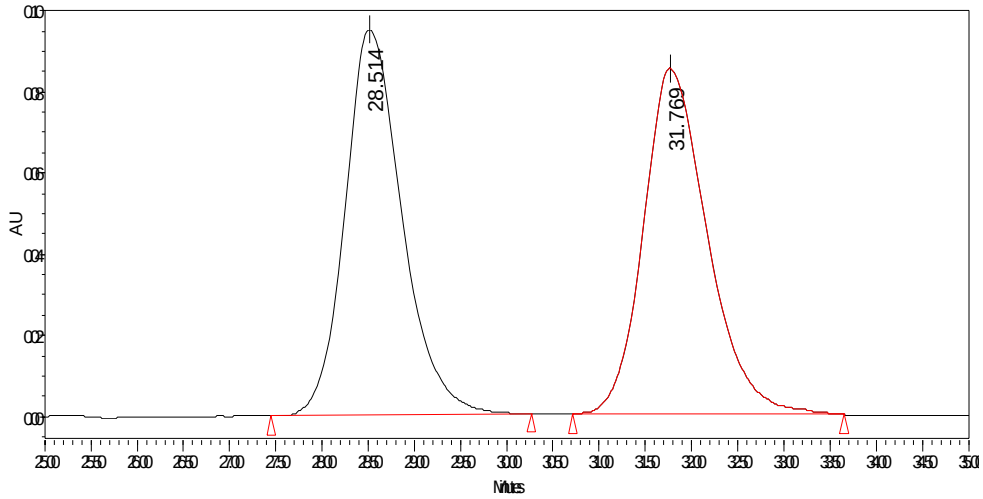
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	33.452	7651	0.06	110	0.08	31.617	36.100
2	43.854	12544045	99.94	141544	99.92	41.917	48.767

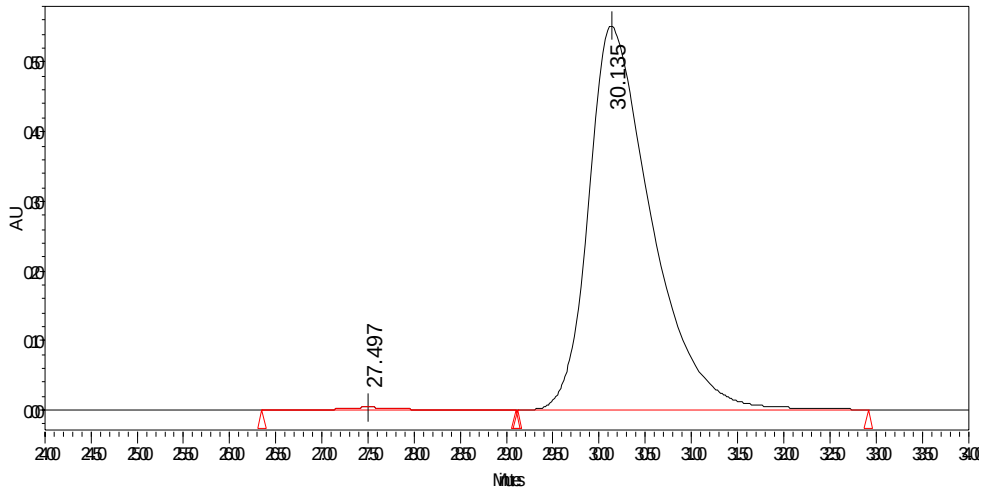
1-(3-chlorophenyl)-2-hydroxy-4-phenylbutane-1,4-dione (5h)



5h

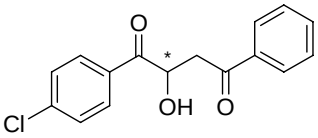
($C_{16}H_{13}ClO_3$) a white solid; 87% yield, 99% ee. $[\alpha]_D^{25}$ = +28.0 (*c* 0.210 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 27.5 min (minor) and 30.1 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.39-3.50 (m, 2H), 4.01 (d, *J* = 5.2 Hz, 1H), 5.54-5.57 (m, 1H), 7.41-7.51 (m, 3H), 7.56-7.61 (m, 2H), 7.86-7.89 (m, 1H), 7.93-8.00 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 42.9, 70.5, 126.9, 128.3, 128.7, 128.9, 130.2, 133.8, 135.3, 135.5, 136.5, 197.5, 199.5 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{13}ClO_3$ ([M+Na^+]) = 311.0445, Found 311.0452.





	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	27.497	148782	0.57	3636	0.65	26.350	29.100
2	30.135	25984210	99.43	552308	99.35	29.117	32.917

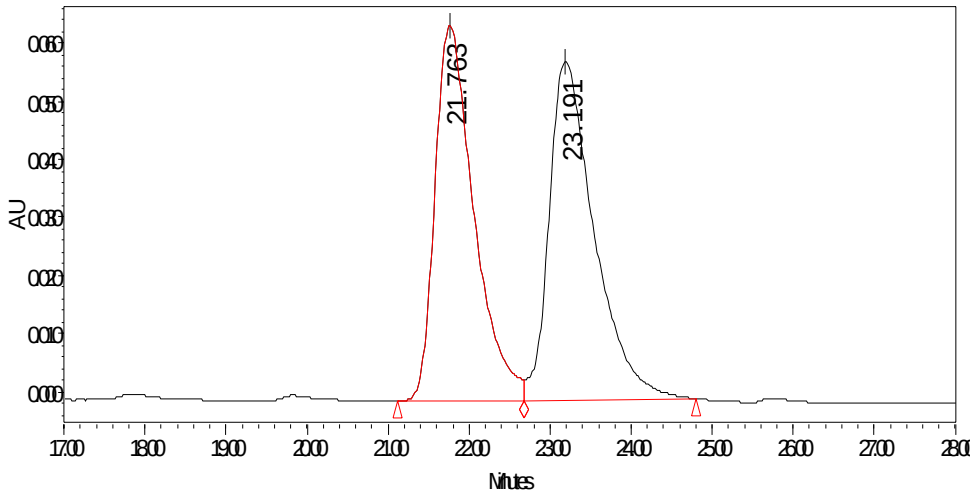
1-(4-chlorophenyl)-2-hydroxy-4-phenylbutane-1,4-dione (5i)

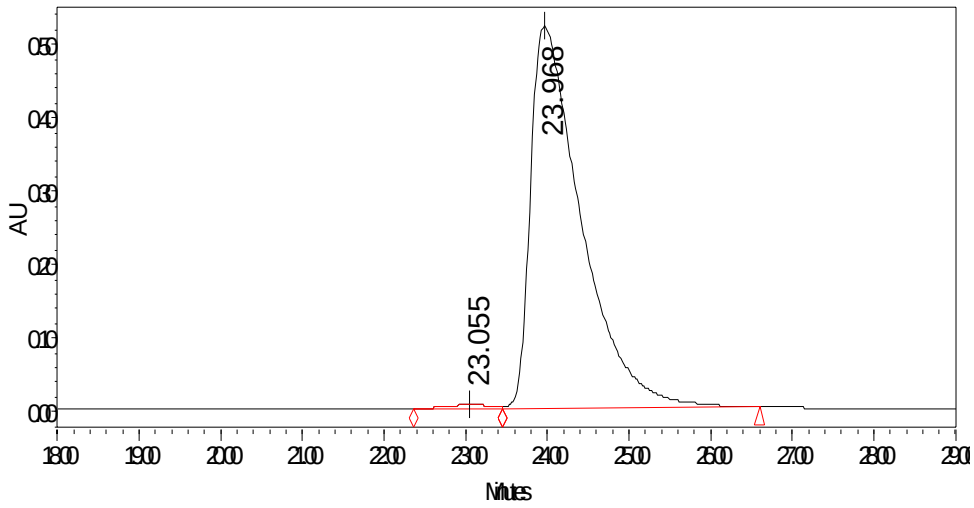


5i

($C_{16}H_{13}ClO_3$) a white solid; 92% yield, 99% ee. $[\alpha]_D^{25}$ = +4.5 (*c* 0.210 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 5/95, flow

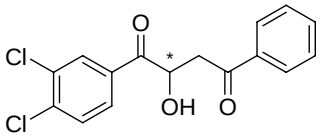
rate = 1.0 mL/min, λ = 254 nm, retention time: 23.1 min (minor) and 24.0 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.35-3.49 (m, 2H), 4.02 (d, *J* = 6.0 Hz, 1H), 5.57-5.60 (m, 1H), 7.42-7.53 (m, 4H), 7.56-7.61 (m, 1H), 7.93-8.03 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.1, 70.3, 128.3, 128.7, 129.4, 130.2, 132.2, 133.7, 136.5, 140.4, 197.5, 199.5 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{13}ClO_3$ ([M+Na^+]) = 311.0445, Found 311.0446.





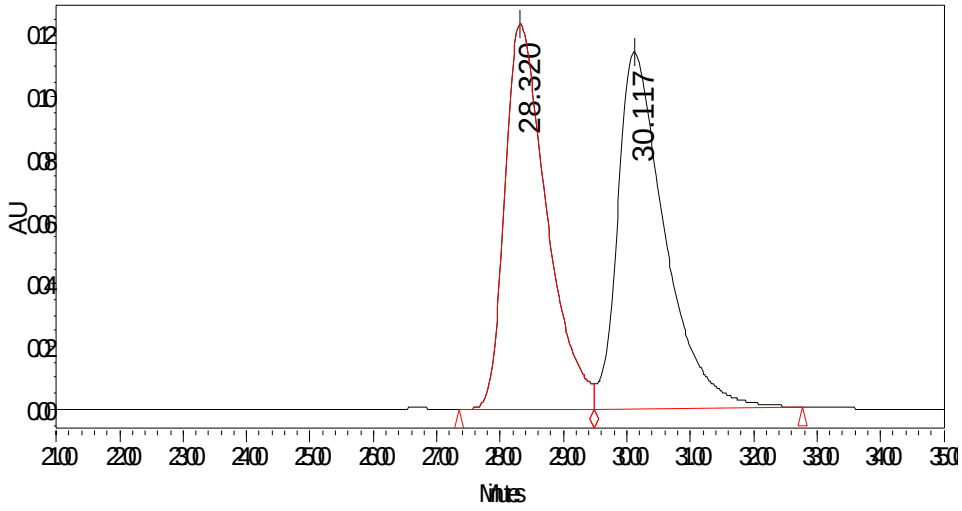
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	23.055	151042	0.67	4042	0.77	22.367	23.450
2	23.968	22470433	99.33	519280	99.23	23.450	26.600

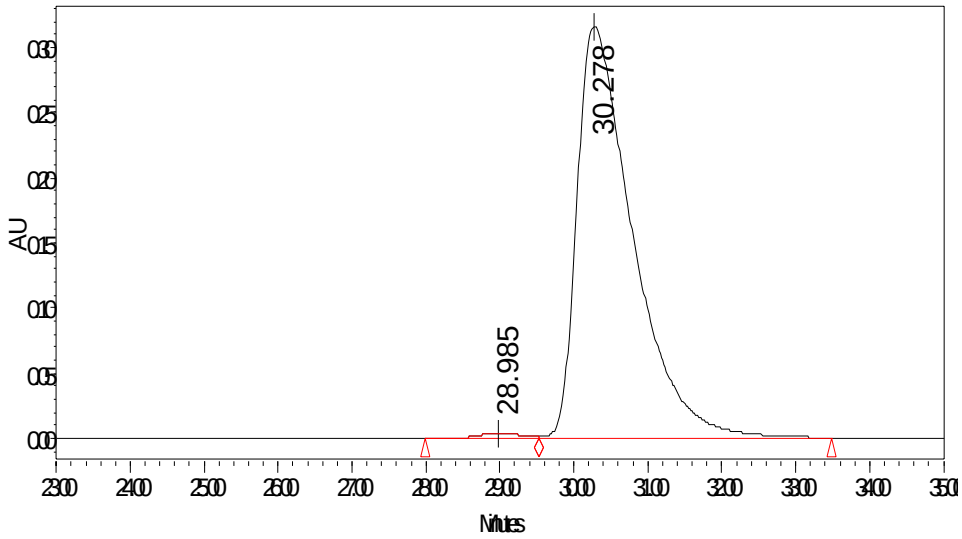
1-(3,4-dichlorophenyl)-2-hydroxy-4-phenylbutane-1,4-dione (5j)



5j

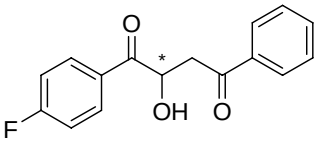
($C_{16}H_{12}Cl_2O_3$) a white solid; 85% yield, 98% ee. $[\alpha]_D^{25}$ = +14.2 (*c* 0.272 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 5/95, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 29.0 min (minor) and 30.3 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.42-3.55 (m, 2H), 4.01 (d, *J* = 3.6 Hz, 1H), 5.48 (s, 1H), 7.46-7.48 (m, 2H), 7.56-7.62 (m, 2H), 7.84-7.86 (m, 1H), 7.94-7.97 (m, 2H), 8.11-8.13 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 42.6, 70.7, 127.9, 128.3, 128.8, 130.8, 130.9, 133.7, 133.9, 136.3, 197.8, 198.5 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{12}Cl_2O_3$ ([M+Na^+]) = 345.0056, Found 345.0057.





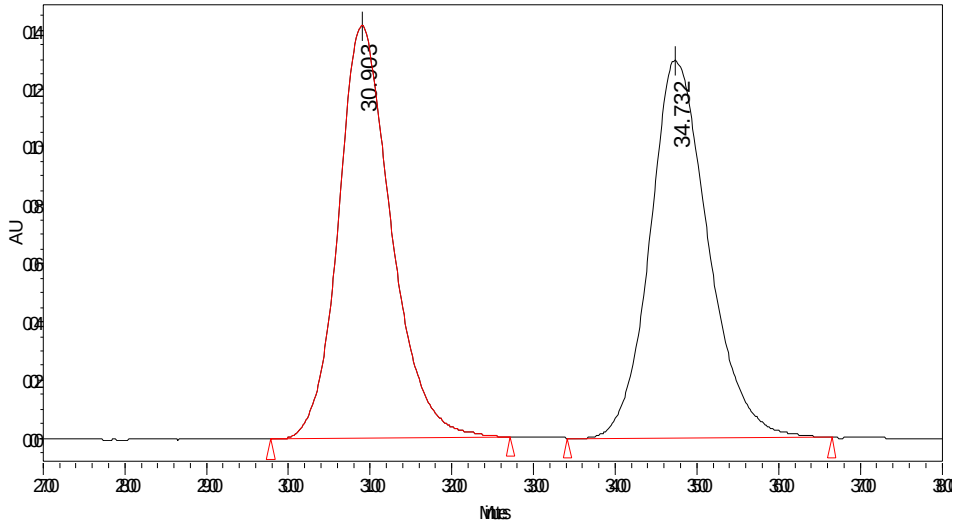
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	28.985	176555	1.05	4238	1.32	27.983	29.533
2	30.278	16589919	98.95	316211	98.68	29.533	33.483

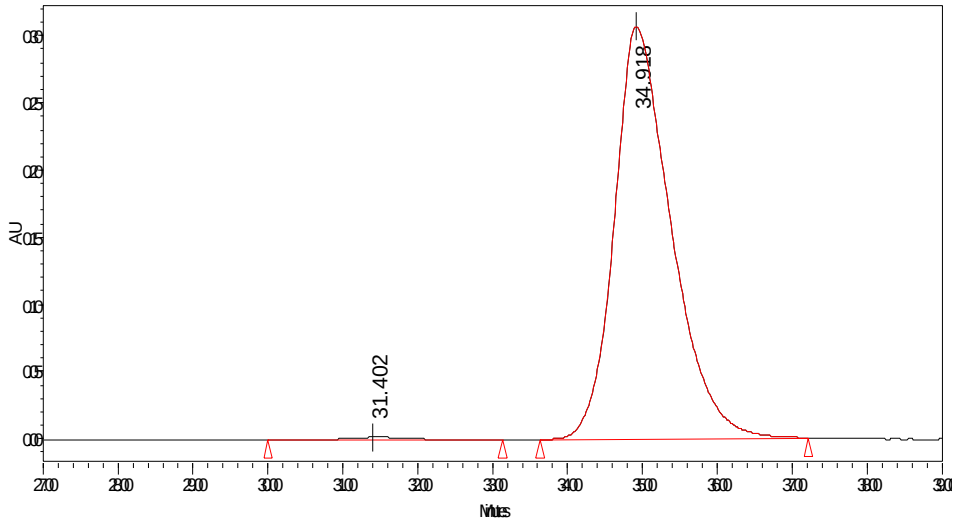
1-(4-fluorophenyl)-2-hydroxy-4-phenylbutane-1,4-dione (5k)



5k

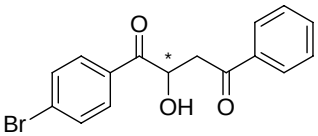
($C_{16}H_{13}FO_3$) a white solid; 91% yield, 99% ee. $[\alpha]_D^{25}$ = +30.1 (*c* 0.212 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 31.4 min (minor) and 34.9 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.34-3.49 (m, 2H), 4.02 (d, *J* = 6.0 Hz, 1H), 5.60-5.63 (m, 1H), 7.15-7.26 (m, 2H), 7.45-7.49 (m, 2H), 7.53-7.59 (m, 1H), 7.93-7.96 (m, 2H), 8.04-8.08 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 42.2, 69.2, 115.1(d, J_{CF} = 22 Hz), 127.3, 127.7, 129.1, 130.5 (d, J_{CF} = 10 Hz), 132.7, 135.5, 165.3 (d, J_{CF} = 255 Hz), 196.5, 198.0 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{13}FO_3$ ([M+Na^+]) = 295.0741, Found 295.0738.





	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	31.402	85073	0.52	1531	0.50	30.000	33.133
2	34.918	16184107	99.48	307230	99.50	33.633	37.217

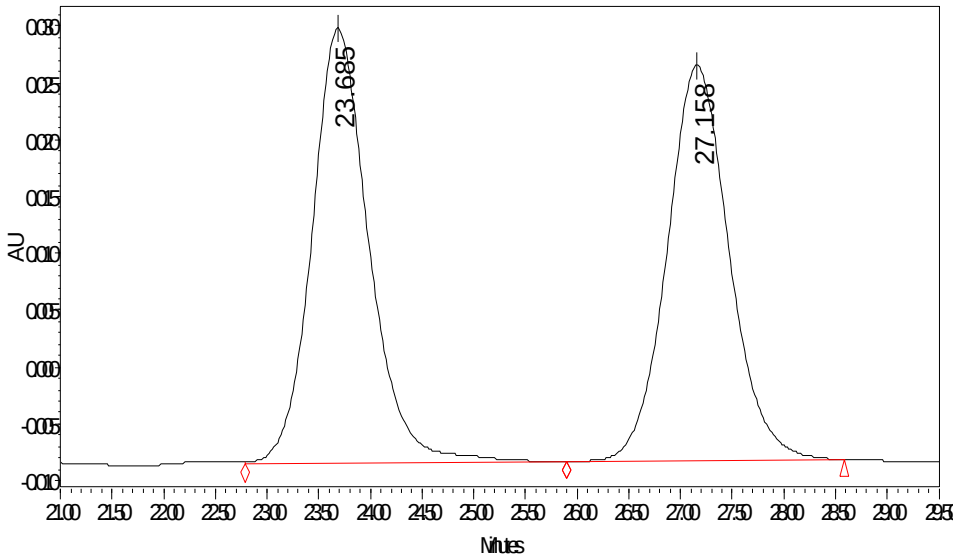
1-(4-bromophenyl)-2-hydroxy-4-phenylbutane-1,4-dione (5l)

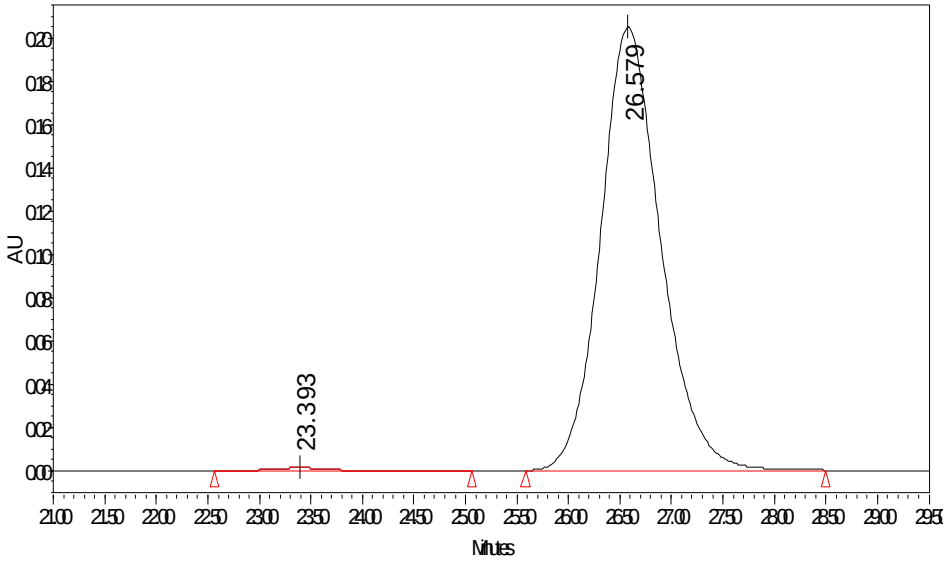


5l

($C_{16}H_{13}BrO_3$) a white solid; 82% yield, 99% ee. $[\alpha]_D^{25}$ = -5.0 (*c* 0.400 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 15/85, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 23.4 min (minor) and

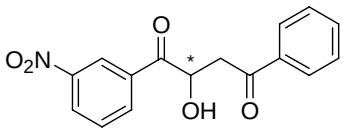
26.5 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.35-3.50 (m, 2H), 4.01 (d, J = 6.0 Hz, 1H), 5.54-5.59 (m, 1H), 7.44-7.48 (m, 2H), 7.56-7.58 (m, 1H), 7.59-7.61 (m, 2H), 7.86-7.89 (m, 2H), 7.90-7.95 (m, 2H) ppm.





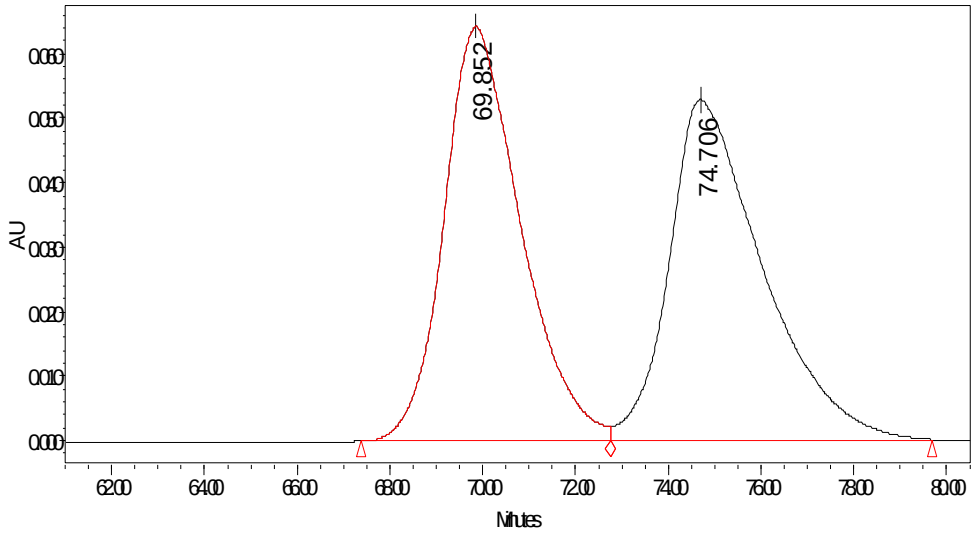
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	23.393	50205	0.60	1347	0.65	22.567	25.067
2	26.579	8321552	99.40	204945	99.35	25.583	28.500

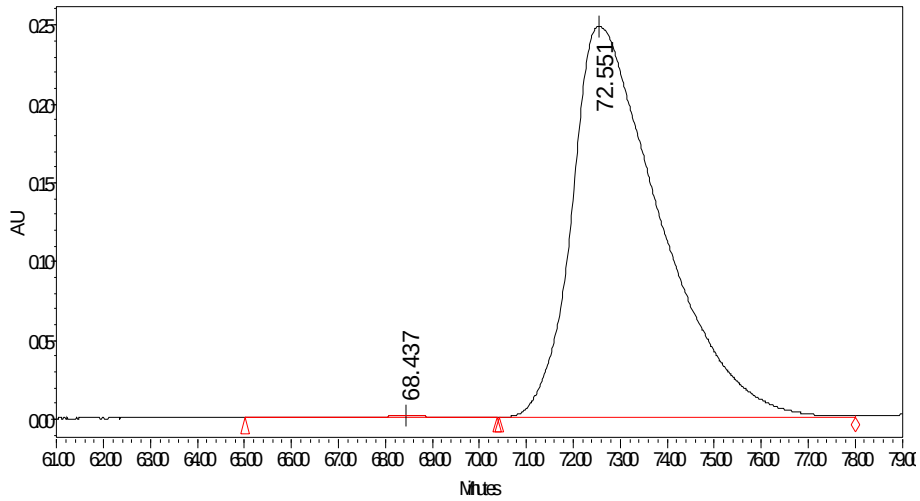
2-hydroxy-1-(3-nitrophenyl)-4-phenylbutane-1,4-dione (5m)



5m

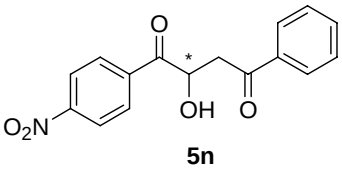
($C_{16}H_{13}NO_5$) a colourless viscous liquid; 80% yield, >99% ee. $[\alpha]_D^{25}$ = +17.6 (*c* 0.340 in CH_2Cl_2) HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 15/85, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 68.4min (minor) and 72.6 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.56-3.58 (m, 2H), 4.11 (d, *J* = 6.8 Hz, 1H), 5.46-5.53 (m, 1H), 7.46-7.50 (m, 2H), 7.60-7.69 (m, 1H), 7.71-7.73 (m, 1H), 7.95-7.96 (m, 2H), 8.36-8.39 (m, 1H), 8.42-8.45 (m, 1H), 8.89 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 42.1, 71.3, 123.9, 127.7, 128.3, 128.8, 130.0, 134.0, 134.6, 135.9, 136.3, 148.5, 198.3, 198.5 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{13}NO_5$ ([M+Na^+]) = 322.0686, Found 322.0695.





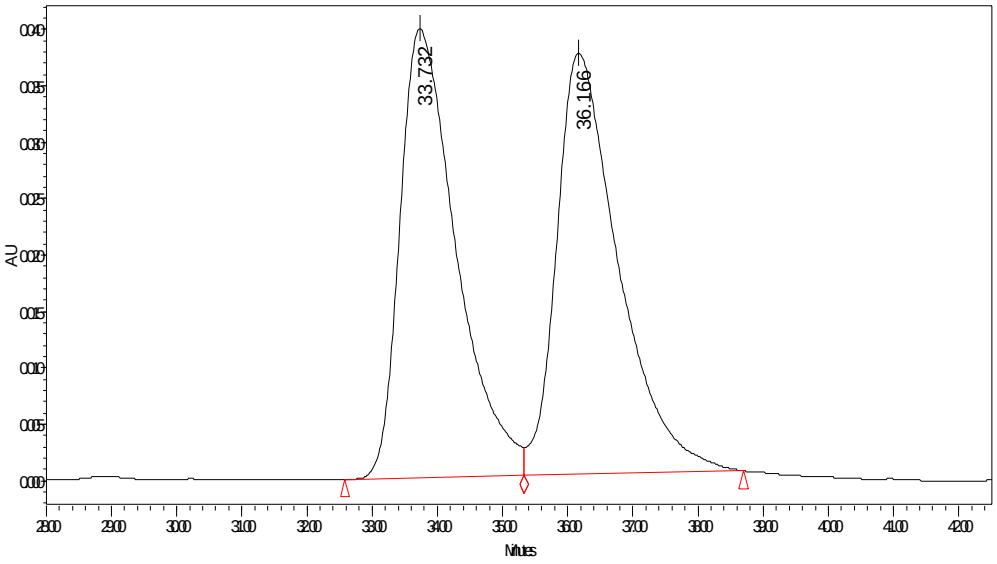
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	68.437	87204	0.27	905	0.36	65.017	70.383
2	72.551	32587128	99.73	248212	99.64	70.433	78.000

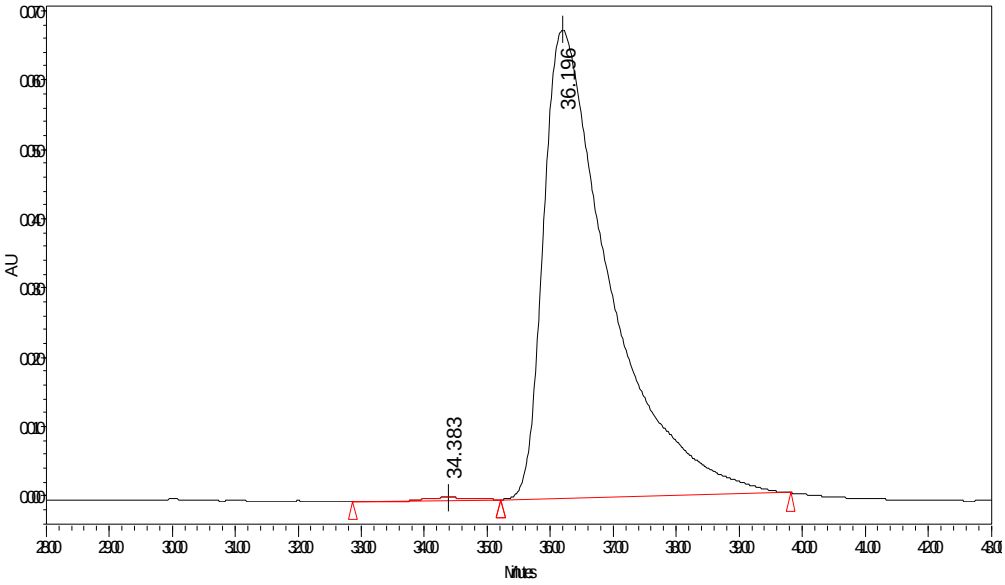
2-hydroxy-1-(4-nitrophenyl)-4-phenylbutane-1,4-dione (5n)



5n

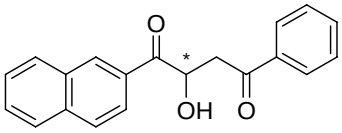
($C_{16}H_{13}NO_5$) a white solid; 72% yield, 99% ee. $[\alpha]_D^{25}$ = +5.8 (*c* 0.160 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 34.4 min (minor) and 36.2 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.53-3.55 (m, 2H), 4.03 (d, *J* = 6.8 Hz, 1H), 5.47-5.52 (m, 1H), 7.46-7.51 (m, 2H), 7.59-7.63 (m, 1H), 7.94-7.96 (m, 2H), 8.18-8.20 (m, 2H), 8.32-8.35 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 42.2, 71.3, 123.9, 128.3, 128.8, 130.0, 134.0, 136.2, 139.2, 150.5, 198.1, 199.4 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{13}NO_5$ ([M+Na^+]) = 322.0686, Found 322.0692.





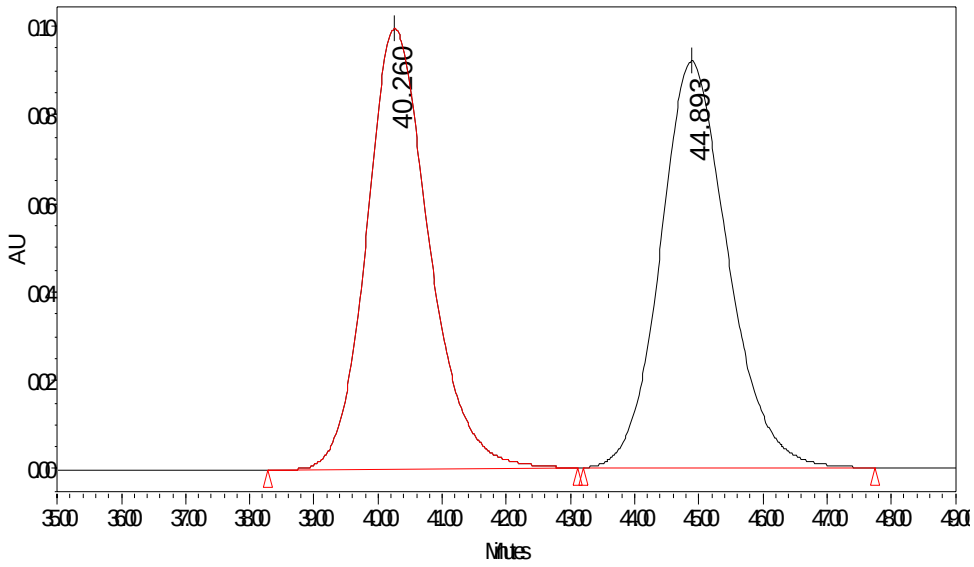
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	34.383	20635	0.41	393	0.58	32.867	35.217
2	36.196	4957682	99.59	67551	99.42	35.217	39.817

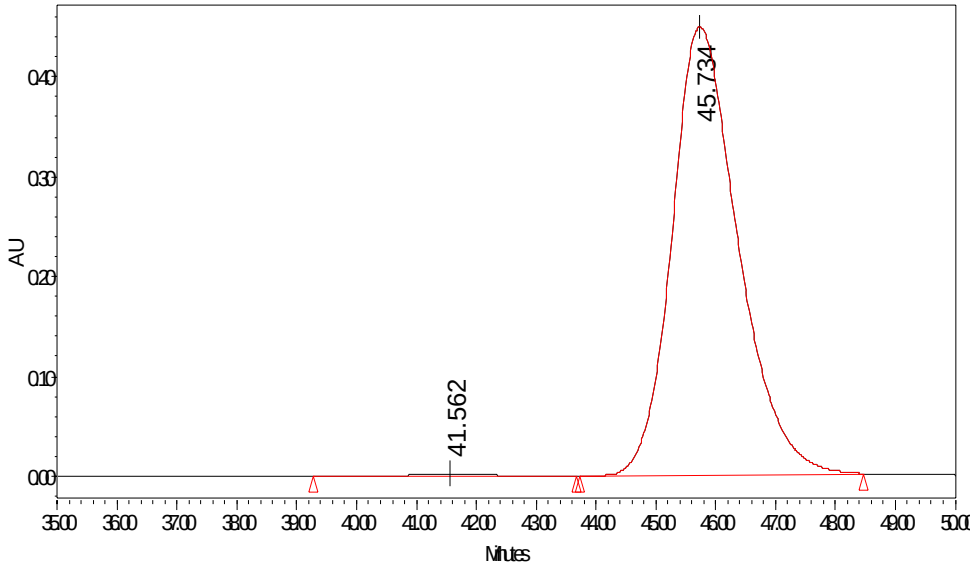
2-hydroxy-1-(naphthalen-2-yl)-4-phenylbutane-1,4-dione (5o)

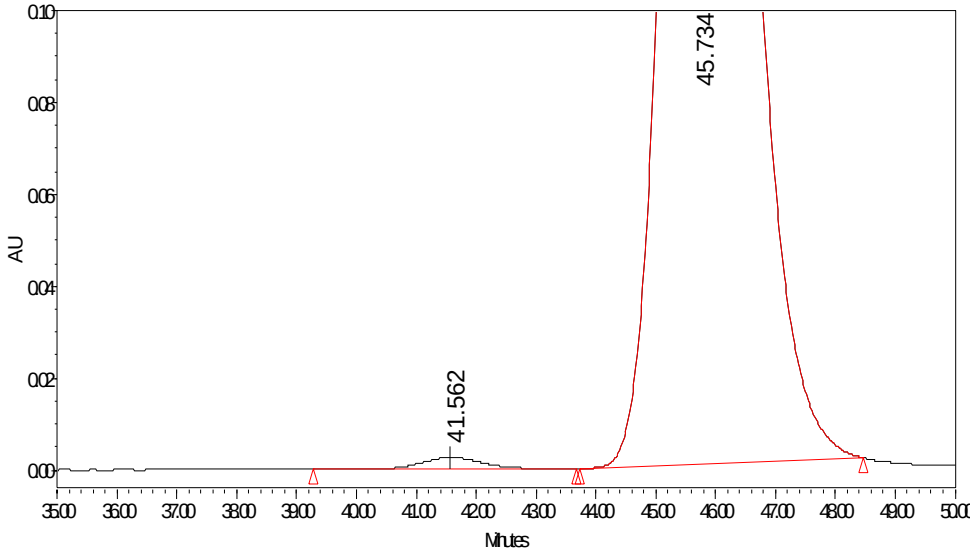


5o

($C_{20}H_{16}O_3$) a white solid; 86% yield, 99% ee. $[\alpha]_D^{25}$ = -36.9 (*c* 0.260 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 41.6 min (minor) and 45.7 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.43-3.51 (m, 2H), 4.10 (d, *J* = 4.0 Hz, 1H), 5.83-5.85 (m, 1H), 7.44-7.46 (m, 2H), 7.55-7.64 (m, 3H), 7.90-8.06 (m, 6H), 8.55 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.8, 70.2, 124.1, 127.1, 127.9, 128.4, 128.7, 128.9, 129.0, 129.8, 130.7, 130.9,132.4, 133.6, 135.9, 136.7, 197.3, 200.8 ppm; HRMS (ESI-TOF) calcd for $C_{20}H_{16}O_3$ ([M+H$^+$]) = 305.1172, Found 305.1180.

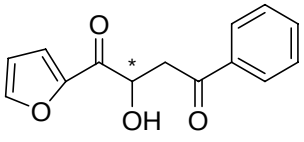






	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	41.562	178077	0.51	2536	0.56	39.283	43.583
2	45.734	34574217	99.49	448607	99.44	43.733	48.767

1-(furan-2-yl)-2-hydroxy-4-phenylbutane-1,4-dione (5p)

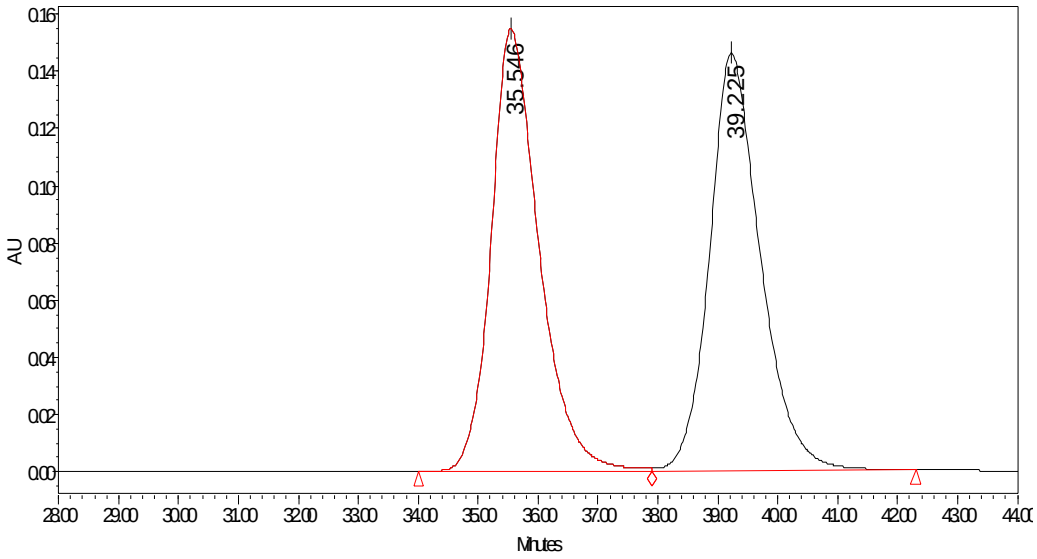


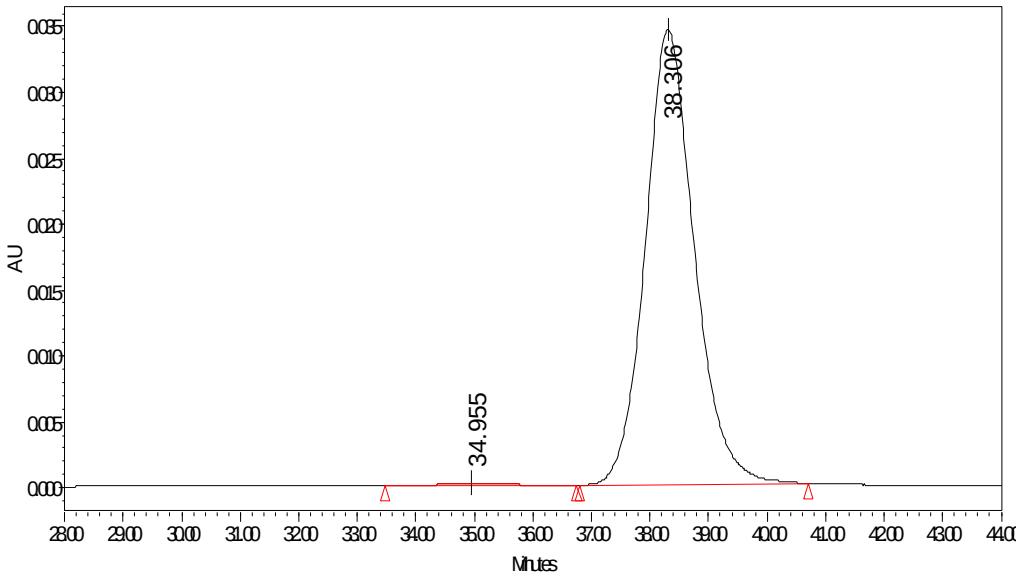
5p

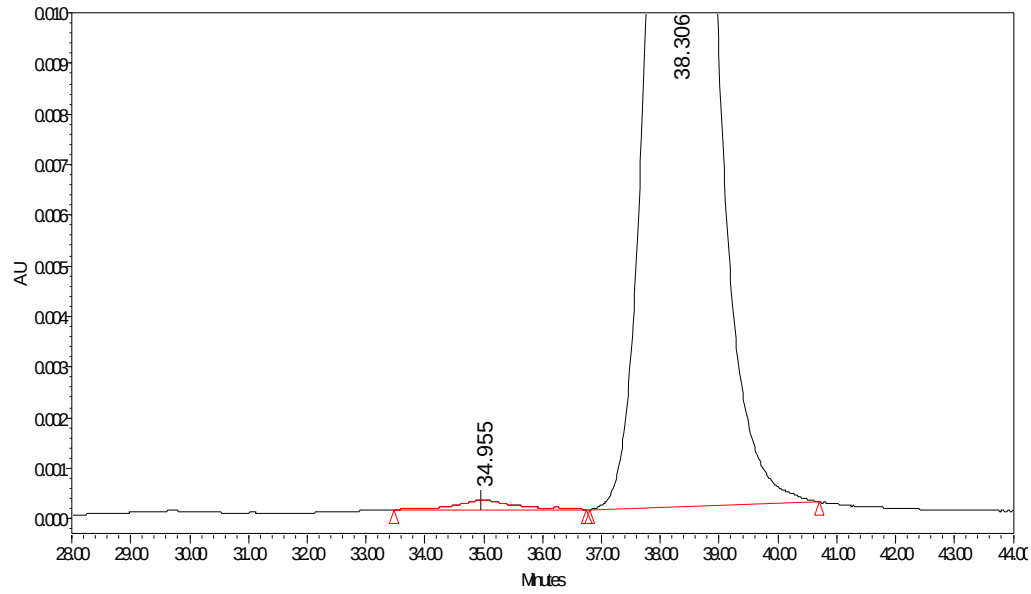
($C_{14}H_{12}O_4$) a white solid; 75% yield, 99% ee. $[\alpha]_D^{29}$ = +5.0 (*c* 0.180 in CH_2Cl_2)

HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 15/85, flow rate =

1.0 mL/min, λ = 254 nm, retention time: 35.0 min (minor) and 38.3 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.46-3.60 (m, 2H), 3.90 (d, *J* = 6.0 Hz, 1H), 5.30-5.34 (m, 1H), 6.59-6.60 (m, 1H), 7.45-7.50 (m, 3H), 7.56-7.63 (m, 2H), 7.96-7.98 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.1, 70.8, 112.7, 119.7, 128.3, 128.7, 133.6, 136.5, 147.1, 150.3, 188.8, 197.5 ppm; HRMS (ESI-TOF) calcd for $C_{14}H_{12}O_4$ ([M+Na^+]) = 267.0628, Found 67.0656.

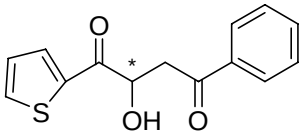






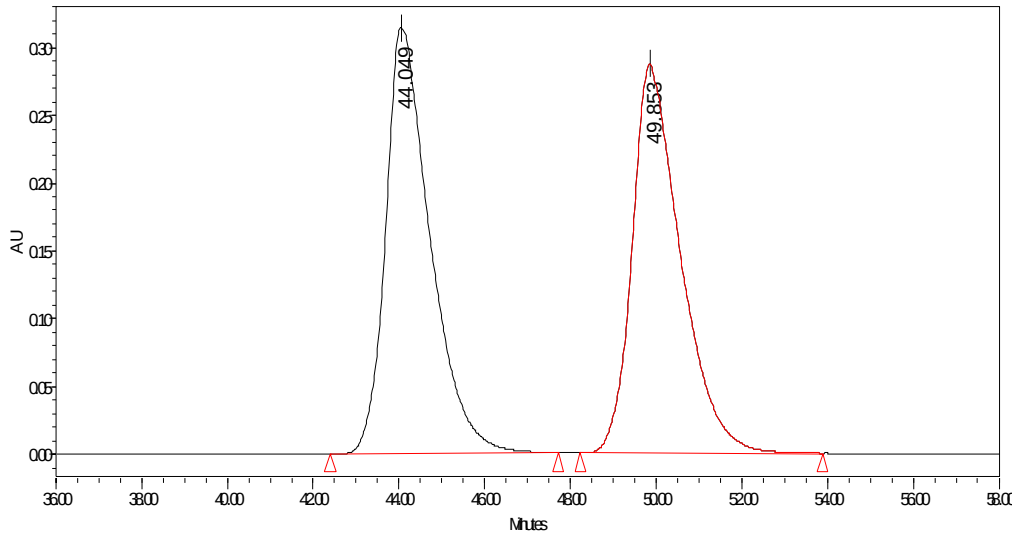
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	34.955	13858	0.69	194	0.56	33.467	36.750
2	38.306	2008563	99.31	34539	99.44	36.800	40.700

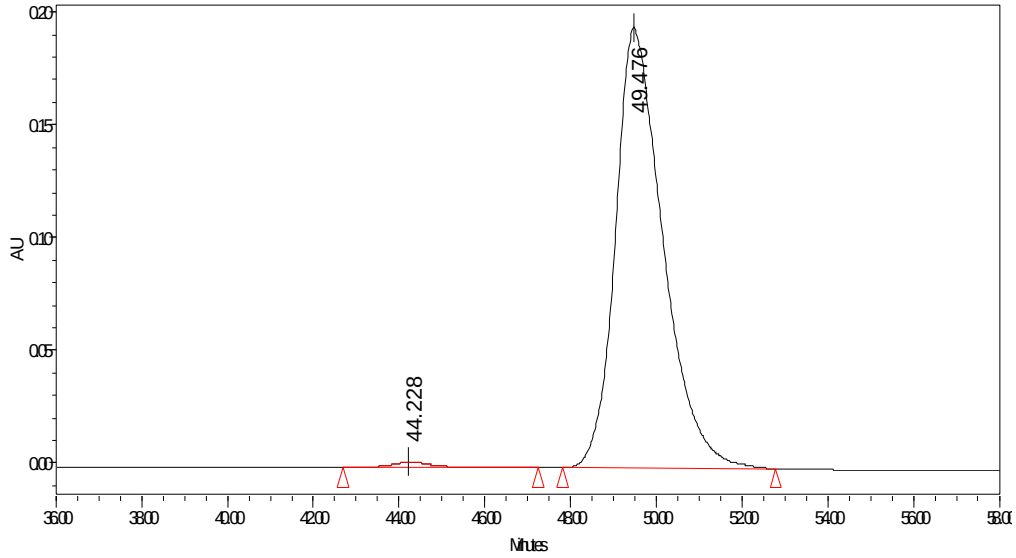
2-hydroxy-4-phenyl-1-(thiophen-2-yl)butane-1,4-dione (5q)



5q

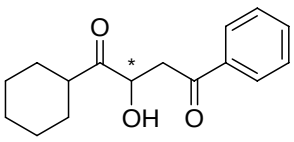
($C_{14}H_{12}SO_3$) a white solid; 86% yield, 98% ee. $[\alpha]_D^{25}$ = +58.6 (*c* 0.380 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 44.2 min (minor) and 49.5 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.43-3.57 (m, 2H), 3.98 (d, *J* = 4.4 Hz, 1H), 5.41-5.43 (m, 1H), 7.16-7.18 (m, 1H), 7.46-7.49 (m, 2H), 7.58-7.60 (m, 1H), 7.71-7.73 (m, 1H), 7.94-7.97 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.7, 71.6, 128.3, 128.4, 128.8, 133.8, 135.0, 136.5, 139.9, 193.1, 198.0 ppm; HRMS (ESI-TOF) calcd for $C_{14}H_{12}SO_3$ ([M+Na^+]) = 283.0399, Found 283.0387.





	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	44.228	173672	1.14	2555	1.29	42.700	47.250
2	49.476	15103936	98.86	195580	98.71	47.817	52.783

1-cyclohexyl-2-hydroxy-4-phenylbutane-1,4-dione (5r)

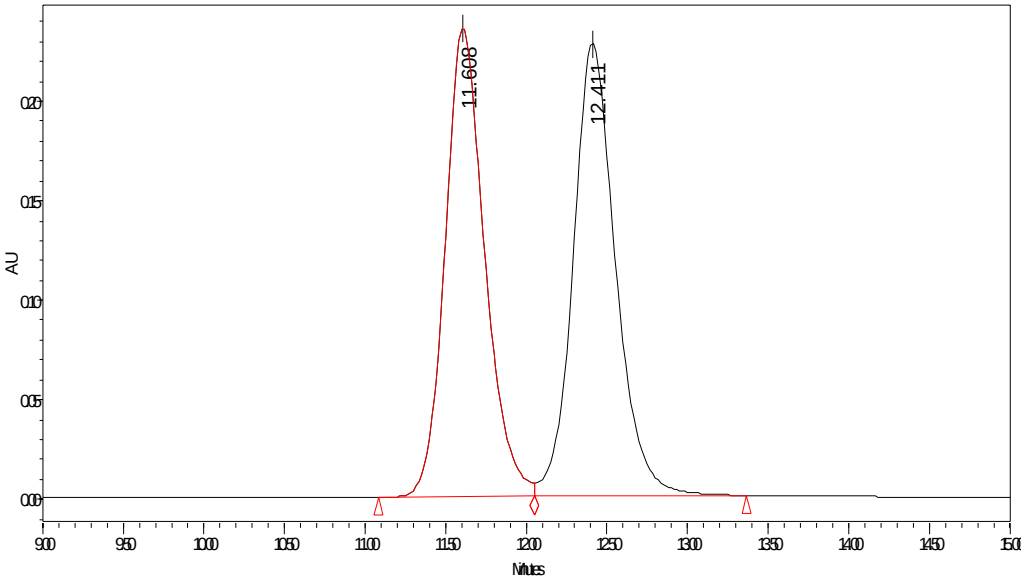


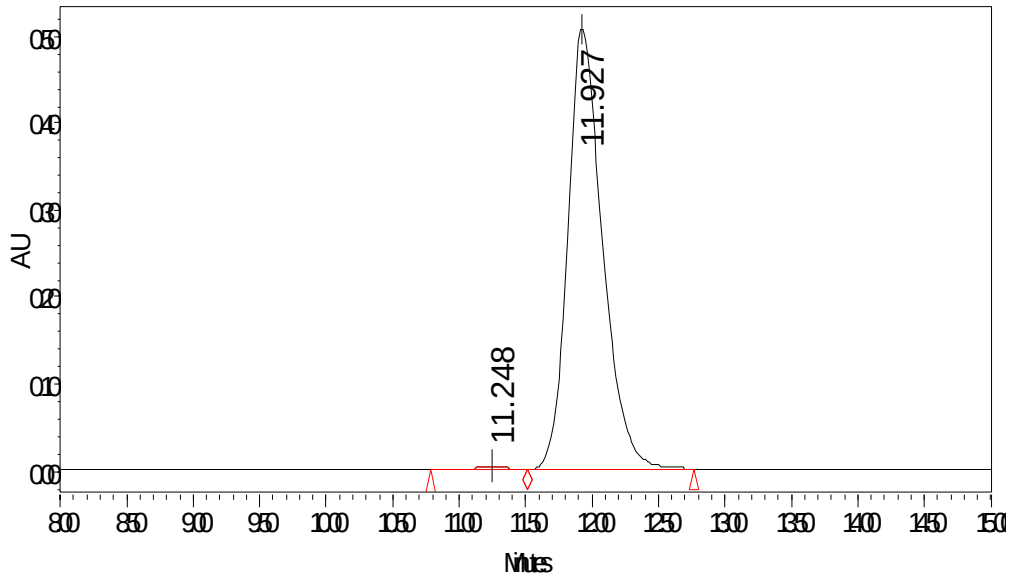
5r

($C_{16}H_{20}O_3$) a white solid; 70% yield, 99% ee. $[\alpha]_D^{25}$ = -4.6 (*c* 0.258 in CH_2Cl_2)

HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 11.2 min (minor) and 11.9 min (major);

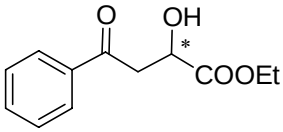
^{1}H NMR (400 MHz, $CDCl_3$) 1.24-1.38 (m, 5H), 1.41-1.52 (m, 1H), 1.59-1.87 (m, 4H), 2.80-2.82 (m, 1H), 3.34–3.54 (m, 2H), 3.82 (d, J_1 = 6.8 Hz, 1H), 4.70-4.71 (m, 1H), 7.26-7.50 (m, 2H), 7.58-7.62 (s, 1H), 7.94-7.97 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 25.4, 25.7, 25.8, 28.1, 29.2, 41.8, 45.8, 72.0, 128.3, 128.7, 133.7, 136.5, 198.3, 214.5 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{20}O_3$ ([M+Na$^+$]) = 283.1305, Found 283.1315.





	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	11.248	69610	0.75	4869	0.95	10.783	11.517
2	11.927	9215727	99.25	508244	99.05	11.517	12.767

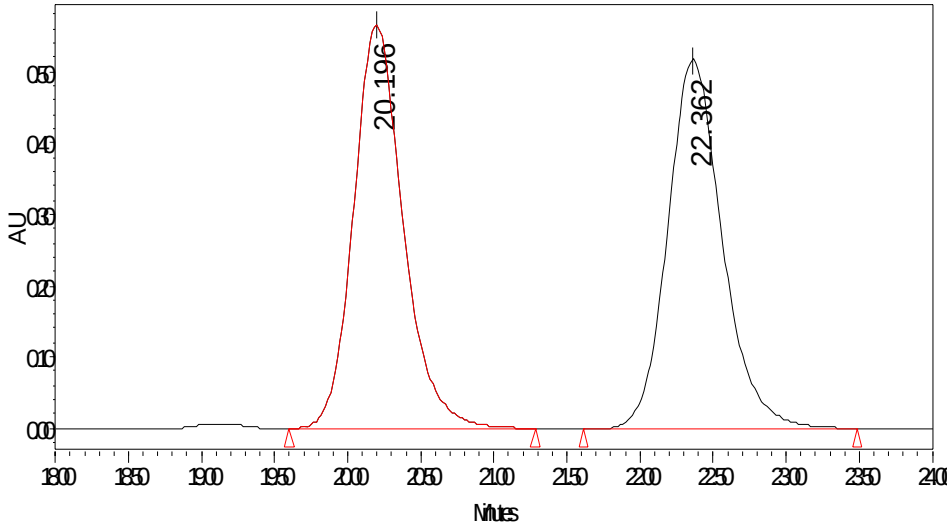
(*S*) 2-Hydroxy-4-oxo-4-phenyl-butyric acid ethyl ester (5s)

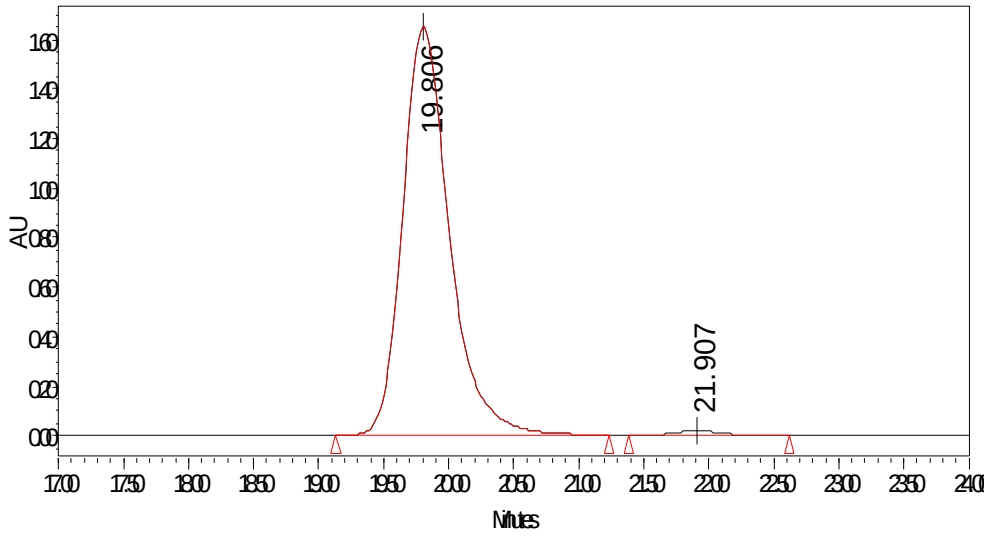


5s

($C_{12}H_{14}O_4$) a slightly yellow liquid; 85% yield, 98% ee. $[\alpha]_D^{29}$ = +6.2 (*c* 0.286 in CH_2Cl_2), $[\alpha]_D^{29}$ = -8.4 (*c* 0.300 in EtOH) HPLC DAICEL CHIRALCEL AD-H, 2-propanol/*n*-hexane = 20/80, flow rate = 0.5 mL/min, λ = 254 nm, retention time: 19.8 min (major) and 21.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.25-1.31 (m,3H), 3.36 (s, 1H), 3.43-3.50 (dd, *J* = 17.6, 4.0Hz, 1H), 3.50-3.57 (dd, *J* = 17.6, 6.0 Hz, 1H), 4.24-4.30 (m, 2H), 4.65-4.67 (m, 1H), 7.46-7.50 (m, 2H), 7.57-7.60 (m, 1H), 7.94-7.97 (m, 2H) ppm.

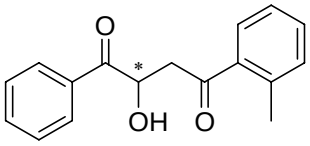
The absolute configuration was determined by the comparison of HPLC retention time and the optical rotation.[2a, 4]





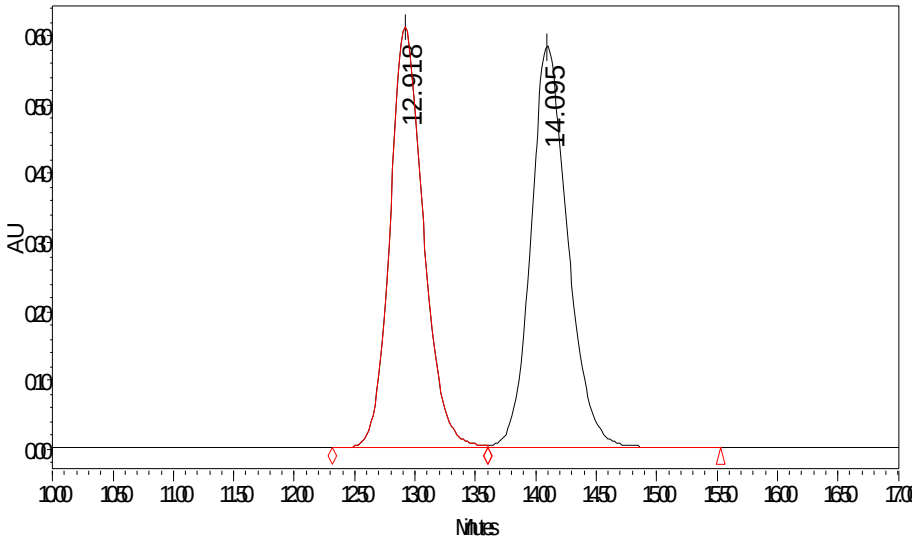
	Retention Time	Peak Type	Area	% Area	Height	% Height
1	19.806	Unknown	41228525	98.97	1650494	98.92
2	21.907	Unknown	427257	1.03	17951	1.08

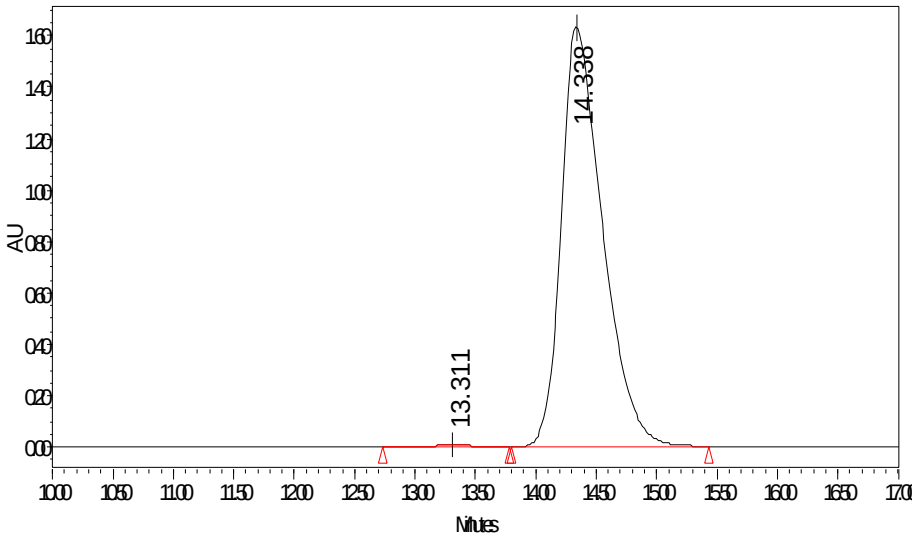
2-hydroxy-1-phenyl-4-o-tolylbutane-1,4-dione (5t)

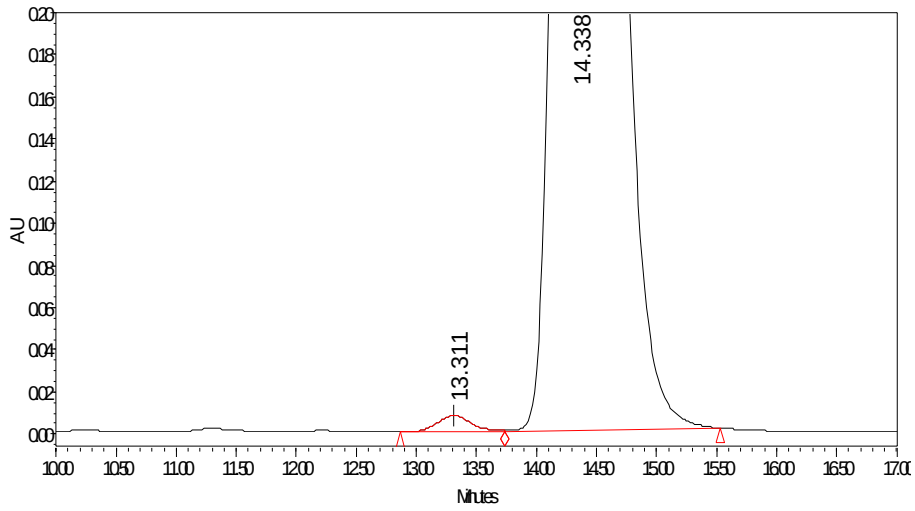


5t

($C_{17}H_{16}O_3$) a white solid; 92% yield, >99% ee. $[\alpha]_D^{25}$ = -8.1 (*c* 0.320 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 13.3 min (minor) and 14.3 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 2.51 (s, 3H), 3.30-3.33 (m, 2H), 4.03 (d, *J* = 6.0 Hz, 1H), 5.62-5.65 (m, 1H), 7.22-7.26 (m, 2H), 7.35-7.40 (m, 1H), 7.48-7.53 (m, 2H), 7.60-7.64 (m, 2H), 7.97-8.00 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 21.4, 46.4, 70.5, 125.8, 128.7, 128.9, 130.0, 131.8, 132.1, 133.6, 134.0, 137.2, 138.7, 200.7, 200.8 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_3$ ([M-H$^+$]) = 267.1021, Found 267.1028.

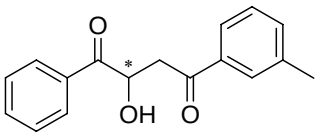

12.918
14.095
AU
Minutes


14.338
13.311
AU
Minutes



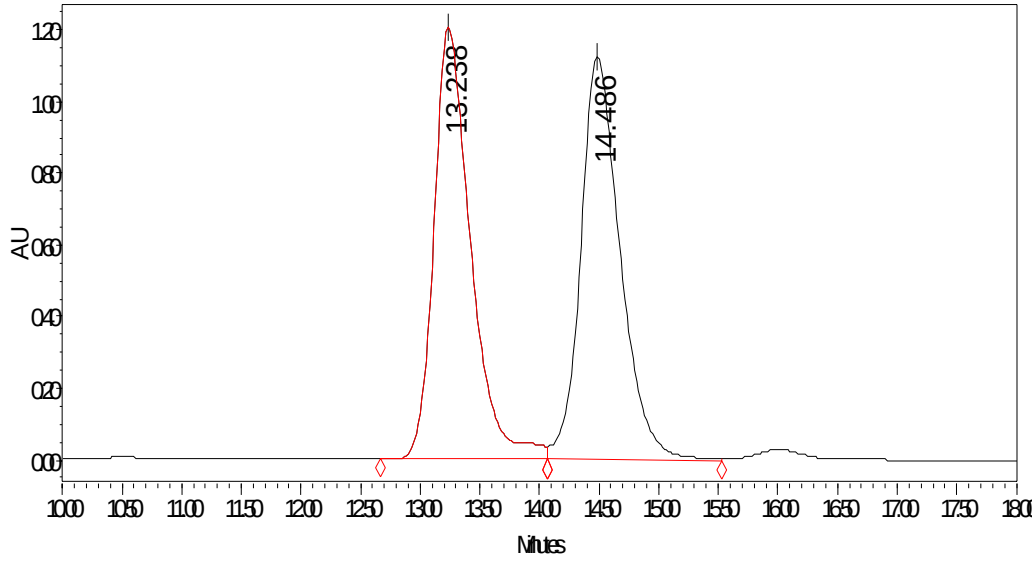
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	13.311	151393	0.39	7794	0.48	12.867	13.733
2	14.338	38777878	99.61	1631999	99.52	13.733	15.533

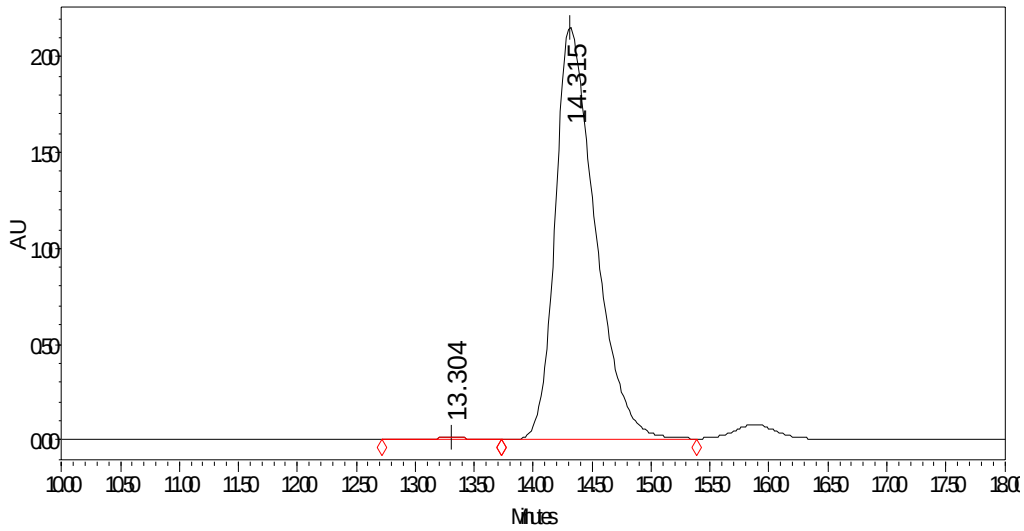
2-hydroxy-1-phenyl-4-m-tolylbutane-1,4-dione (5u)

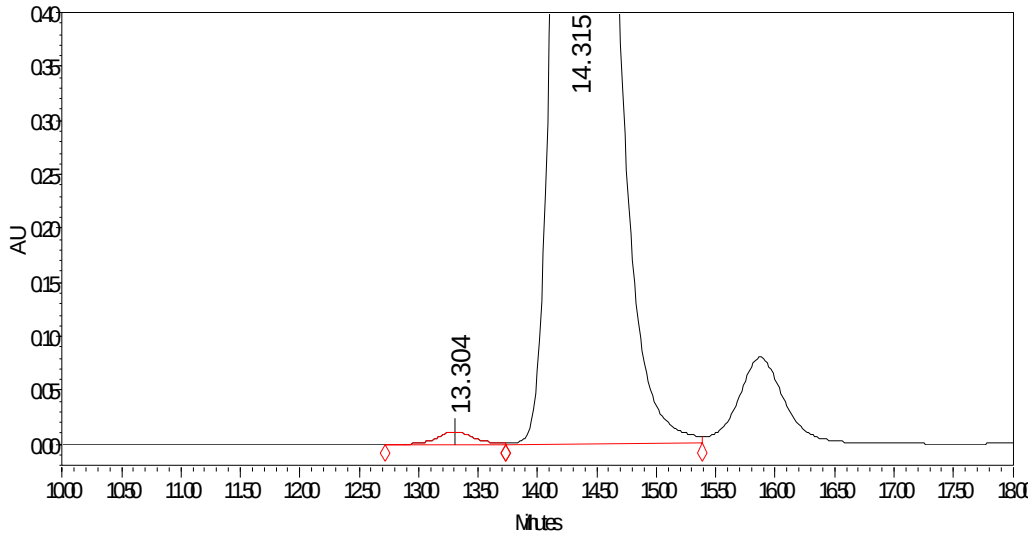


5u

($C_{17}H_{16}O_3$) a white solid; 92% yield, >99% ee. $[\alpha]_D^{25}$ = +10.8 (*c* 0.426 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 13.3 min (minor) and 14.3 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 2.40 (s, 3H), 3.33-3.45 (m, 2H), 4.02 (d, *J* = 6.0 Hz, 1H), 5.65-5.69 (m, 1H), 7.31-7.39 (m, 2H), 7.47-7.52 (m, 2H), 7.59-7.63 (m, 1H), 7.72-7.75 (m, 2H), 7.86-7.89 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 21.3, 43.6, 70.1, 125.6, 128.6, 128.7, 128.8, 130.0, 133.6, 133.9, 134.4, 136.7, 138.5, 197.4, 200.8 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_3$ ([M-H$^+$]) = 267.1021, Found 267.1035.

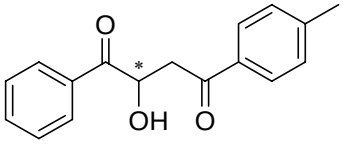

13.238
14.486
AU
Minutes


14.315
13.304
AU
Minutes



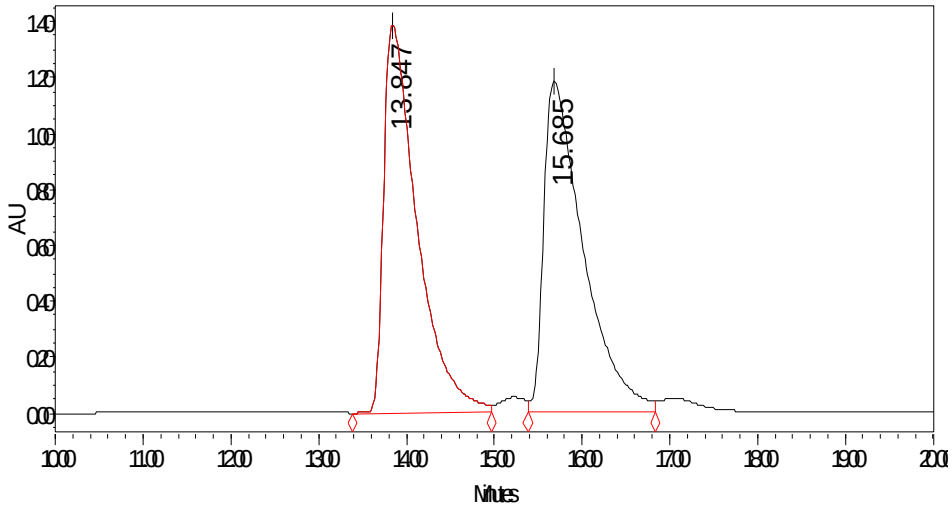
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	13.304	244026	0.48	11522	0.53	12.717	13.733
2	14.315	50733155	99.52	2153255	99.47	13.733	15.383

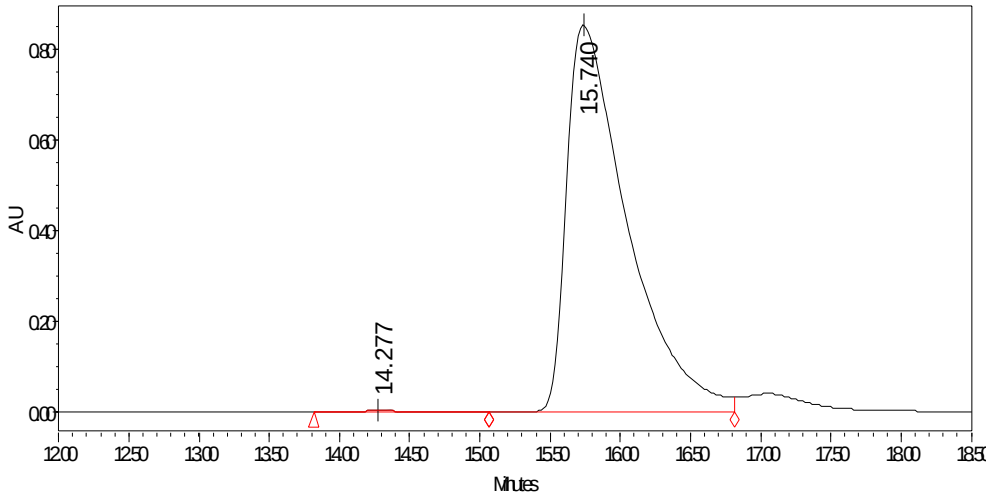
2-hydroxy-1-phenyl-4-p-tolylbutane-1,4-dione (5v)

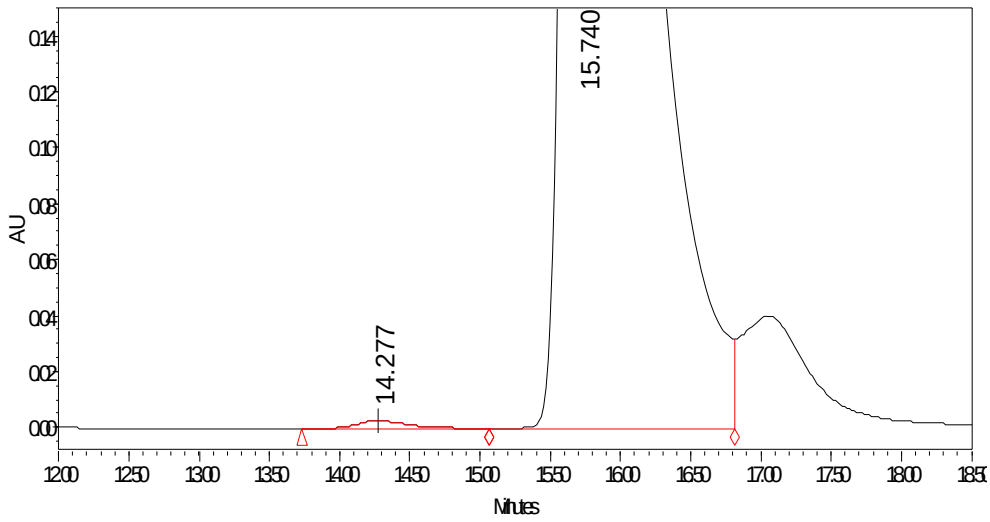


5v

($C_{17}H_{16}O_3$) a white solid; 94% yield, >99% ee. $[\alpha]_D^{25}$ = +16.8 (*c* 0.384 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 14.3 min (minor) and 15.7 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 2.40 (s, 3H), 3.32-3.43 (m, 2H), 4.03 (d, *J* = 6.0 Hz, 1H), 5.64-5.69 (m, 1H), 7.23-7.26 (m, 2H), 7.47-7.51 (m, 2H), 7.59-7.62 (m, 1H), 7.82-7.85 (m, 2H), 7.98-8.03 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 21.7, 43.4, 70.2, 128.5, 128.7, 128.9, 129.4, 133.7, 133.8, 134.3, 144.5, 196.8, 200.8 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_3$ ([M-H$^+$]) = 267.1021, Found 267.1022.

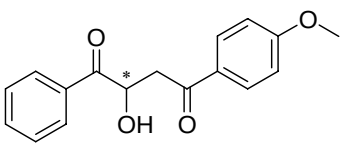

13.847
15.685
AU
Minutes


15.740
14.277
AU
Minutes


15.740
14.277
AU
Minutes

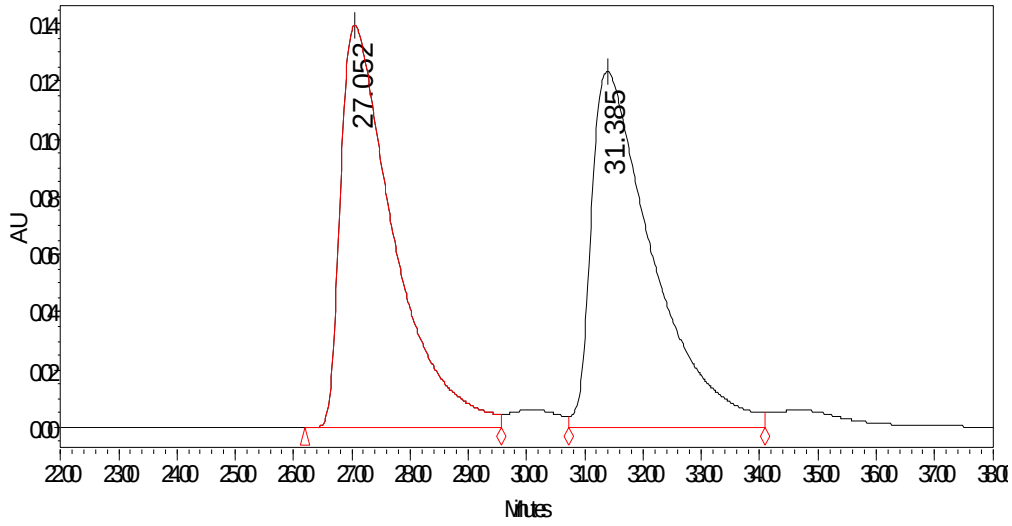
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	14.277	71443	0.28	2779	0.32	13.733	15.067
2	15.740	25888176	99.72	855497	99.68	15.067	16.817

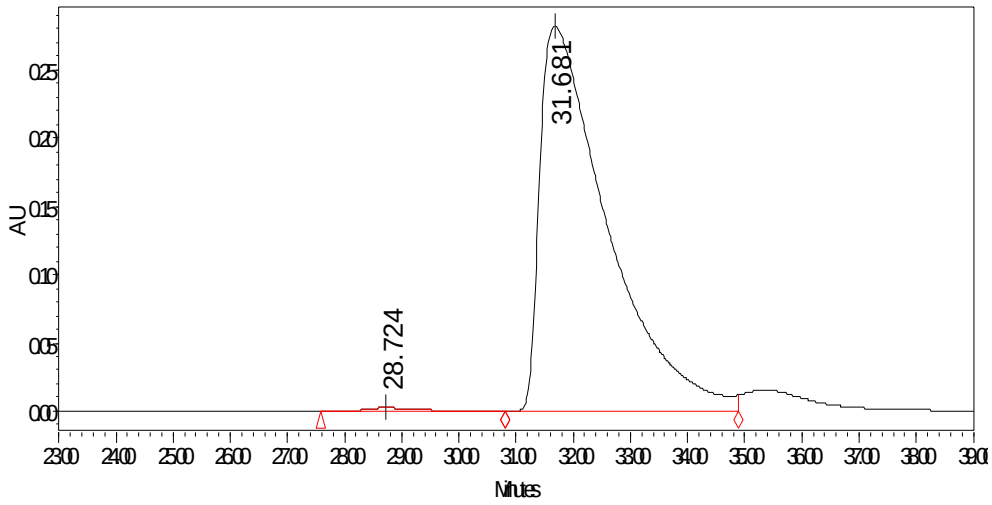
2-hydroxy-4-(4-methoxyphenyl)-1-phenylbutane-1,4-dione (5w)



5w

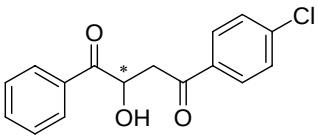
($C_{17}H_{16}O_4$) a white solid; 95% yield, 99% ee. $[\alpha]_D^{25}$ = +4.8 (*c* 0.460 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 28.7 min (minor) and 31.7 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.32-3.44 (m, 2H), 3.88 (s, 3H), 4.05 (d, *J* = 6.0 Hz, 1H), 5.66-5.70 (m, 1H), 6.93-6.96 (m, 2H), 7.50-7.61 (m, 2H), 7.62-7.65 (m, 1H), 7.93-7.97 (m, 2H), 8.00-8.03 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.2, 55.5, 70.3, 113.8, 128.8, 128.9, 129.8, 130.7, 133.7, 133.9, 163.9, 195.7, 200.9 ppm; HRMS (ESI-TOF) calcd for $C_{17}H_{16}O_4$ ([M+H$^+$]) = 285.1121, Found 285.1132.





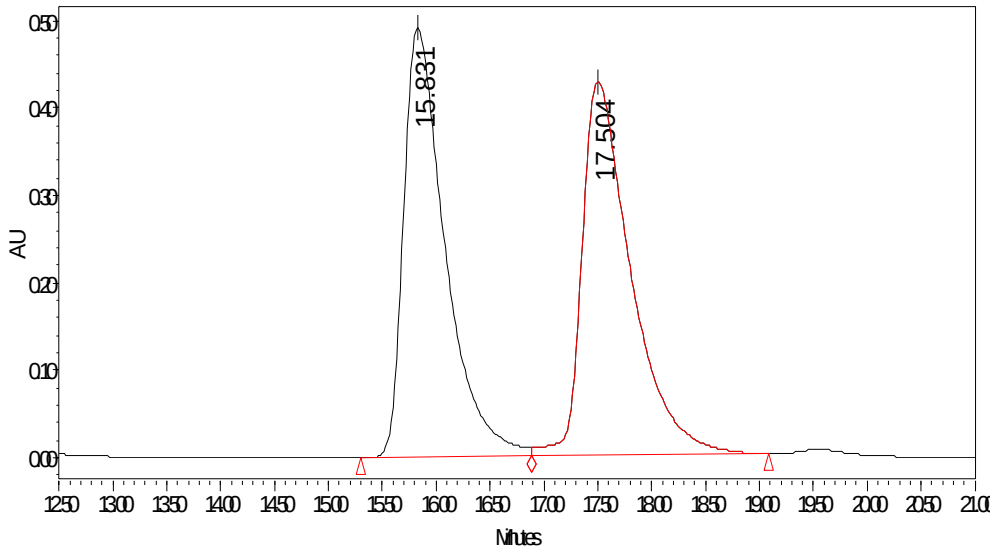
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	28.724	172509	0.74	2619	0.92	27.583	30.817
2	31.681	23155692	99.26	281826	99.08	30.817	34.883

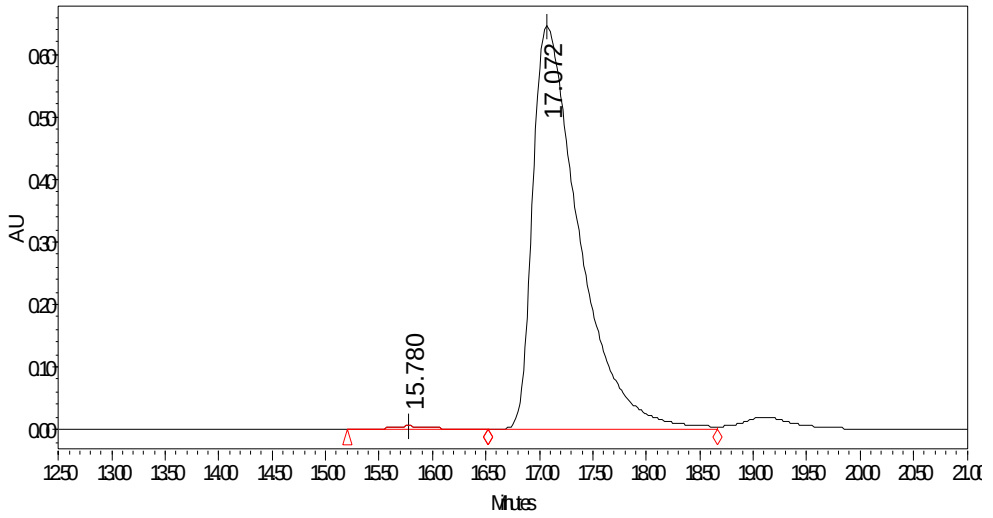
4-(4-chlorophenyl)-2-hydroxy-1-phenylbutane-1,4-dione (5x)



5x

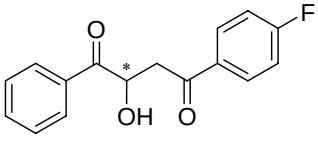
($C_{16}H_{13}ClO_3$) a white solid; 87% yield, 99% ee. $[\alpha]_D^{25}$ = +4.8 (*c* 0.540 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 15.8 min (minor) and 17.1 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.32-3.44 (m, 2H), 4.03 (d, *J* = 6.0 Hz, 1H), 5.66-5.70 (m, 1H), 7.44-7.46 (m, 2H), 7.52-7.56 (m, 2H), 7.62-7.68 (m, 1H), 7.89-7.92 (m, 2H), 8.00-8.02 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.5, 70.1, 128.7, 129.0, 129.1, 129.8, 133.5, 134.1, 135.0, 140.2, 196.1, 200.6 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{13}ClO_3$ ([M+Na^+]) = 311.0445, Found 311.0454.





	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	15.780	105041	0.54	4315	0.66	15.200	16.517
2	17.072	19492728	99.46	644712	99.34	16.517	18.667

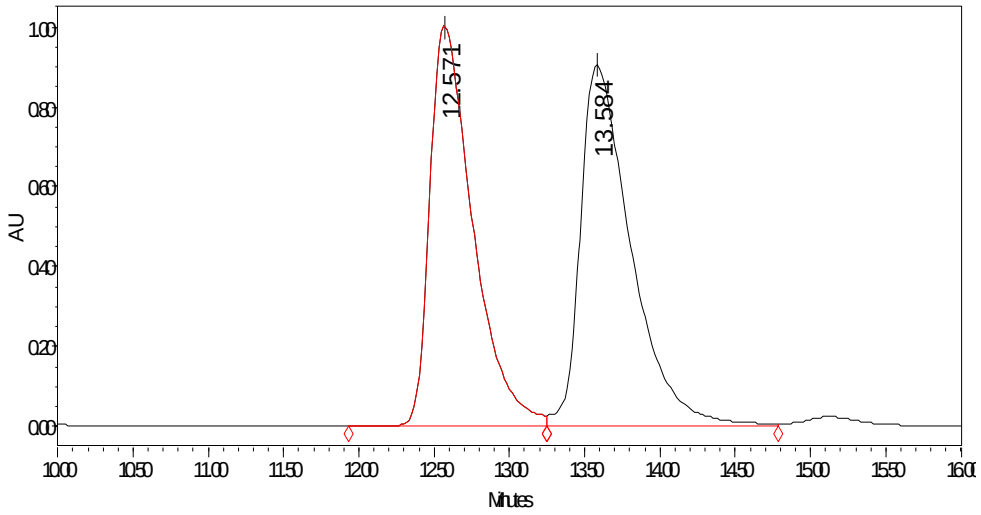
4-(4-fluorophenyl)-2-hydroxy-1-phenylbutane-1,4-dione (5y)

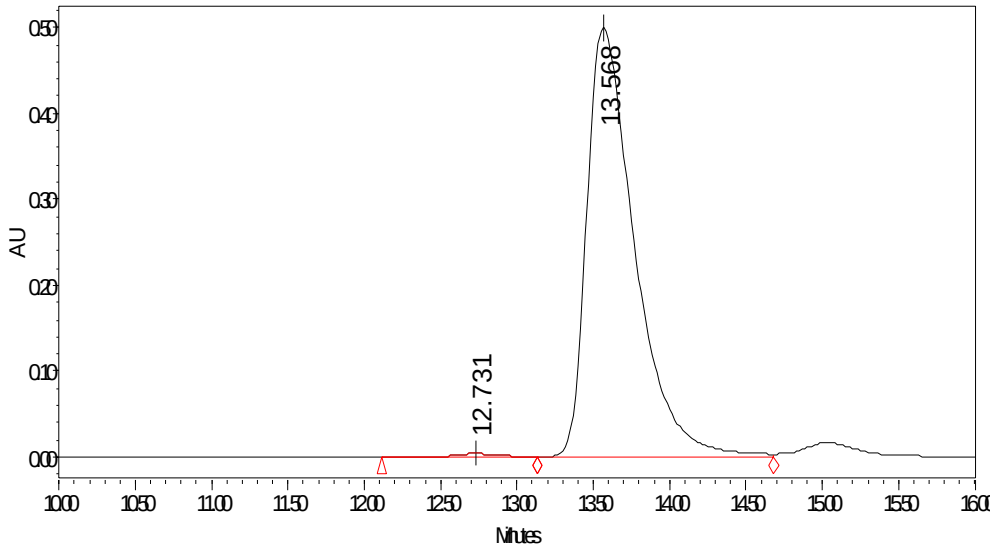


5y

($C_{16}H_{13}FO_3$) a white solid; 87% yield, 99% ee. $[\alpha]_D^{25}$ = +12.7 (*c* 0.448 in CH_2Cl_2) HPLC DAICEL CHIRALCEL IB, 2-propanol/*n*-hexane = 10/90,

flow rate = 1.0 mL/min, λ = 254 nm, retention time: 12.7 min (minor) and 13.6 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.31-3.40 (m, 2H), 4.03 (d, J = 5.6 Hz, 1H), 5.64-5.68 (m, 1H), 7.10-7.20 (m, 2H), 7.48-7.52 (m, 2H), 7.59-7.64 (m, 1H), 7.95-8.00 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.5, 70.1, 115.8(d, J_{CF} = 22 Hz), 128.7, 129.0, 131.0 (d, J_{CF} = 9 Hz), 133.1 (d, J_{CF} = 4 Hz), 133.5, 134.0, 166.0 (d, J_{CF} = 255 Hz), 195.6, 200.7 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{13}FO_3$ ([M+Na^+]) = 295.0741, Found 295.0748.

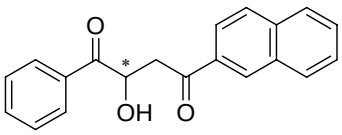




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time

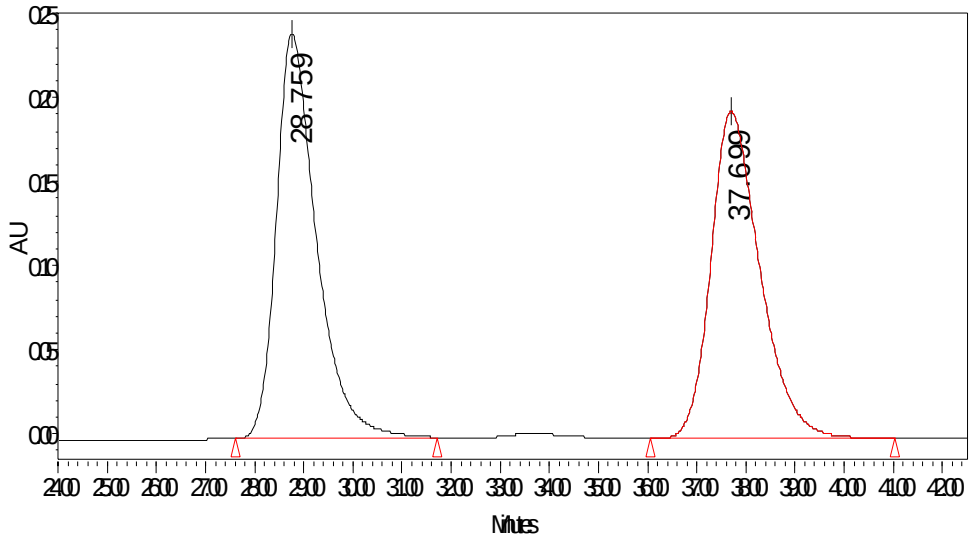
1	12.731	63214	0.58	3433	0.68	12.117	13.133
2	13.568	10838880	99.42	499134	99.32	13.133	14.683

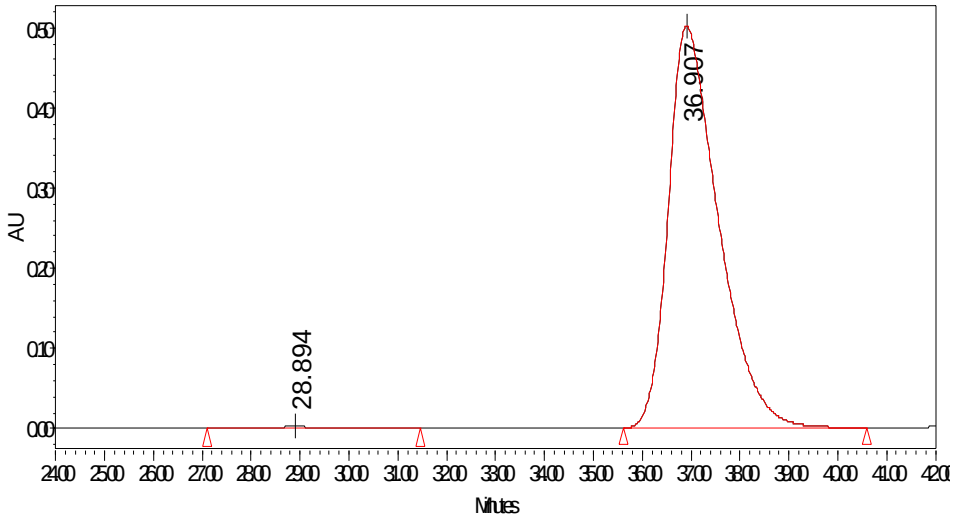
2-hydroxy-1-phenyl-4-m-tolylbutane-1,4-dione (5z)

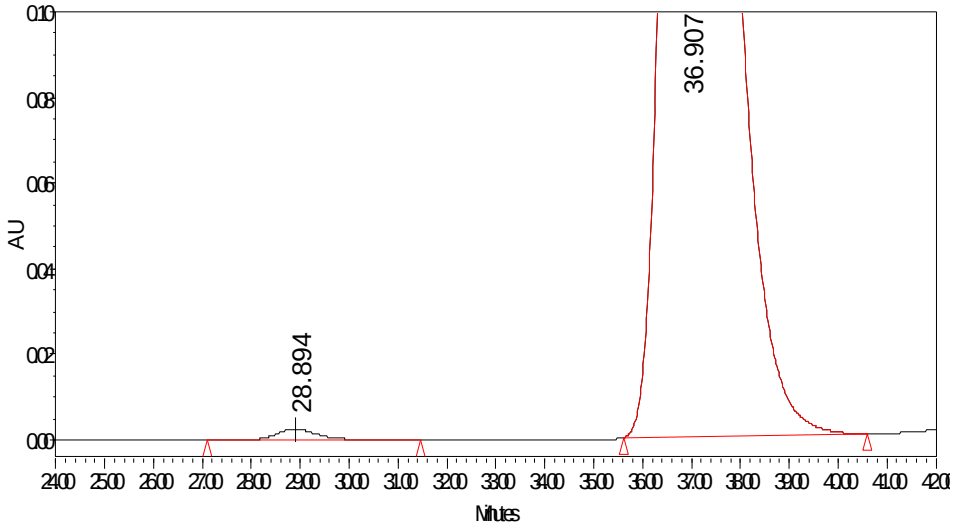


5z

($C_{20}H_{16}O_3$) a white solid; 91% yield, >99% ee. $[\alpha]_D^{25}$ = -5.1 (*c* 0.440 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 30/70, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 28.9 min (minor) and 36.9 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) 3.47-3.60 (m, 2H), 4.09 (d, *J* = 6.0 Hz, 1H), 5.62-5.65 (m, 1H), 7.50-7.63 (m, 5H), 7.85-7.93 (m, 3H), 8.00-8.04 (m, 3H), 8.43 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 43.6, 70.2, 123.8, 126.9, 127.8, 128.6, 128.8, 129.0, 129.7, 130.4, 132.4, 133.7, 134.0, 135.8, 197.1, 200.8 ppm; HRMS (ESI-TOF) calcd for $C_{20}H_{16}O_3$ ([M+H$^+$]) = 305.1172, Found 305.1178.



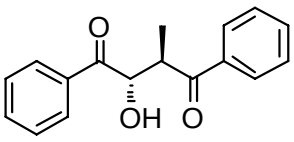




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	28.894	157801	0.44	2644	0.52	27.100	31.467
2	36.907	35818752	99.56	501377	99.48	35.617	40.600

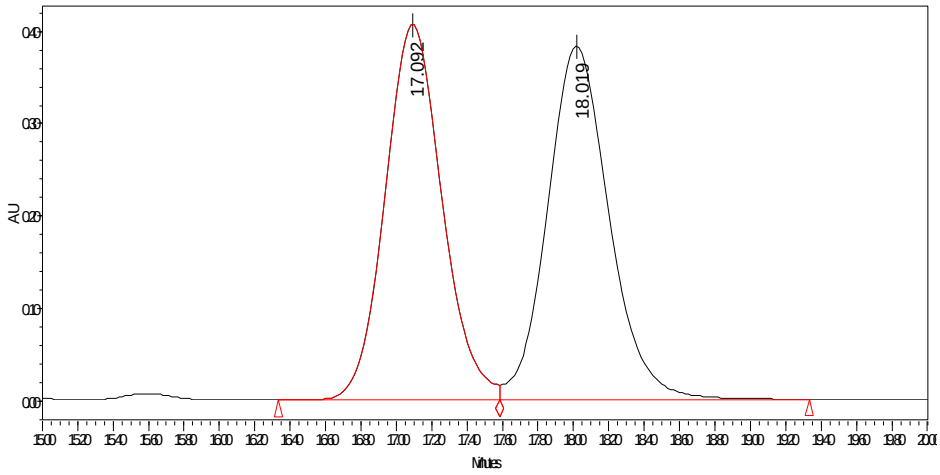
Configuration Assignment: The absolute and relative configurations were determined by the comparison of the optical rotation and ^{1}H NMR Spectra.[2]

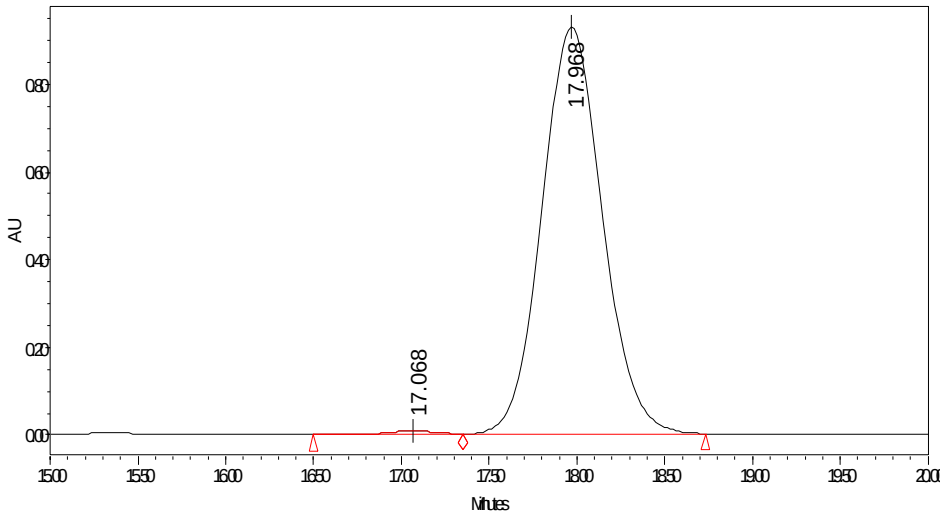
(*2S,3R*)-2-hydroxy-3-methyl-1,4-diphenylbutane-1,4-dione (*anti*-5aa)



anti-**5aa**

($C_{17}H_{16}O_3$) a slightly yellow liquid; 88% yield, 99% ee. $[\alpha]_D^{25}$ = 84.1 (*c* 0.156 in CH_2Cl_2) HPLC DAICEL CHIRALCEL AD-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 17.1 min (minor) and 18.0 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) *anti*: 1.27 (d, *J* = 7.6 Hz, 3H), 3.89-3.95 (m, 1H), 4.30 (d, *J* = 9.2 Hz, 1H), 5.13 (dd, J_1 = 4.4 Hz, J_2 = 9.2 Hz, 1H), 7.37-7.41 (m, 4H), 7.49-7.53 (m, 2H), 7.84 (d, *J* = 7.2 Hz, 2H), 7.91 (d, *J* = 7.6 Hz, 2H) ppm.

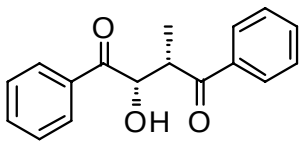




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	17.068	166332	0.74	8142	0.87	16.500	17.350
2	17.968	22238348	99.26	928889	99.13	17.350	18.717

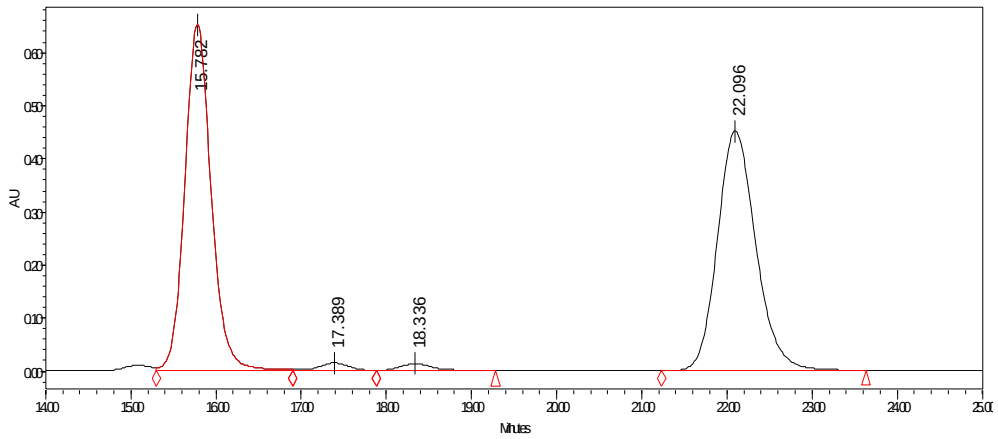
Configuration Assignment: The absolute and relative configurations were determined by the comparison of the optical rotation and ^{1}H NMR Spectra. [2]

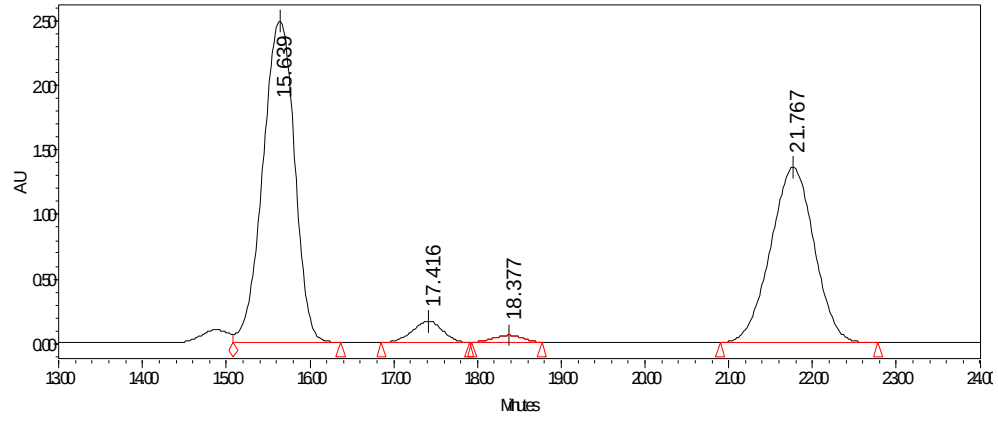
(*2S,3S*)-2-hydroxy-3-methyl-1,4-diphenylbutane-1,4-dione (*syn*-5aa)



syn-**5aa**

($C_{17}H_{16}O_3$) a slightly yellow liquid; 75% yield, 18% ee. HPLC DAICEL CHIRALCEL AD-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 15.8 min and 22.1 min or HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 15/85, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 18.1 min and 20.6 min; ^{1}H NMR (400 MHz, $CDCl_3$) *syn*: 1.04 (d, *J* = 7.2 Hz, 3H), 3.63 (d, *J* = 6.0 Hz, 1H), 3.72-3.79 (m, 1H), 5.40 (s, 1H), 7.37-7.59 (m, 6 H), 7.82 (d, *J* = 7.6 Hz, 2H), 7.86 (d, *J* = 7.6 Hz, 2H) ppm.

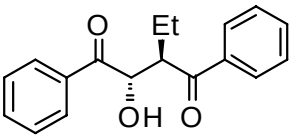




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	15.639	65732057	54.55	2497752	61.26	15.083	16.367
2	17.416	4091875	3.40	162945	4.00	16.850	17.900
3	18.377	1341326	1.11	55496	1.36	17.933	18.767
4	21.767	49324722	40.94	1360830	33.38	20.900	22.783

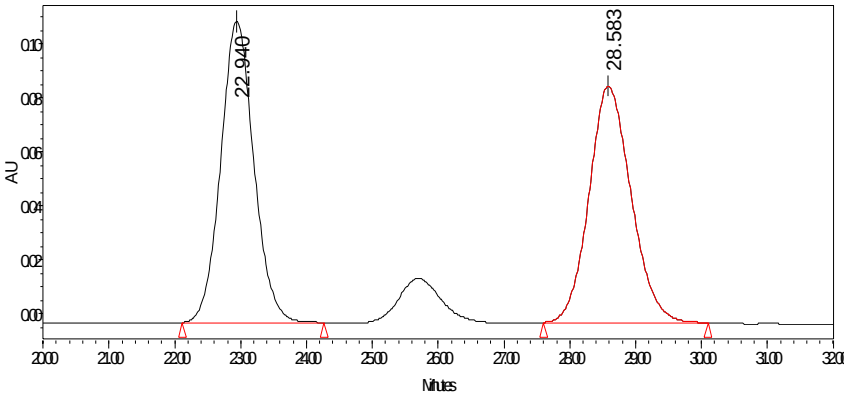
Configuration Assignment: The absolute and relative configurations were determined by analogy.

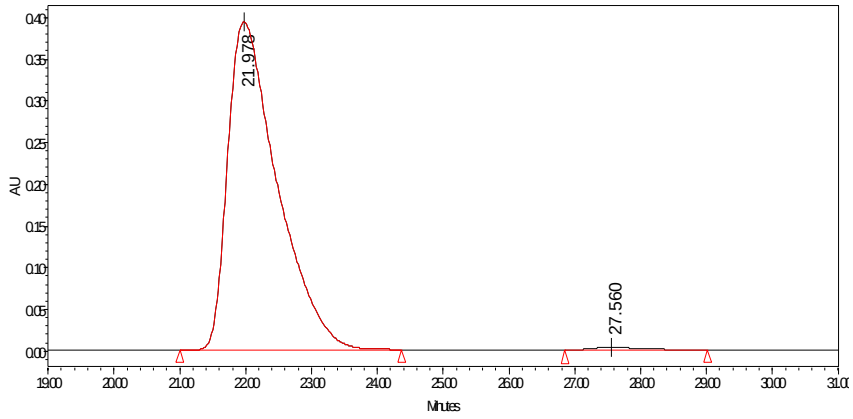
(*2S,3R*)-2-hydroxy-3-ethyl-1,4-diphenylbutane-1,4-dione (*anti*-5ab)



anti-**5ab**

($C_{18}H_{18}O_3$) a slightly yellow liquid; 83% yield, 98% ee. $[\alpha]_D^{25} = 99.4$ (*c* 0.332 in CH_2Cl_2) HPLC DAICEL CHIRALCEL OJ-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 22.0 min (major) and 27.6 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) *anti*: 1.01-1.08 (m, 3H), 1.82-1.94 (m, 2H), 3.80-3.85 (m, 1H), 4.49 (d, *J* = 6.0 Hz, 1H), 5.24 (dd, J_1 = 4.0 Hz, J_2 = 9.6 Hz, 1H), 7.42-7.50 (m, 4H), 7.54-7.60 (m, 2H), 7.87 (d, *J* = 7.2 Hz, 2H), 7.95 (d, *J* = 7.6 Hz, 2H) ppm, ^{13}C NMR (100 MHz, $CDCl_3$) 12.3, 22.8, 49.1, 75.2, 128.4, 128.6, 128.7, 129.2, 133.4, 133.6, 135.1, 137.2, 200.3, 205.3 ppm; HRMS (ESI-TOF) calcd for $C_{18}H_{18}O_3$ ([M+Na$^+$]) = 305.1148, Found 305.1156.

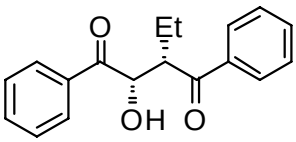




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	21.978	20614971	98.95	393604	99.03	21.000	24.367
2	27.560	217803	1.05	3870	0.97	26.850	29.017

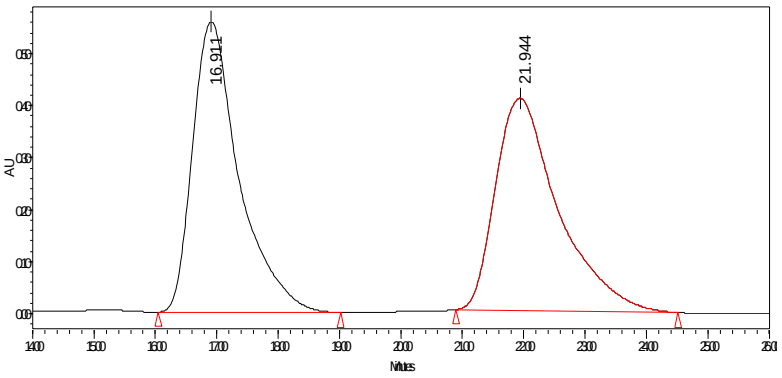
Configuration Assignment: The absolute and relative configurations were determined by analogy.

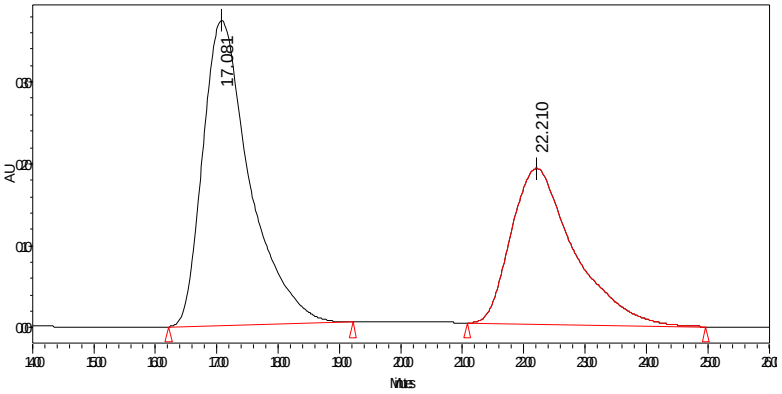
(*2S,3S*)-2-hydroxy-3-ethyl-1,4-diphenylbutane-1,4-dione (*syn*-5ab)



syn-**5ab**

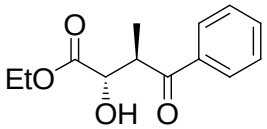
($C_{18}H_{18}O_3$) a slightly yellow liquid; 76% yield, 16% ee. $[\alpha]_D^{25}$ = 23.9 (*c* 0.244 in CH_2Cl_2) HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 10/90, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 14.1 min (major) and 22.2 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) *syn*: 0.79-0.82 (m, 3H), 1.55-1.64 (m, 2H), 3.58 (d, *J* = 6.0 Hz, 1H), 3.70-3.77 (m, 1H), 5.32-5.35 (m, 1H), 7.44-7.50 (m, 4H), 7.52-7.60 (m, 2H), 7.87 (d, *J* = 8.0 Hz, 4H) ppm, ^{13}C NMR (100 MHz, $CDCl_3$) 12.1, 21.4, 52.2, 73.6, 128.0, 128.2, 128.7, 128.8, 133.2, 133.9, 134.8, 137.3, 201.0, 201.3 ppm.





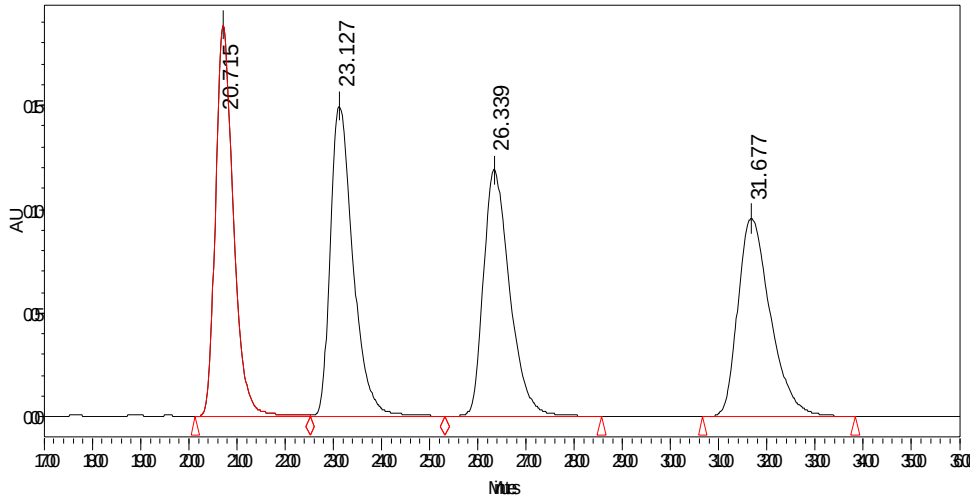
Configuration Assignment: The absolute and relative configurations were determined by the comparison of the optical rotation and ^{1}H NMR Spectra.[2]

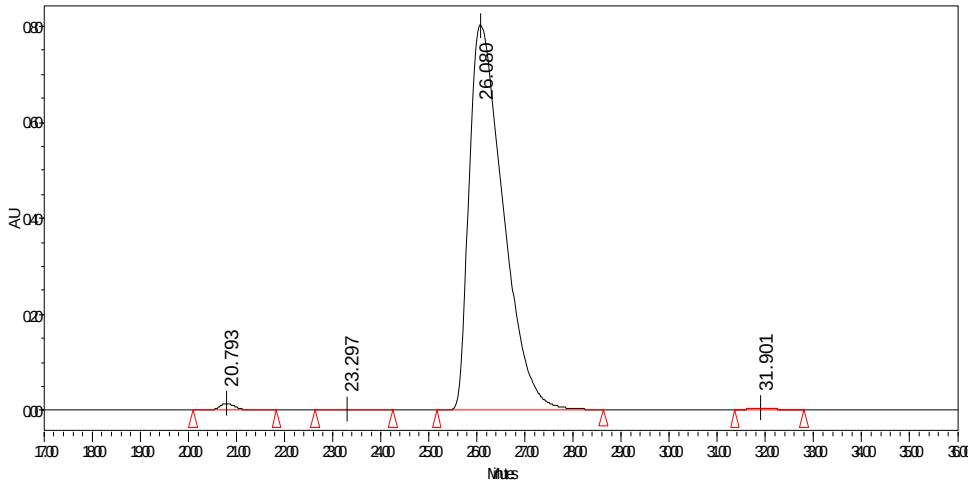
(*2S,3R*)-ethyl- 2-hydroxy-3-methyl-4-oxo-4-phenylbutanoate (*anti*-5ac)



anti-**5ac**

($C_{13}H_{16}O_4$) a slightly yellow liquid; 85% yield, *anti*/*syn* = 99:1, >99% ee (*anti*). $[\alpha]_D^{25}$ = 20.6 (*c* 0.344 in CH_2Cl_2) HPLC DAICEL CHIRALCEL AS-H+AD-H, 2-propanol/*n*-hexane = 20/80, flow rate = 0.8 mL/min, λ = 254 nm, retention time: 20.8 min，23.3 min, 26.1 min and 31.9 min; ^{1}H NMR (400 MHz, $CDCl_3$) *anti*: 1.20 (t, *J* = 7.2 Hz, 3H), 1.37 (d, *J* = 7.2 Hz, 3H), 3.56 (d, *J* = 6.0 Hz, 1H), 3.94-4.01 (m, 1H), 4.11-4.20 (m, 2H), 4.39 (s, 1H), 7.46-7.50 (m, 2H), 7.57-7.61 (m, 1H), 7.92-7.94 (m, 2H) ppm.

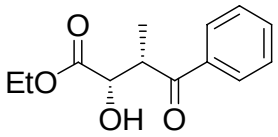




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	20.793	346619	0.87	13808	1.68	20.100	21.833
2	23.297	82462	0.21	2717	0.33	22.633	24.250
3	26.080	39259433	98.47	801935	97.46	25.167	28.633
4	31.901	180285	0.45	4411	0.54	31.367	32.800

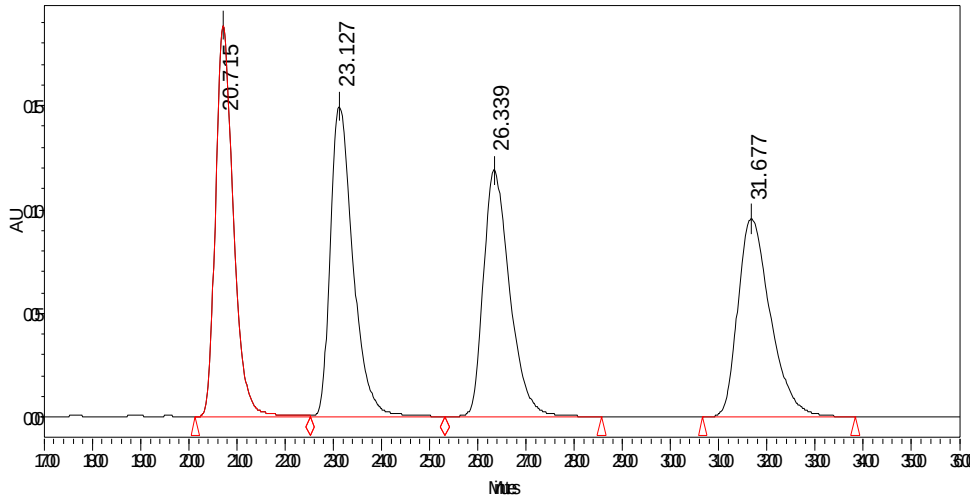
Configuration Assignment: The absolute and relative configurations were determined by the comparison of the optical rotation and ^{1}H NMR Spectra. [2]

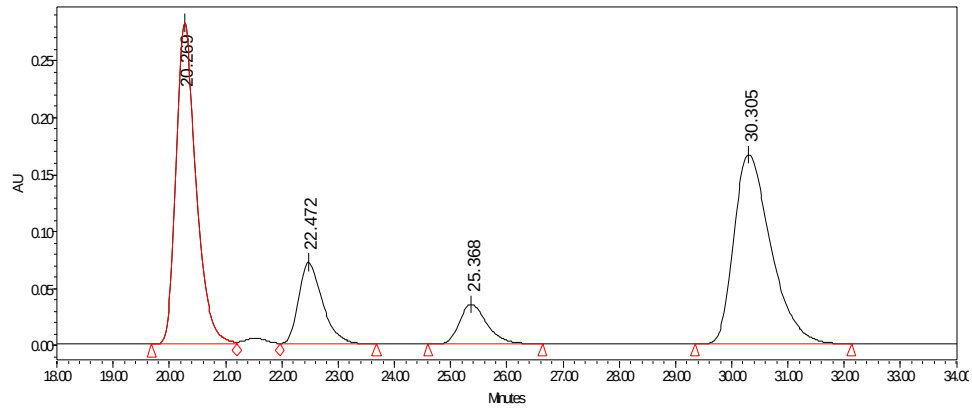
(*2S,3S*)-ethyl -2-hydroxy-3-methyl-4-oxo-4-phenylbutanoate (*syn*-5ac)



syn-**5ac**

($C_{13}H_{16}O_4$) a slightly yellow liquid; 73% yield, *anti*/*syn* = 50:50, -73% ee (*anti*), 55% ee (*syn*). $[\alpha]_D^{25}$ = 7.6 (*c* 0.278 in CH_2Cl_2) HPLC DAICEL CHIRALCEL AS-H+AD-H, 2-propanol/*n*-hexane = 20/80, flow rate = 0.8 mL/min, λ = 254 nm, retention time: 20.8 min，23.3 min, 26.1 min and 31.9 min; ^{1}H NMR (400 MHz, $CDCl_3$) *syn*: 1.13 (t, *J* = 7.2 Hz, 3H), 1.22 (d, *J* = 7.2 Hz, 3H), 3.19 (d, *J* = 4.4 Hz, 1H), 3.87-3.90 (m, 1H), 4.07-4.16 (m, 2H), 4.30-4.35 (m, 1H), 7.40-7.45 (m, 2H), 7.51-7.56 (m, 1H), 7.85-7.91 (m, 2H) ppm.



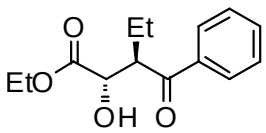


	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	20.269	6956922	38.99	281849	50.82	19.683	21.200
2	22.472	2118783	11.87	71757	12.94	21.967	23.683
3	25.368	1184932	6.64	34828	6.28	24.600	26.633

4	30.305	7584457	42.50	166172	29.96	29.350	32.133

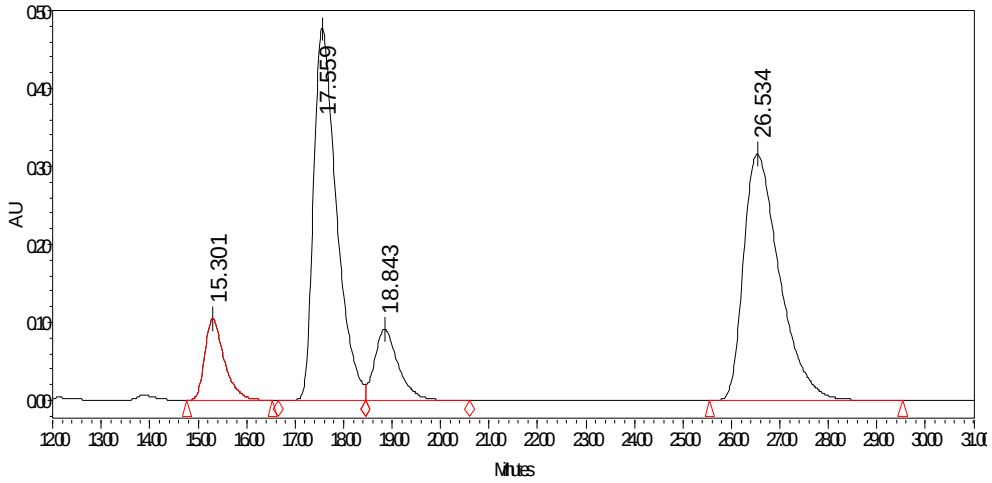
Configuration Assignment: The absolute and relative configurations were determined by the comparison of the optical rotation and ^{1}H NMR Spectra. [2]

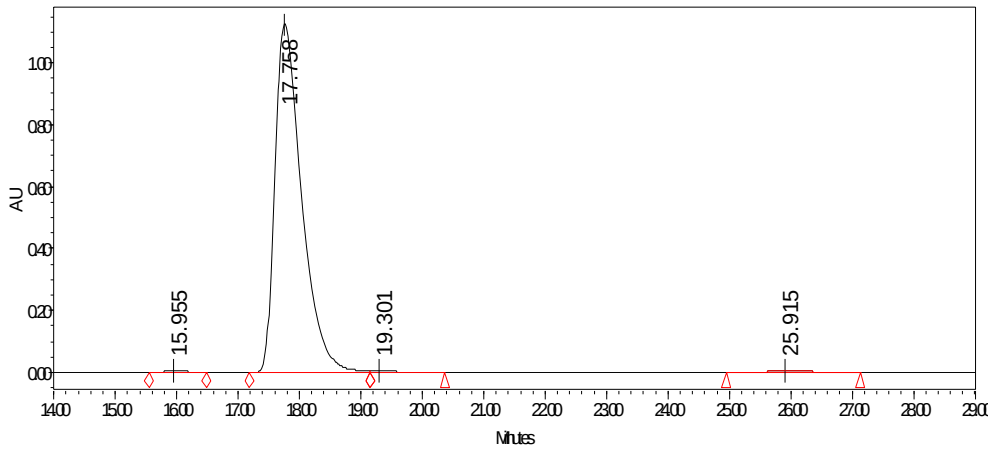
(*2S,3R*) -ethyl 3-benzoyl-2-hydroxypentanoate (*anti*-5ad)



anti-**5ad**

($C_{14}H_{18}O_4$) a slightly yellow liquid; 82% yield, *anti*/*syn* = 98:2, 97% ee (*anti*). $[\alpha]_D^{25}$ = 53.8 (*c* 0.400 in CH_2Cl_2) HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 20/80, flow rate = 0.5 mL/min, λ = 254 nm, retention time: 15.3 min，17.5 min, 18.8 min and 26.5 min; ^{1}H NMR (400 MHz, $CDCl_3$) *anti*: 1.04 (t, *J* = 7.2 Hz, 3H), 1.14 (t, *J* = 7.2 Hz, 3H), 1.84-1.89 (m, 2H), 3.70 (d, *J* = 5.2 Hz, 1H), 3.81-3.85 (m, 1H), 4.07-4.12 (m, 2H), 4.43-4.47 (m, 1H), 7.46-7.50 (m, 2H), 7.57-7.61 (m, 1H), 7.91-7.93 (m, 2H) ppm.

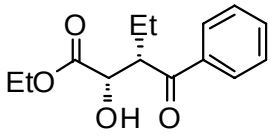




	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	15.955	161177	0.46	6309	0.55	15.550	16.483
2	17.758	34296803	98.29	1124839	98.50	17.183	19.150
3	19.301	149116	0.43	5060	0.44	19.150	20.433
4	25.915	285161	0.82	5762	0.50	25.133	27.283

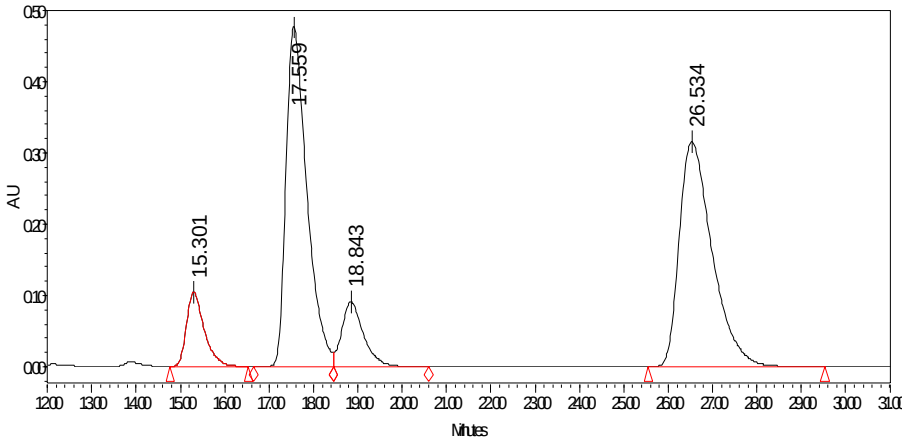
Configuration Assignment: The absolute and relative configurations were determined by the comparison of the optical rotation and ^{1}H NMR Spectra. [2]

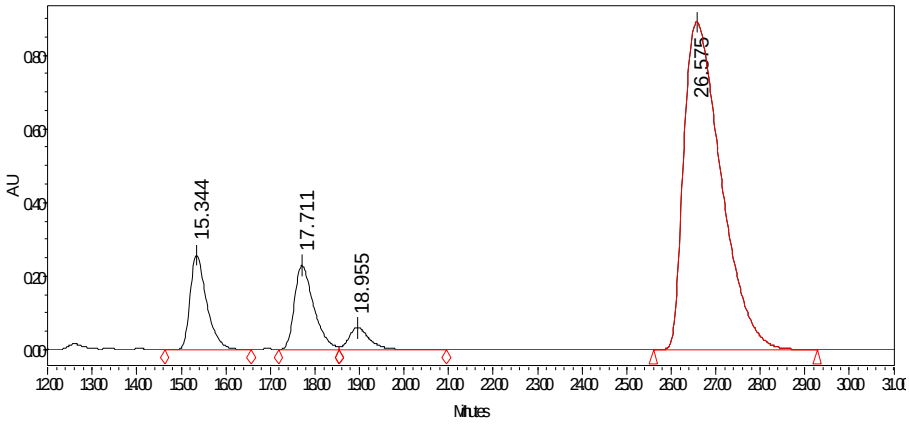
(*2S,3S*) -ethyl 3-benzoyl-2-hydroxypentanoate (*syn*-5ac)



syn-**5ad**

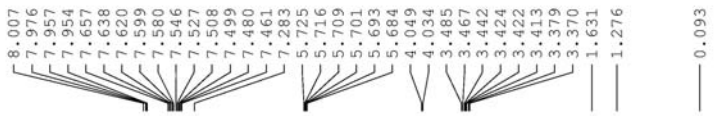
($C_{14}H_{18}O_4$) a slightly yellow liquid; 70% yield, *anti*/*syn* = 60:40, -74% ee (*anti*), 52% ee (*syn*). $[\alpha]_D^{25}$ = -16.8 (*c* 0.274 in CH_2Cl_2) HPLC DAICEL CHIRALCEL AS-H, 2-propanol/*n*-hexane = 20/80, flow rate = 0.5 mL/min, λ = 254 nm, retention time: 15.3 min，17.5 min, 18.8 min and 26.5 min; ^{1}H NMR (400 MHz, $CDCl_3$) *syn*: 0.93 (t, *J* = 7.6 Hz, 3H), 1.20 (t, *J* = 7.2 Hz, 3H), 1.84-2.0 (m, 2H), 3.14 (m, 1H), 3.82-3.85 (m, 1H), 4.19-4.23 (m, 2H), 4.50-4.51 (m, 1H), 7.46-7.50 (m, 2H), 7.57-7.61 (m, 1H), 7.91-7.93 (m, 2H) ppm.

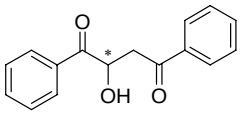




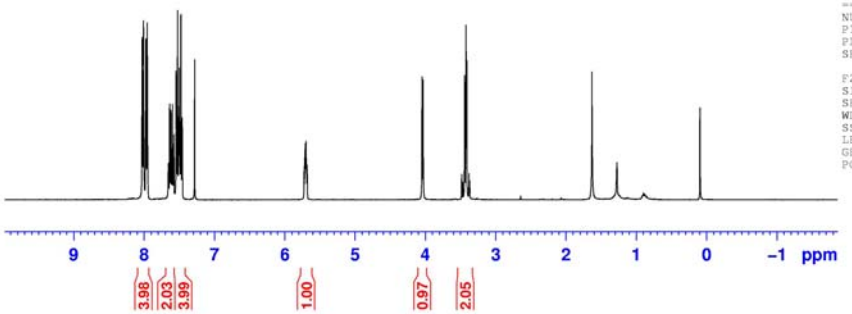
	Retention Time	Area	% Area	Height	% Height	Start Time	End Time
1	15.344	6676634	9.90	256872	17.89	14.633	16.567
2	17.711	6947408	10.31	228179	15.89	17.183	18.550
3	18.955	1966377	2.92	60354	4.20	18.550	20.950
4	26.575	51821550	76.87	890475	62.02	25.600	29.283

(F) Copies of NMR Spectra for New Compounds





5a



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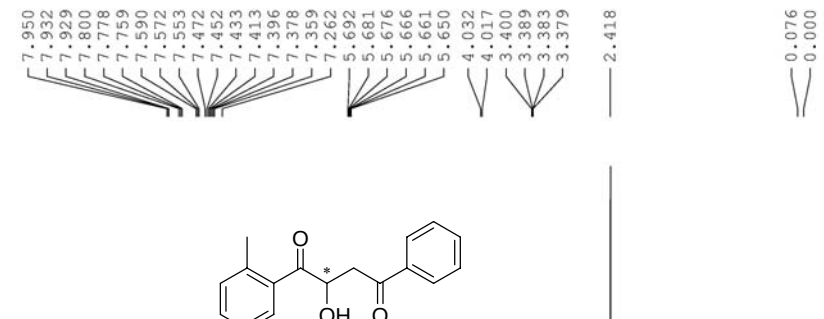
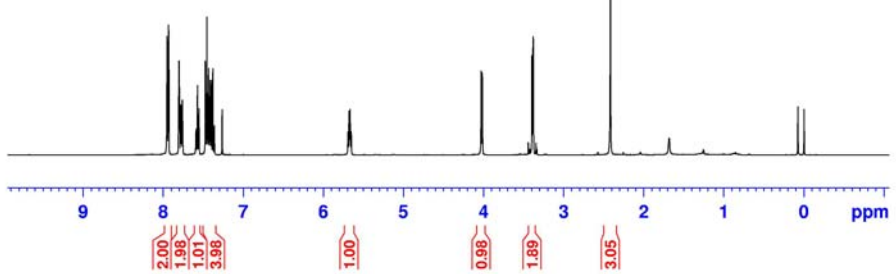


5b



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PROCNO                1

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FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     16
DW                 60.800 usec
DE                  6.50 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
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PL1                -2.00 dB
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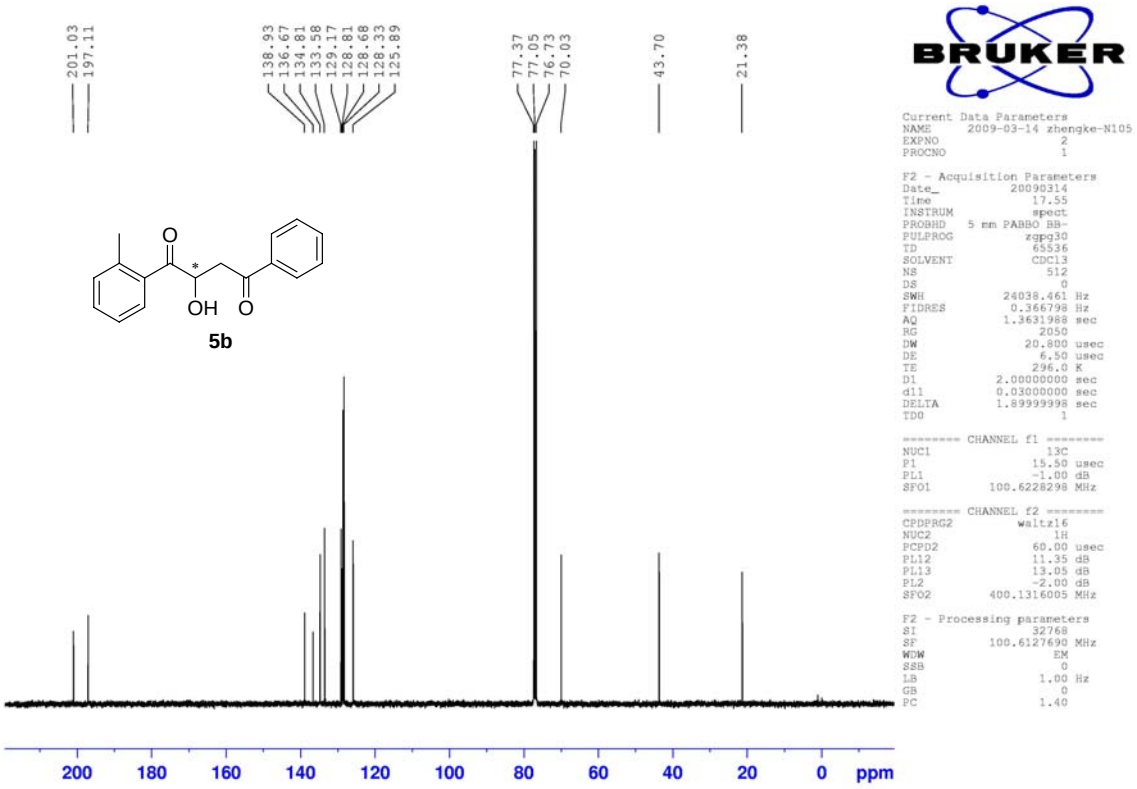
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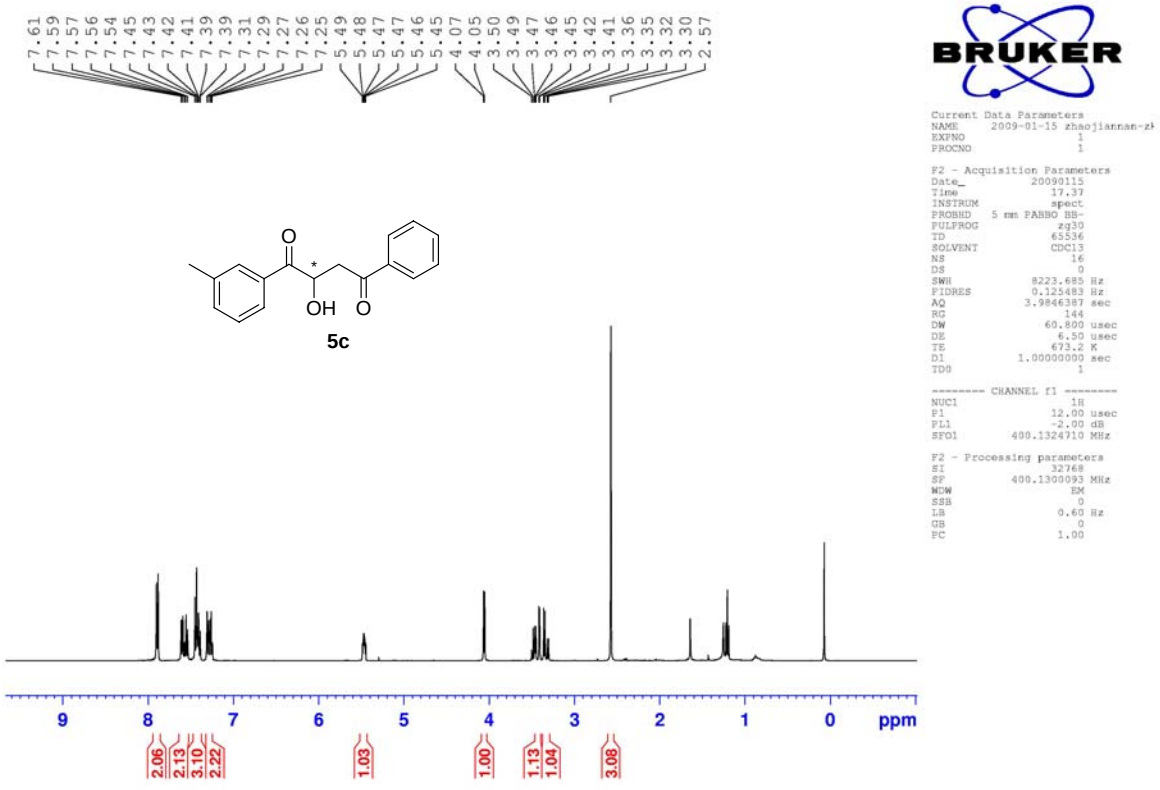
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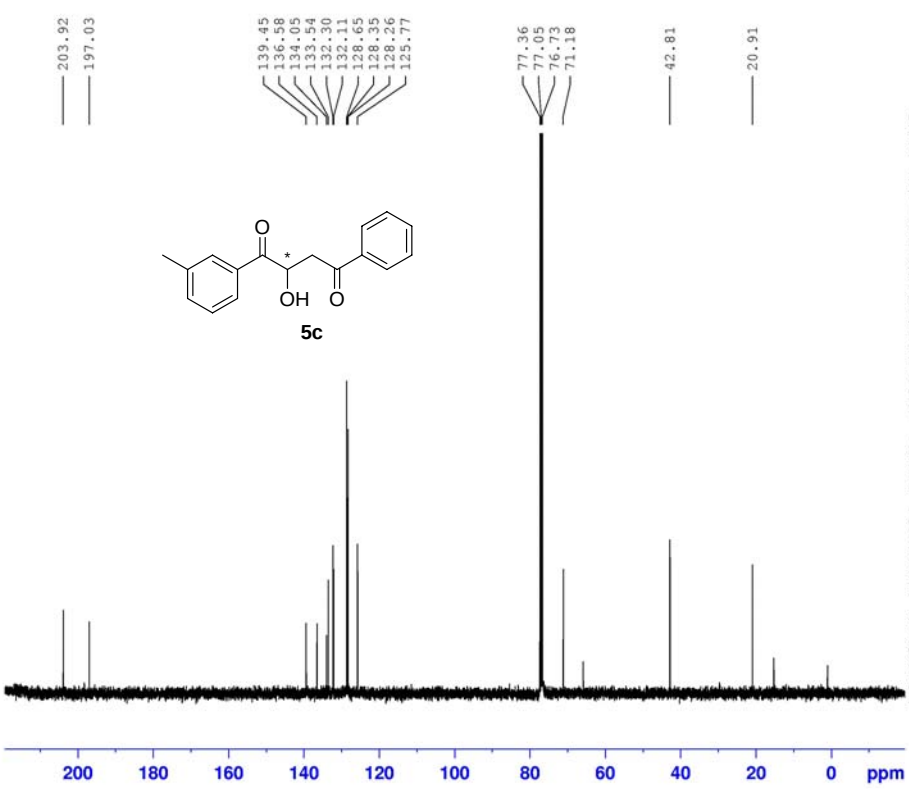

5c

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PROCNO               1

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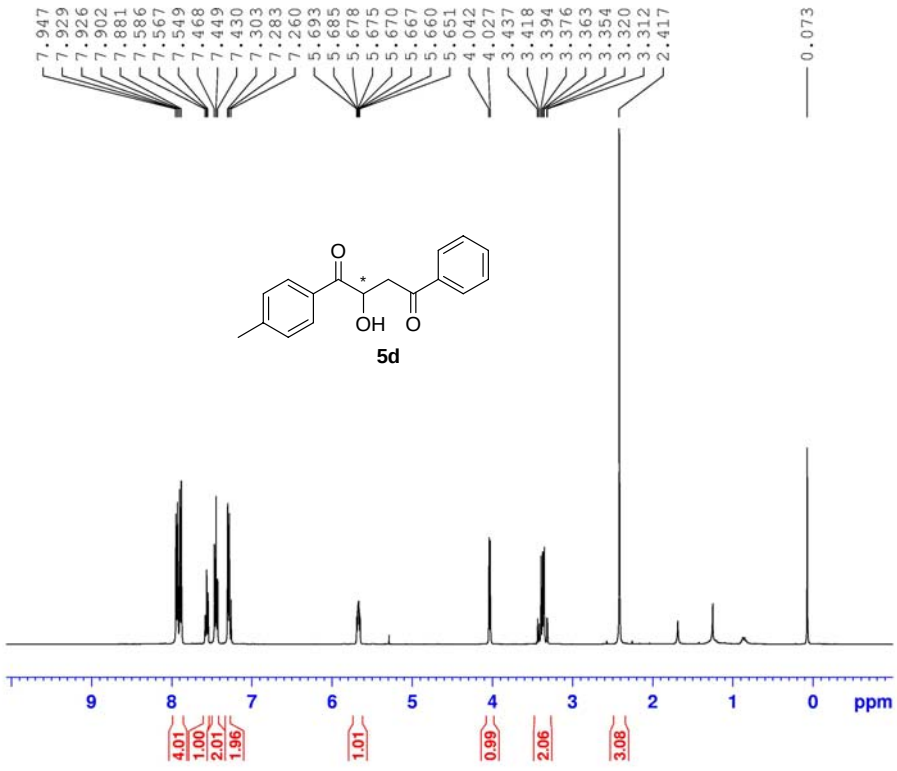
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  271
DS                    0
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE                294.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
PL1               -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             60.00 usec
PL12              11.35 dB
PL13              13.05 dB
PL2               -2.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



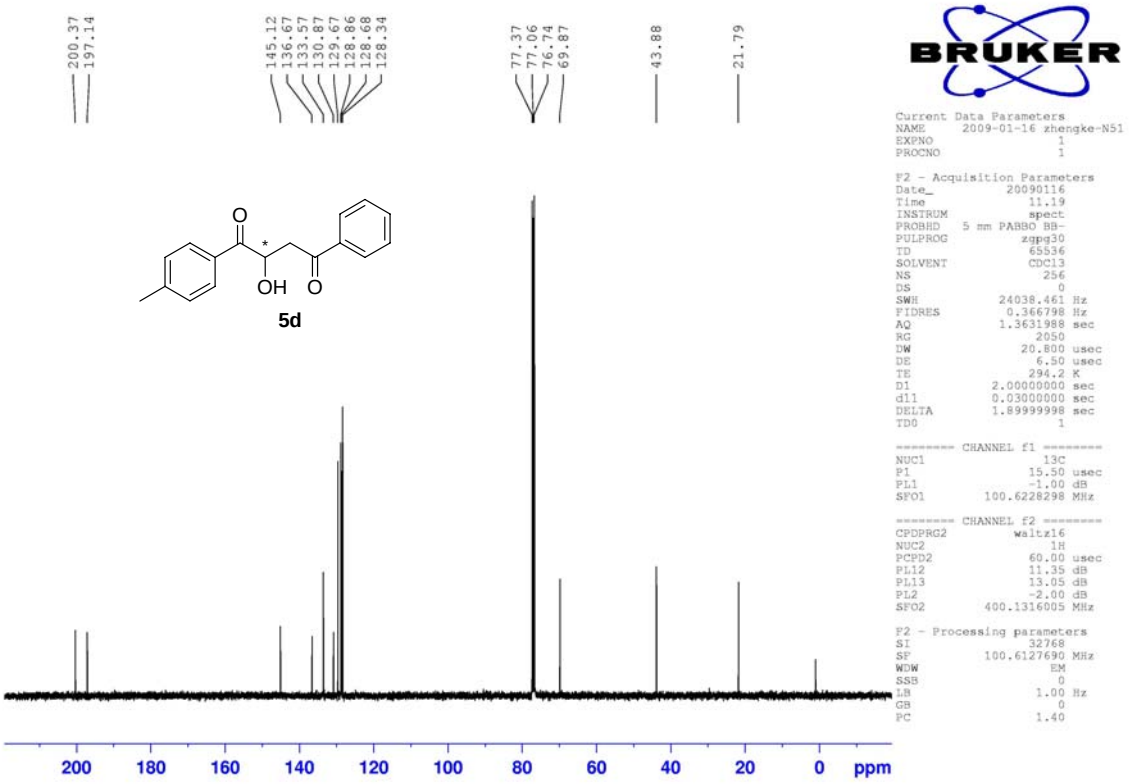
5d

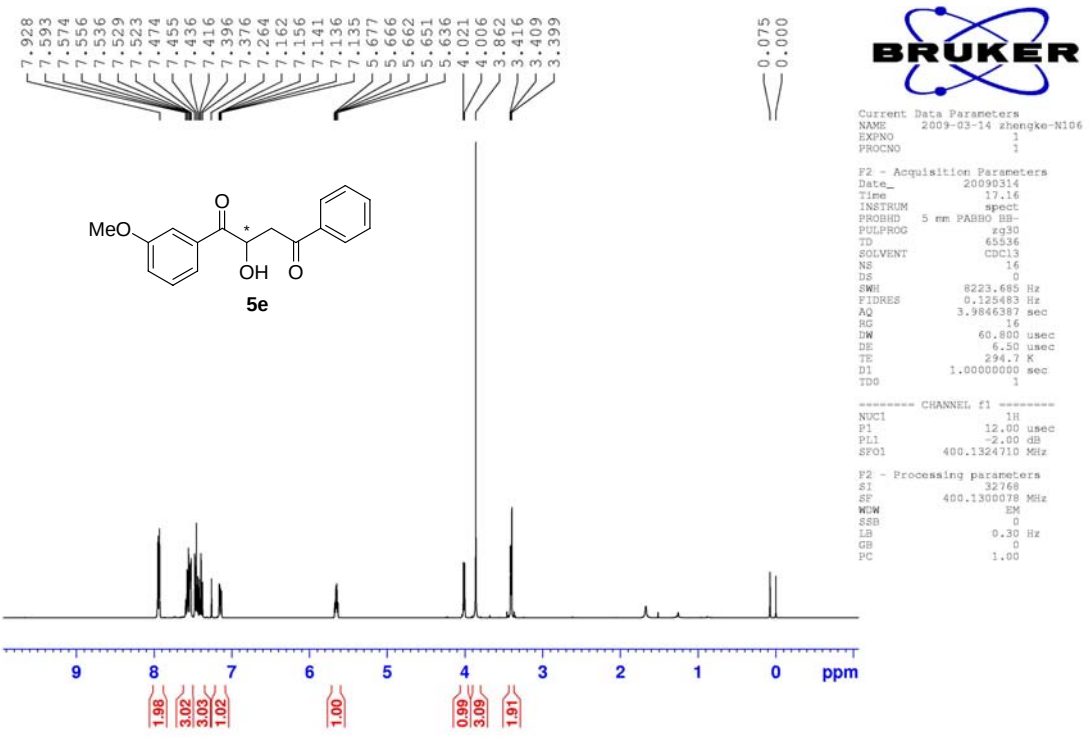
```
Current Data Parameters
NAME        2009-01-15 zhaojiannan-zk-N
EXPNO                1
PROCNO               1

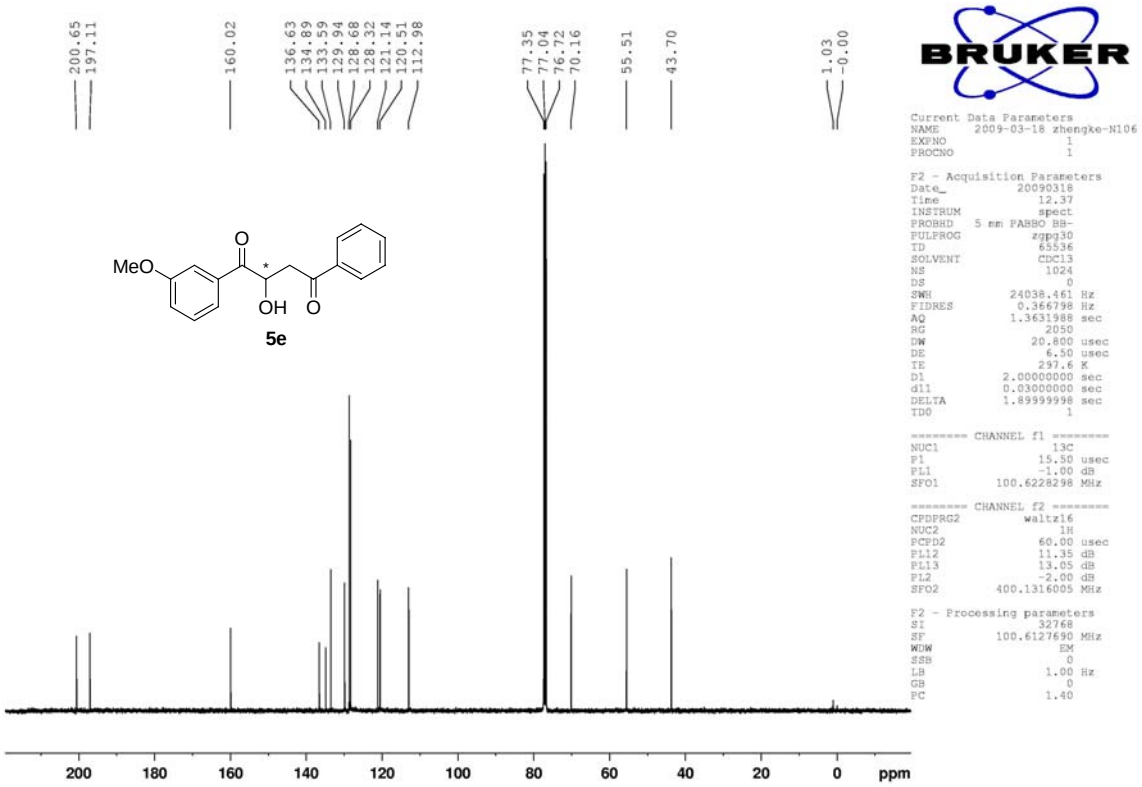
F2 - Acquisition Parameters
Date_            20090115
Time                17.31
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  144
DW               60.800 usec
DE                6.50 usec
TE                673.2 K
D1            1.00000000 sec
TD0                   1

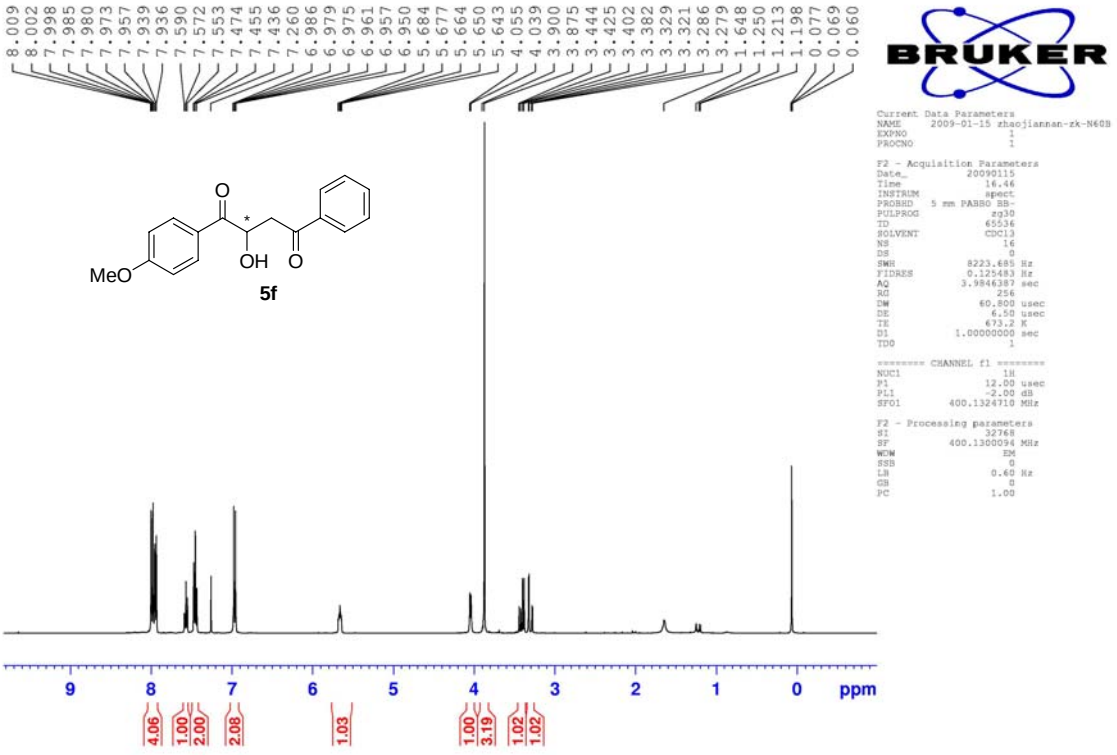
======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

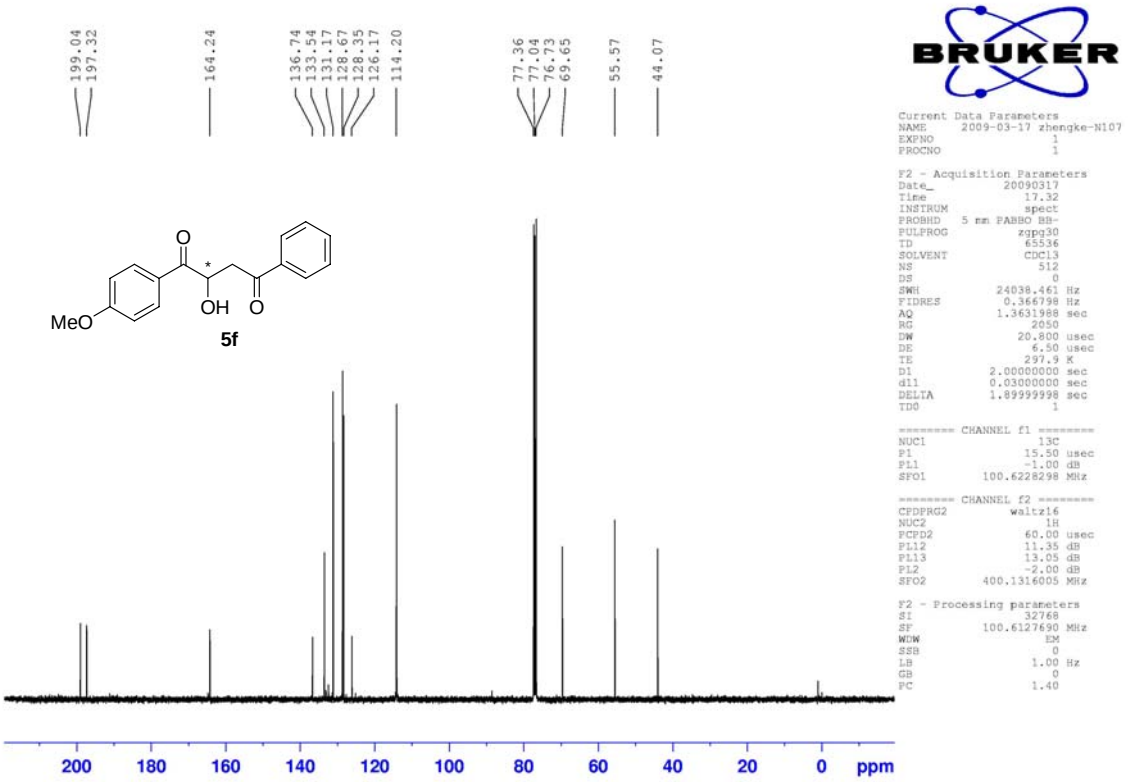
F2 - Processing parameters
SI                 32768
SF           400.1300094 MHz
WDW                   EM
SSB                    0
LB                  0.60 Hz
GB                     0
PC                  1.00
```

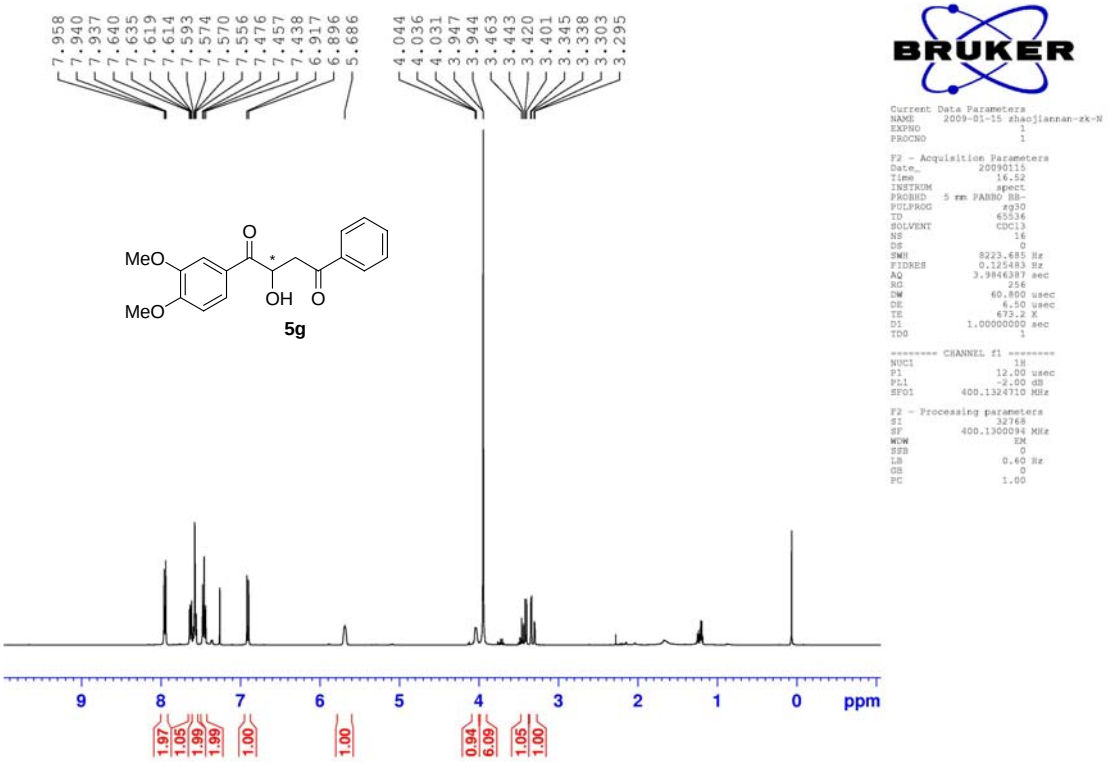

200.37
197.14
145.12
136.67
133.57
130.87
129.67
128.86
128.68
128.34
77.37
77.06
76.74
69.87
43.88
21.79
O
OH
O
5d
200
180
160
140
120
100
80
60
40
20
0
ppm

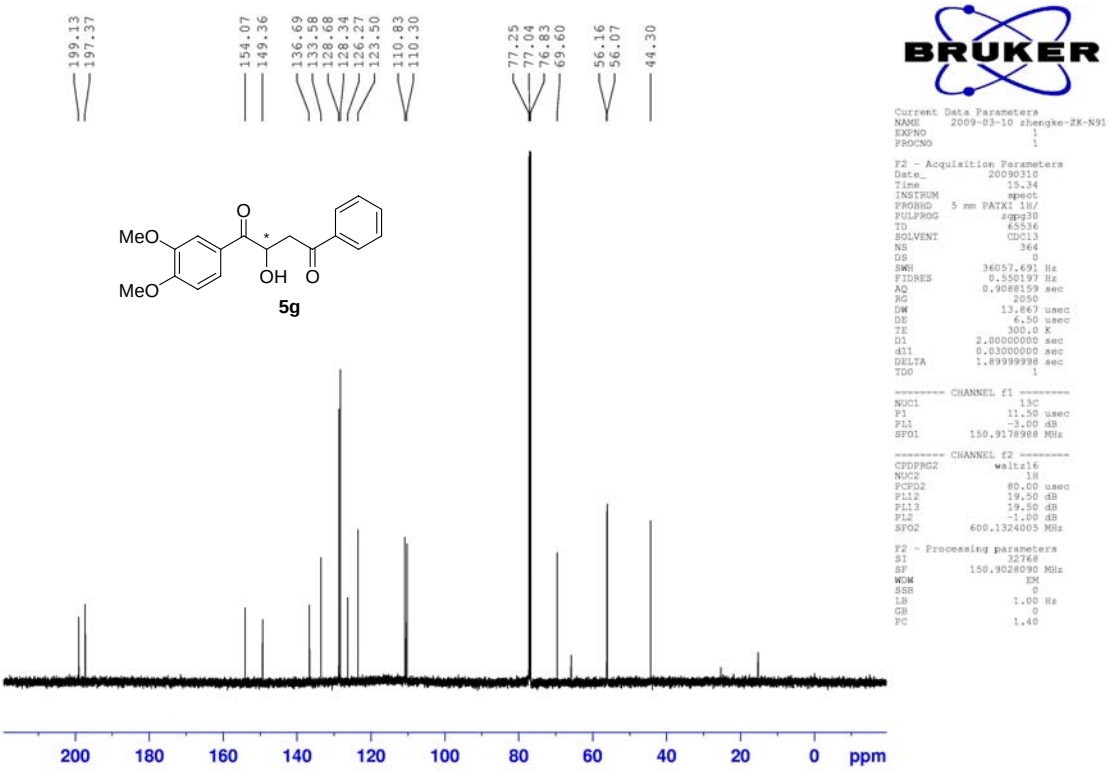

7.928
7.593
7.574
7.556
7.536
7.529
7.523
7.474
7.455
7.436
7.416
7.396
7.376
7.264
7.162
7.156
7.141
7.136
7.135
5.677
5.666
5.662
5.651
5.636
4.021
4.006
3.862
3.416
3.409
3.399
0.075
0.000
MeO
O
OH
O
5e
9
8
7
6
5
4
3
2
1
0
ppm
1.98
3.02
3.03
1.02
1.00
0.99
3.09
1.91

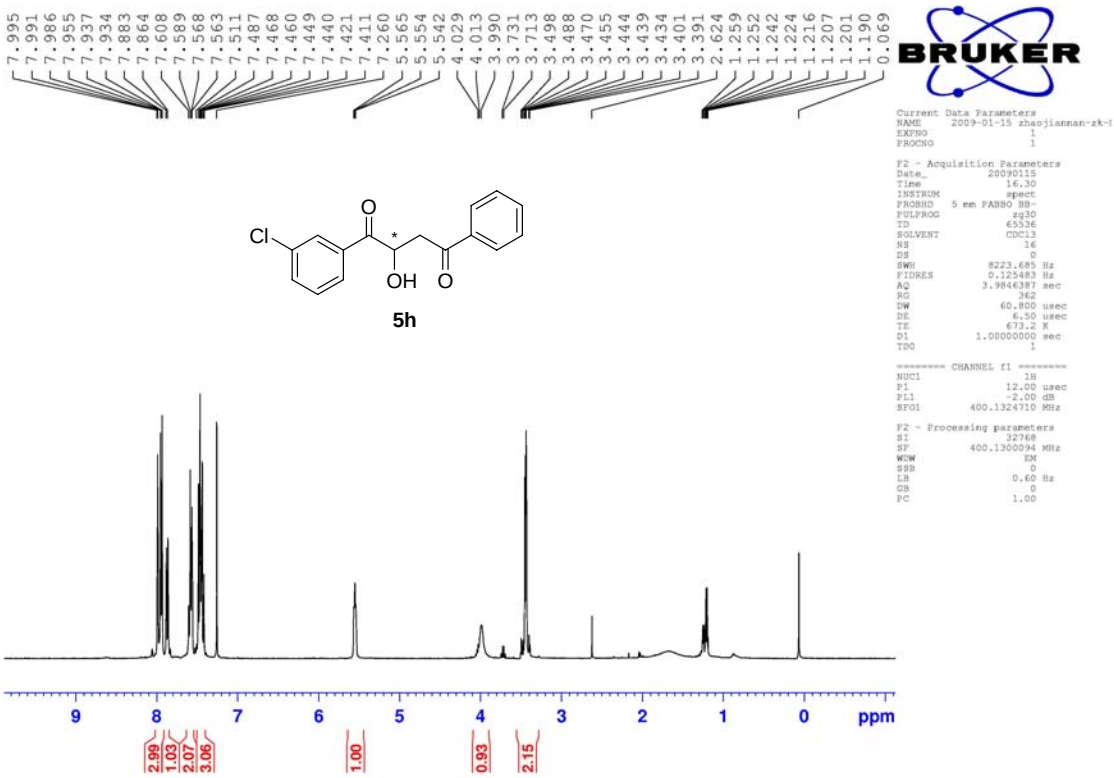

200.65
197.11
160.02
136.63
134.89
133.59
129.94
128.68
128.32
121.14
120.51
112.98
77.35
77.04
76.72
70.16
55.51
43.70
1.03
0.00
MeO
OH
O
5e
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME 2009-03-18 zhengke-N106
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090318
Time 12.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

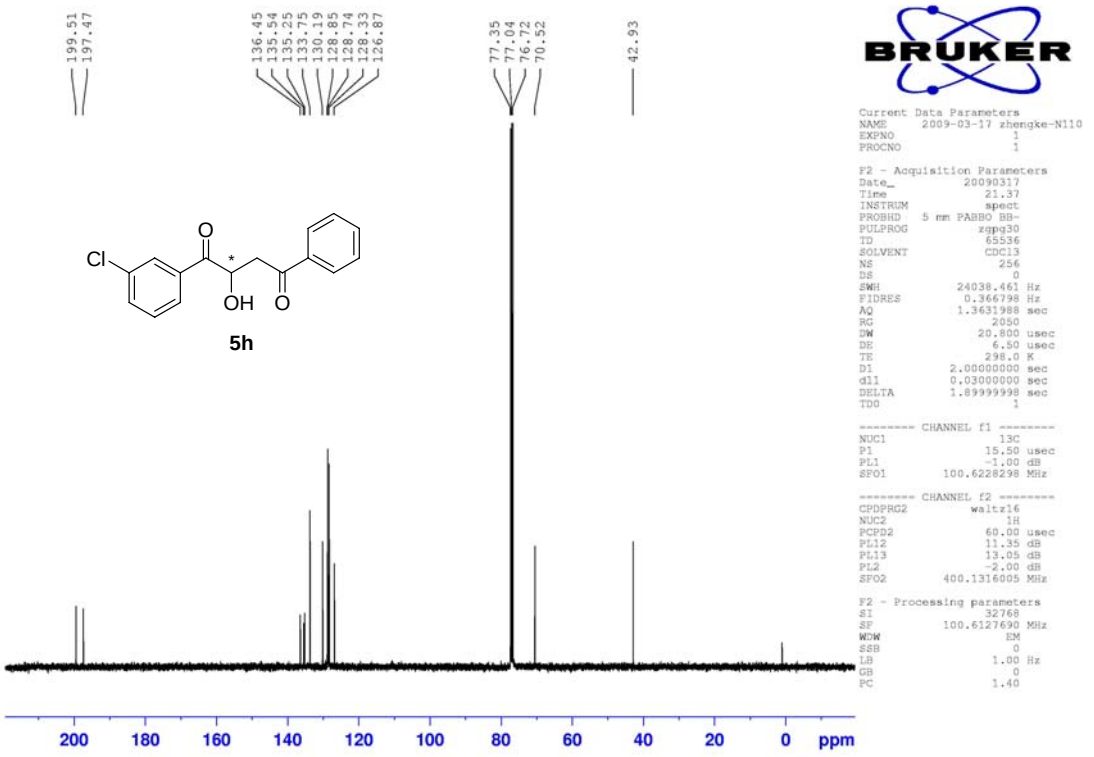

8.009
8.002
7.998
7.985
7.980
7.973
7.957
7.939
7.936
7.590
7.572
7.553
7.474
7.455
7.436
7.260
6.986
6.979
6.975
6.961
6.957
6.950
5.684
5.677
5.664
5.650
5.643
4.055
4.039
3.900
3.875
3.444
3.425
3.402
3.382
3.329
3.321
3.286
3.279
1.648
1.250
1.213
1.198
0.077
0.069
0.060
MeO
OH
O
5f
9
8
7
6
5
4
3
2
1
0
ppm
4.06
1.00
2.00
2.08
1.03
1.00
3.19
1.02
1.02
BRUKER
Current Data Parameters
NAME 2009-01-15 zhaojiannan-zk-N608
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 16.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

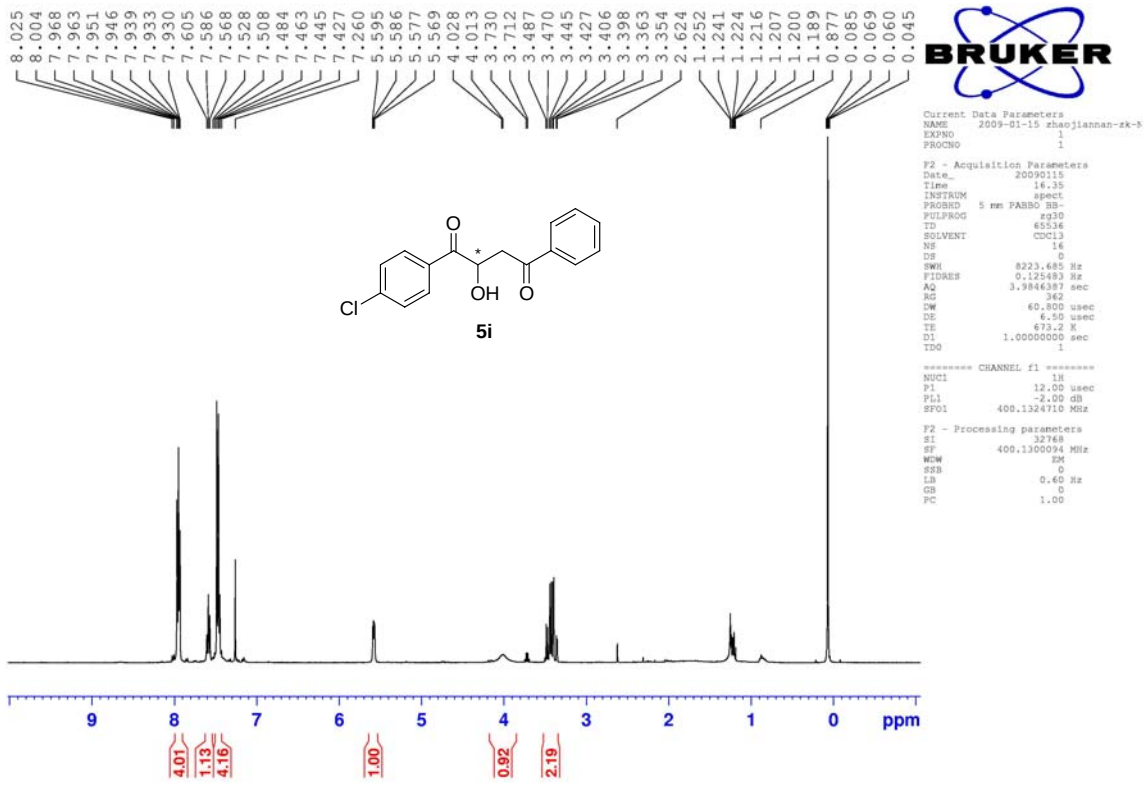

199.04
197.32
164.24
136.74
133.54
131.17
128.67
128.35
126.17
114.20
77.36
77.04
76.73
69.65
55.57
44.07
MeO
OH
O
5f
200
180
160
140
120
100
80
60
40
20
0
ppm

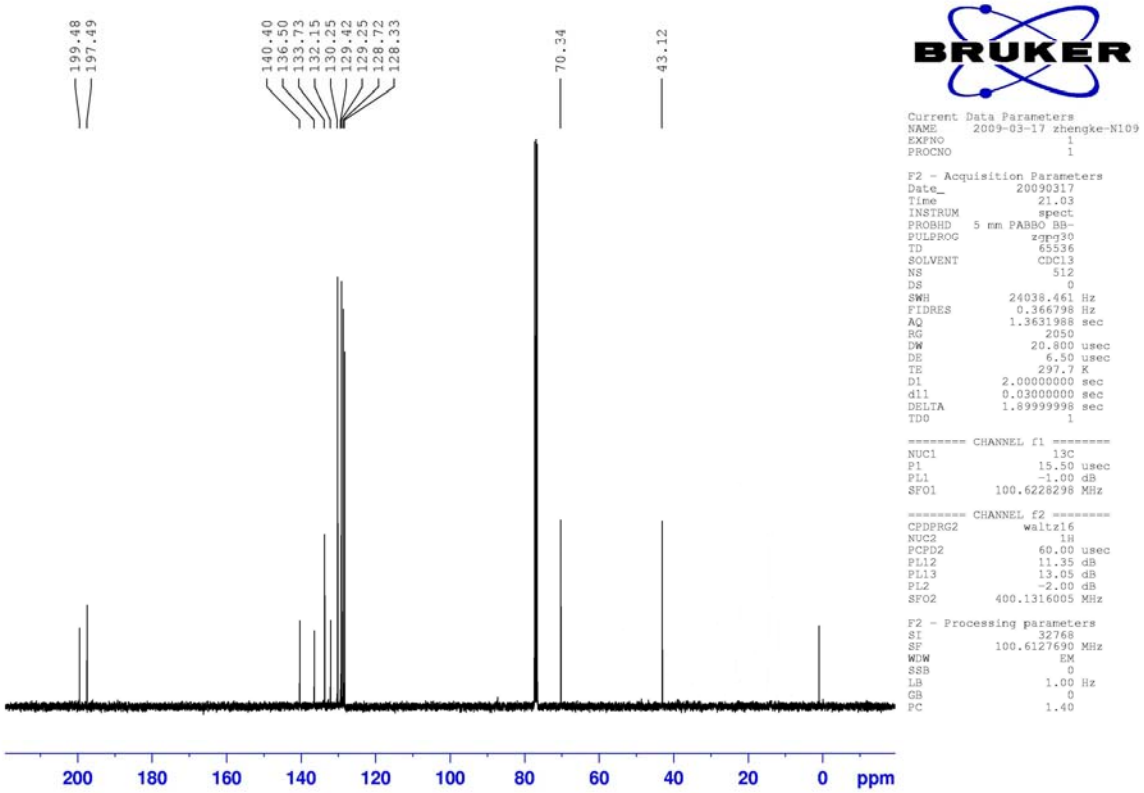

7.958
7.940
7.937
7.640
7.635
7.619
7.614
7.593
7.574
7.570
7.556
7.476
7.457
7.438
6.917
6.896
5.686
4.044
4.036
4.031
3.947
3.944
3.463
3.443
3.420
3.401
3.345
3.338
3.303
3.295
MeO
MeO
OH
O
5g
9
8
7
6
5
4
3
2
1
0
ppm
1.97
1.05
1.99
1.99
1.00
1.00
0.94
6.09
1.05
1.00

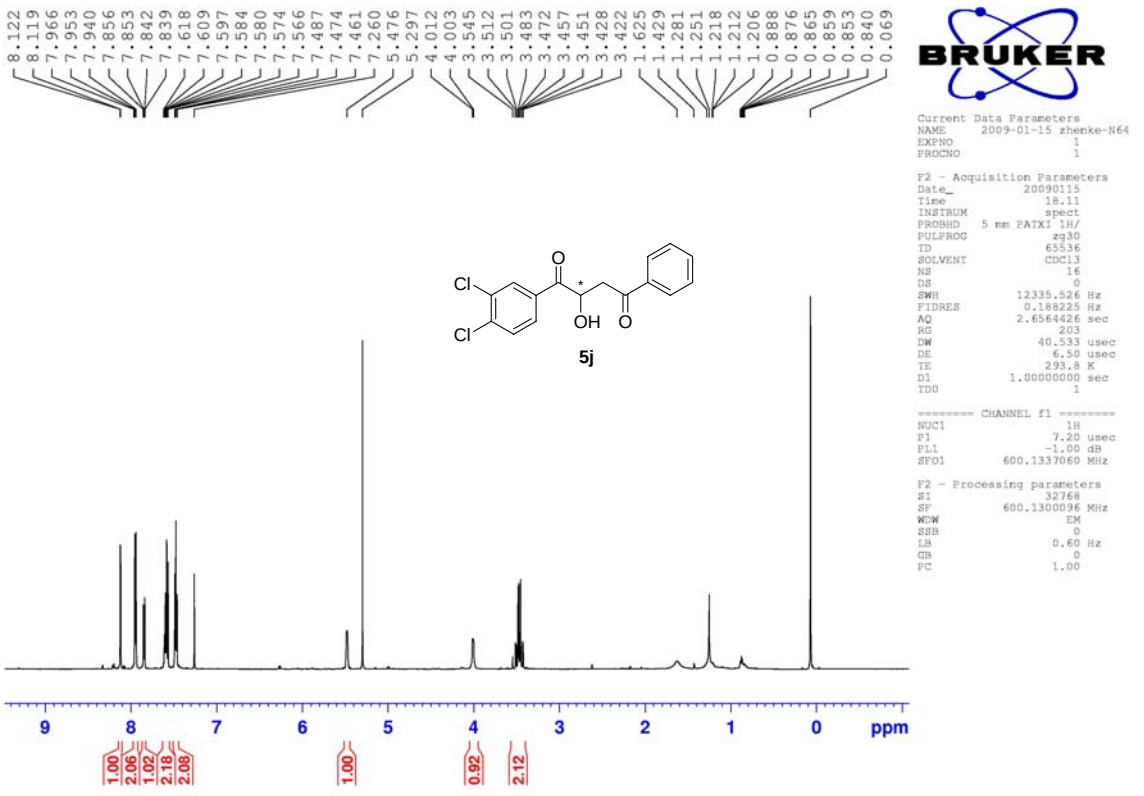

199.13
197.37
154.07
149.36
136.69
133.58
128.68
128.34
126.27
123.50
110.83
110.30
77.25
77.04
76.83
69.60
56.16
56.07
44.30
MeO
MeO
OH
O
5g
200
180
160
140
120
100
80
60
40
20
0
ppm

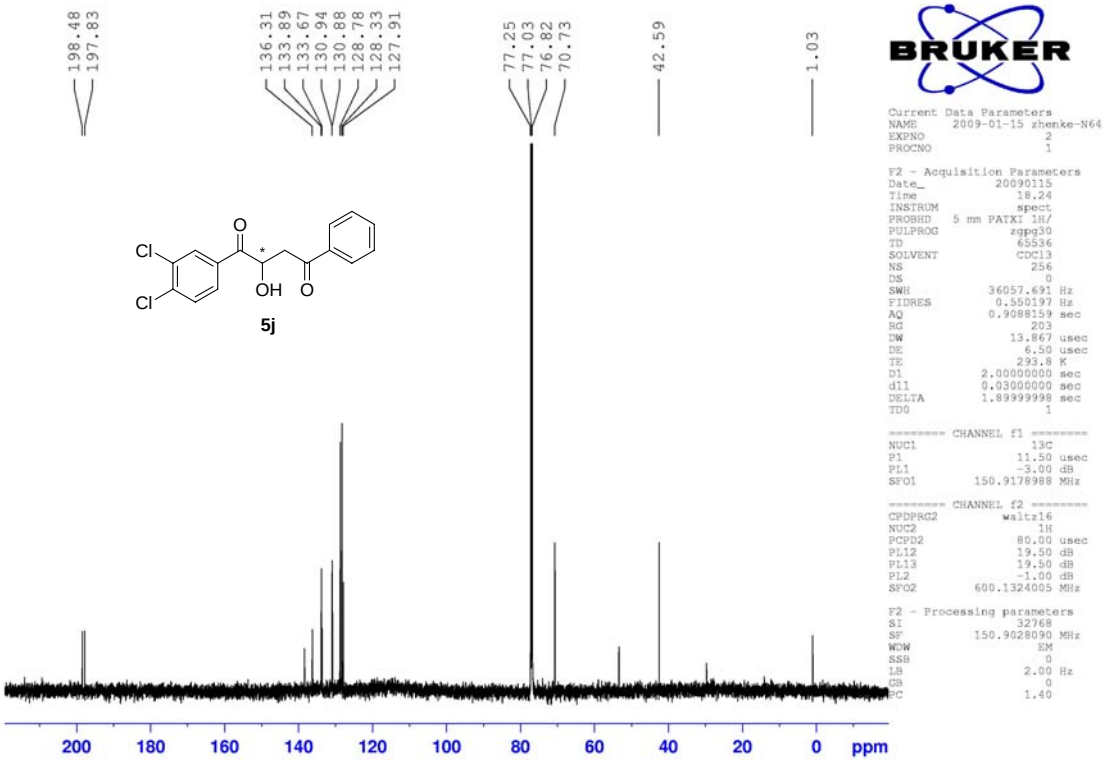

7.995
7.991
7.986
7.955
7.937
7.934
7.883
7.864
7.608
7.589
7.568
7.563
7.511
7.487
7.468
7.460
7.449
7.440
7.421
7.411
7.260
5.565
5.554
5.542
4.029
4.013
3.990
3.731
3.713
3.498
3.488
3.470
3.455
3.444
3.439
3.434
3.401
3.391
2.624
1.259
1.252
1.242
1.224
1.216
1.207
1.201
1.190
0.069
Cl
OH
O
5h
9
8
7
6
5
4
3
2
1
0
ppm
2.99
1.03
2.07
3.06
1.00
0.93
2.15


199.51
197.47
136.45
135.54
135.25
133.75
130.19
128.85
128.74
128.33
126.87
77.35
77.04
76.72
70.52
42.93
BRUKER
Current Data Parameters
NAME 2009-03-17 zhengke-N110
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090317
Time 21.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
O
OH
O
*
5h
200
180
160
140
120
100
80
60
40
20
0
ppm


8.025
8.004
7.968
7.963
7.951
7.946
7.939
7.933
7.930
7.605
7.586
7.568
7.528
7.508
7.484
7.463
7.445
7.427
7.260
5.595
5.586
5.577
5.569
4.028
4.013
3.730
3.712
3.487
3.470
3.445
3.427
3.406
3.398
3.363
3.354
2.624
1.252
1.241
1.224
1.216
1.207
1.200
1.189
0.877
0.085
0.069
0.060
0.045
BRUKER
Current Data Parameters
NAME 2009-01-15 zhaojiannan-zk-5
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 16.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 362
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
Cl
O
OH
O
*
5i
9
8
7
6
5
4
3
2
1
0
ppm
4.01
1.13
4.16
1.00
0.92
2.19


199.48
197.49
140.40
136.50
133.73
132.15
130.25
129.42
129.25
128.72
128.33
70.34
43.12
200
180
160
140
120
100
80
60
40
20
0
ppm


8.122
8.119
7.966
7.953
7.940
7.856
7.853
7.842
7.839
7.618
7.609
7.597
7.584
7.580
7.574
7.566
7.487
7.474
7.461
7.260
5.476
5.297
4.012
4.003
3.545
3.512
3.501
3.483
3.472
3.457
3.451
3.428
3.422
1.625
1.429
1.281
1.251
1.218
1.212
1.206
0.888
0.876
0.865
0.859
0.853
0.840
0.069
Cl
Cl
O
OH
O
5j
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.06
1.02
2.18
2.08
1.00
0.92
2.12



5j

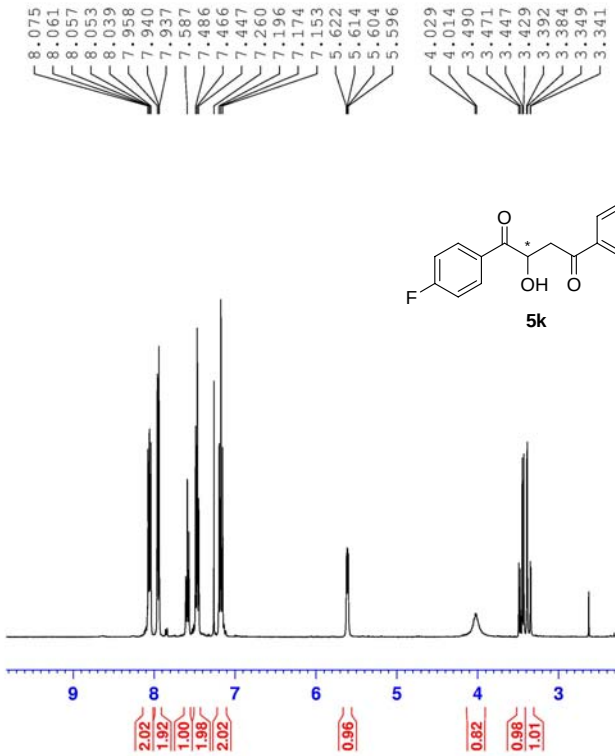

```
Current Data Parameters
NAME      2009-01-15 zhenke-N64
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20090115
Time              18.24
INSTRUM           spect
PROBHD   5 mm PATXI 1H/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 256
DS                   0
SWH          36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG                 203
DW              13.867 usec
DE                6.50 usec
TE              293.8 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                11.50 usec
PL1               -3.00 dB
SFO1        150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL12              19.50 dB
PL13              19.50 dB
PL2               -1.00 dB
SFO2        600.1324005 MHz

F2 - Processing parameters
SI                 32768
SF          150.9028090 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
```



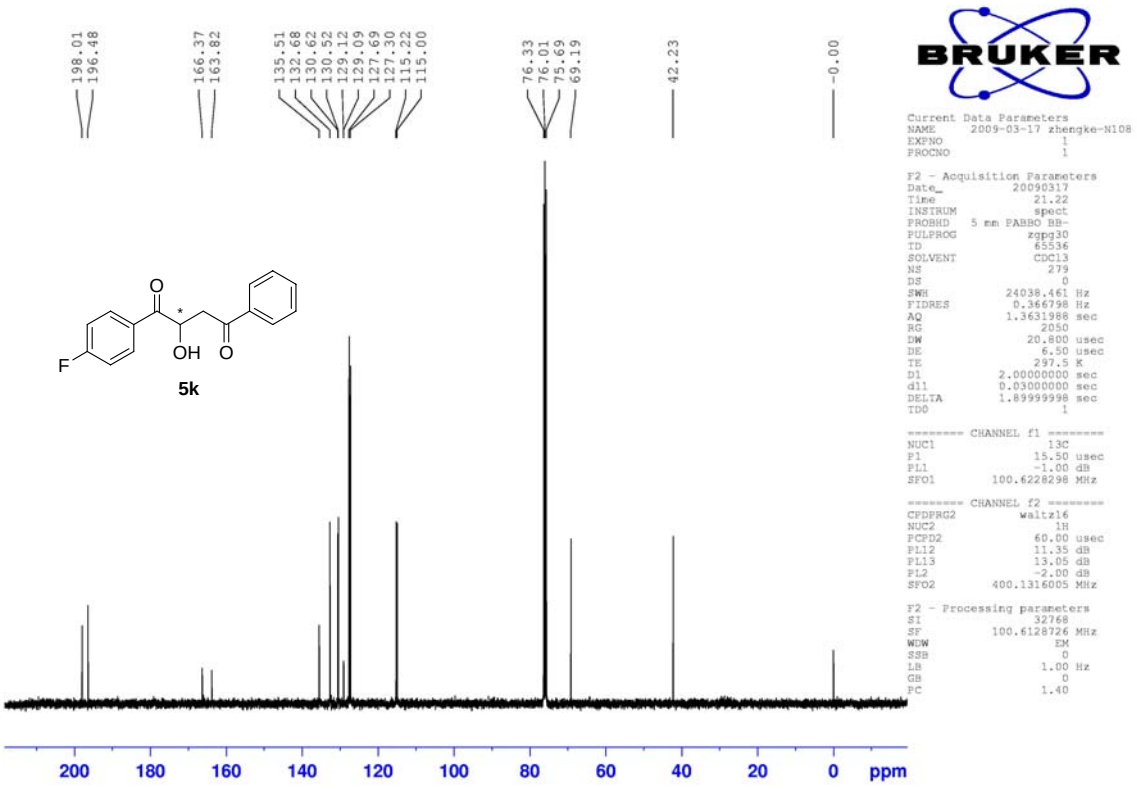
5k

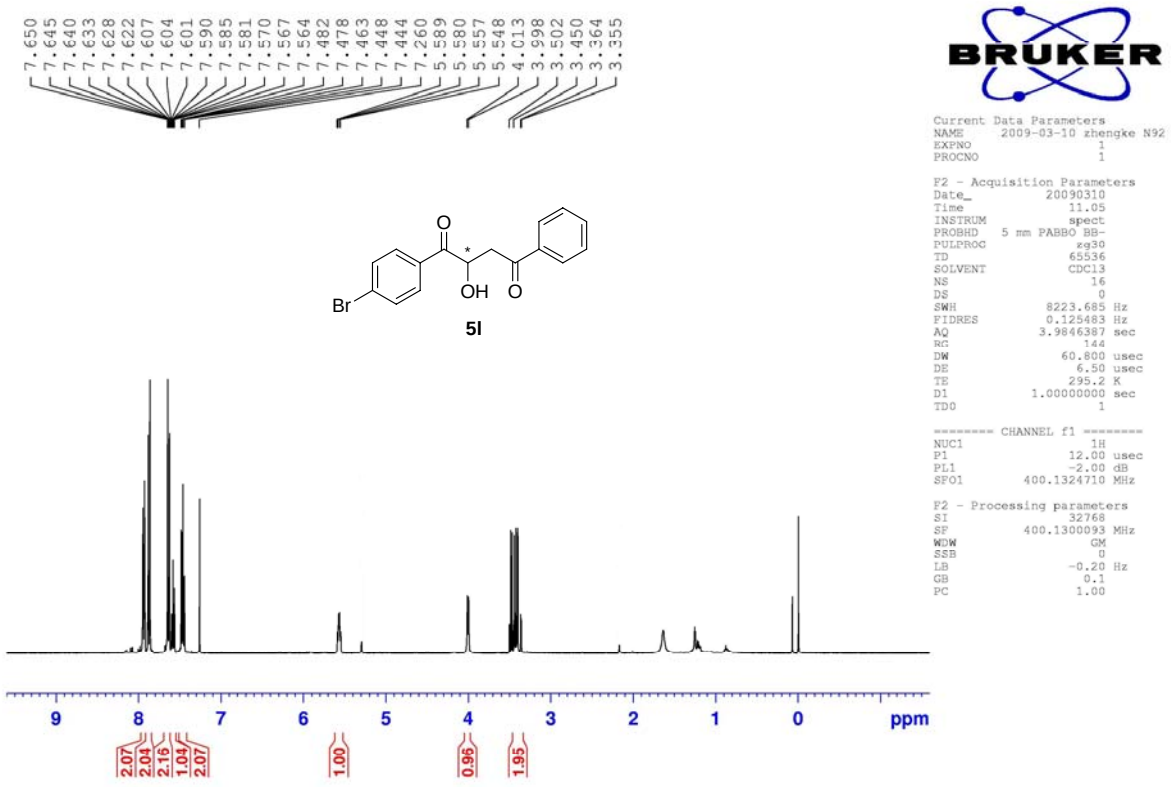

```
Current Data Parameters
NAME      2009-01-15 zhaojiannan-zk-N58B
EXPNO                1
PROCNO               1

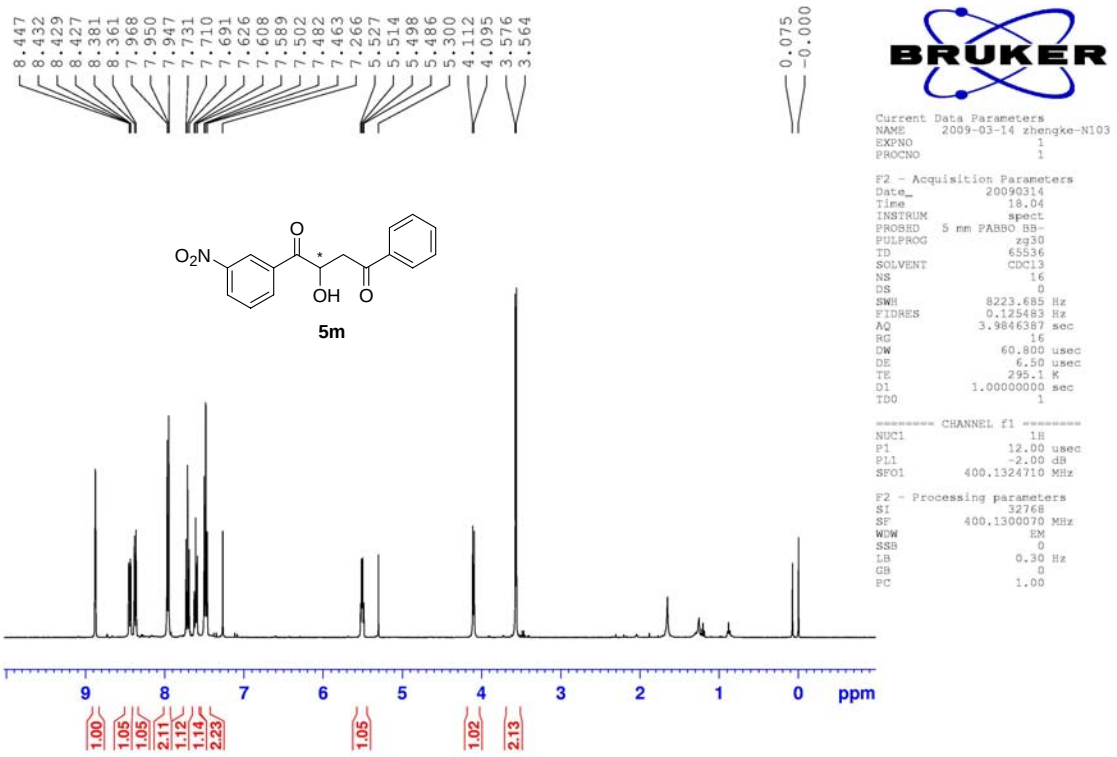
F2 - Acquisition Parameters
Date_          20090115
Time              16.40
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                 362
DW              60.800 usec
DE                6.50 usec
TE              673.2 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300094 MHz
WDW                   EM
SSB                    0
LB                  0.40 Hz
GB                     0
PC                  1.00
```


198.01
196.48
166.37
163.82
135.51
132.68
130.62
130.52
129.12
129.09
127.69
127.30
115.22
115.00
76.33
76.01
75.69
69.19
42.23
-0.00
BRUKER
Current Data Parameters
NAME 2009-03-17 zhengke-N108
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090317
Time 21.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 279
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128726 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OH
O
F
5k
200
180
160
140
120
100
80
60
40
20
0
ppm


7.650
7.645
7.640
7.633
7.628
7.622
7.607
7.604
7.601
7.590
7.585
7.581
7.570
7.567
7.564
7.482
7.478
7.463
7.448
7.444
7.260
5.589
5.580
5.557
5.548
4.013
3.998
3.502
3.450
3.364
3.355
BRUKER
Current Data Parameters
NAME 2009-03-10 zhengke N92
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090310
Time 11.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300093 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
O
OH
O
Br
5l
9
8
7
6
5
4
3
2
1
0
ppm
2.07
2.04
2.16
1.04
2.07
1.00
0.96
1.95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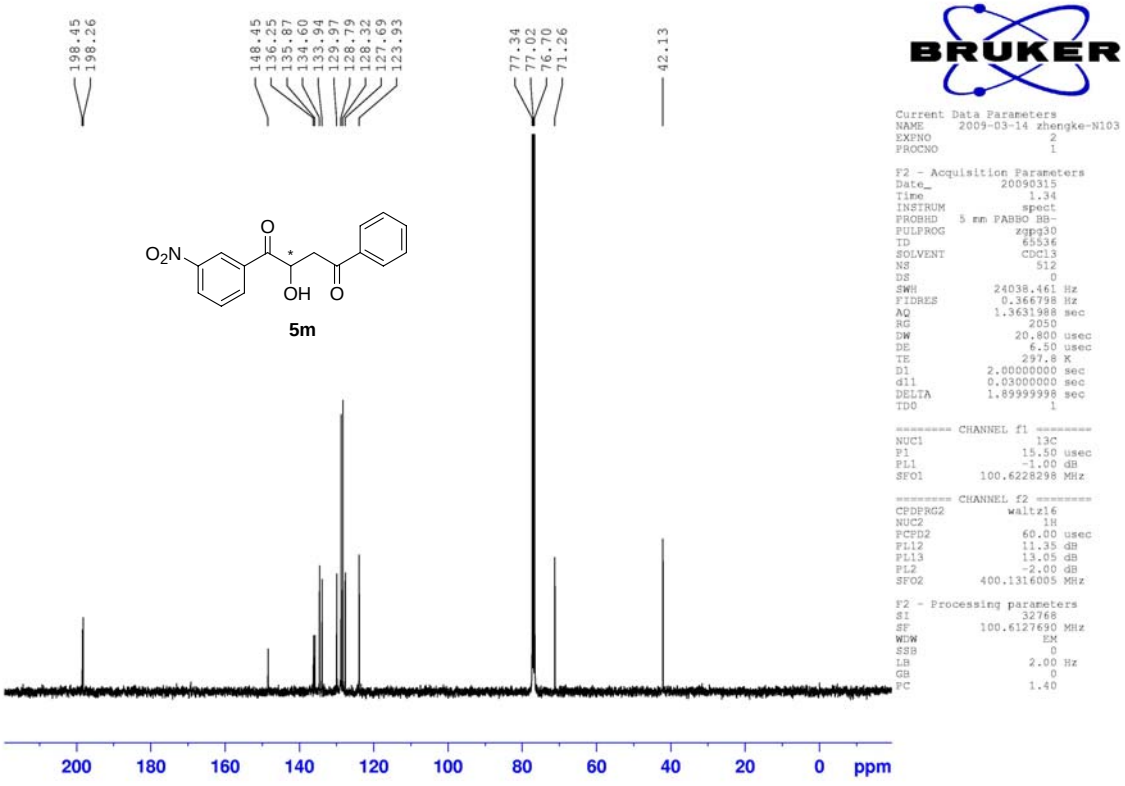
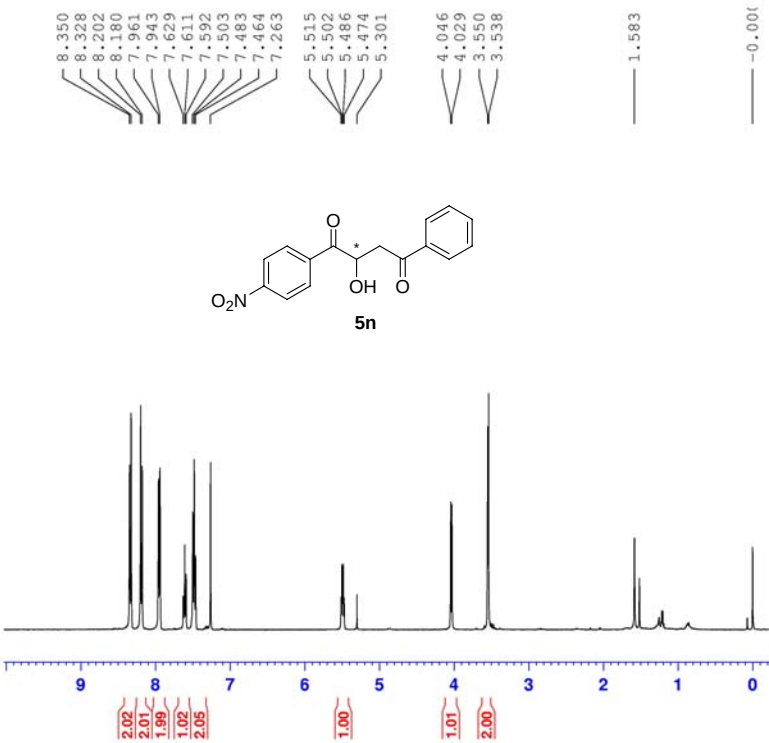
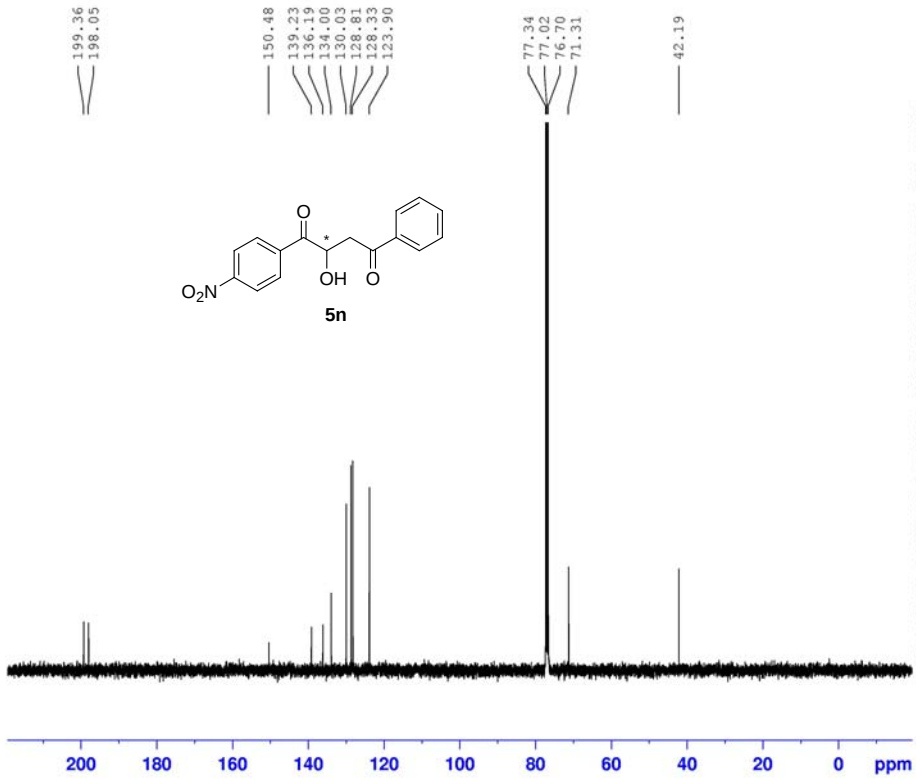
8.432
8.429
8.427
8.381
8.361
7.968
7.950
7.947
7.731
7.710
7.691
7.626
7.608
7.589
7.502
7.482
7.463
7.266
5.527
5.514
5.498
5.486
5.300
4.112
4.095
3.576
3.564
0.075
-0.000
O2N
OH
O
5m
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.05
1.05
2.11
1.12
1.14
2.23
1.05
1.02
2.13
BRUKER
Current Data Parameters
NAME 2009-03-14 zhengke-N103
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090314
Time 18.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

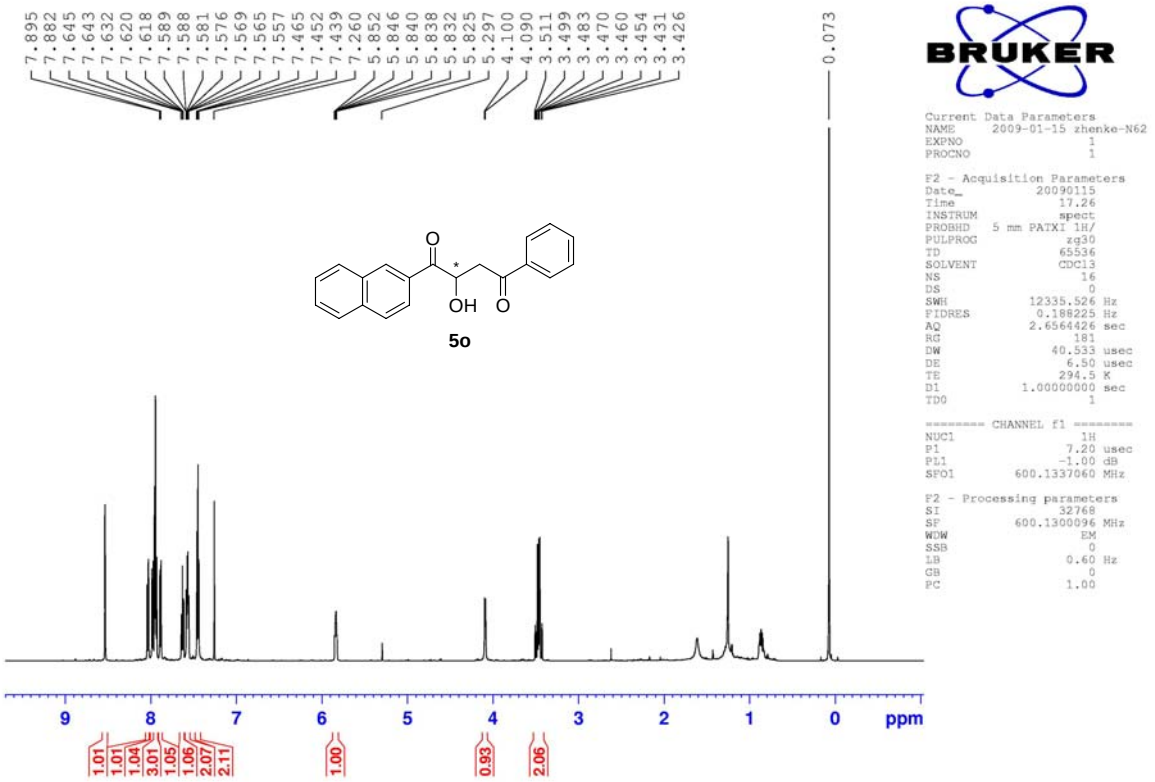

198.45
198.26
148.45
136.25
135.87
134.60
133.94
129.97
128.79
128.32
127.69
123.93
77.34
77.02
76.70
71.26
42.13
O2N
OH
O
5m
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME 2009-03-14 zhengke-N103
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090315
Time 1.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

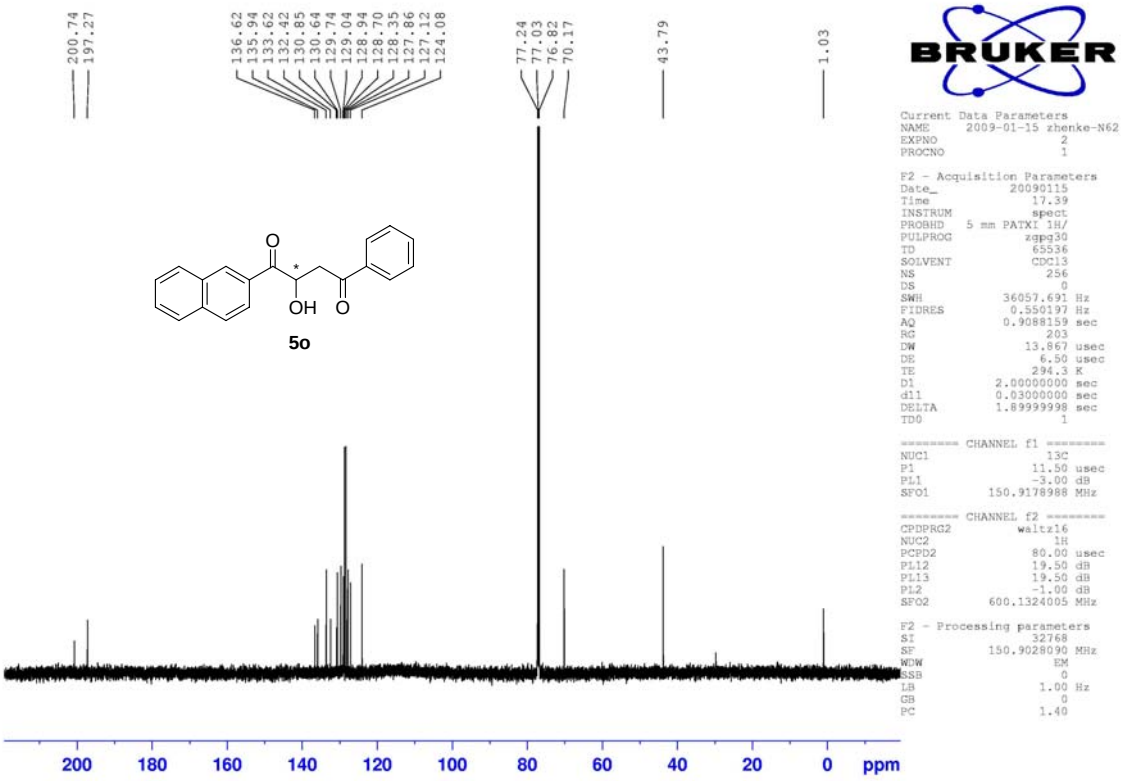

8.350
8.328
8.202
8.180
7.961
7.943
7.629
7.611
7.592
7.503
7.483
7.464
7.263
5.515
5.502
5.486
5.474
5.301
4.046
4.029
3.550
3.538
1.583
O
OH
O
O_2N
5n
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.01
1.99
1.02
2.05
1.00
1.01
2.00

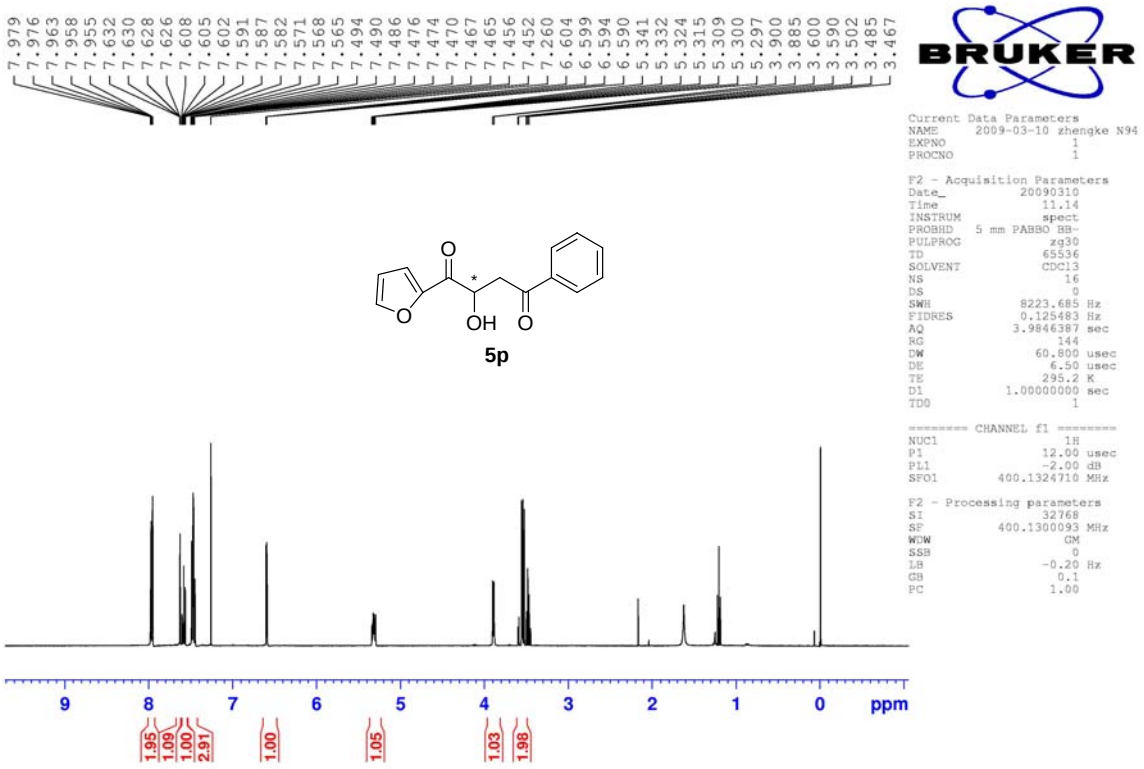

BRUKER
Current Data Parameters
NAME 2009-03-14 zhengke-N104
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090314
Time 17.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

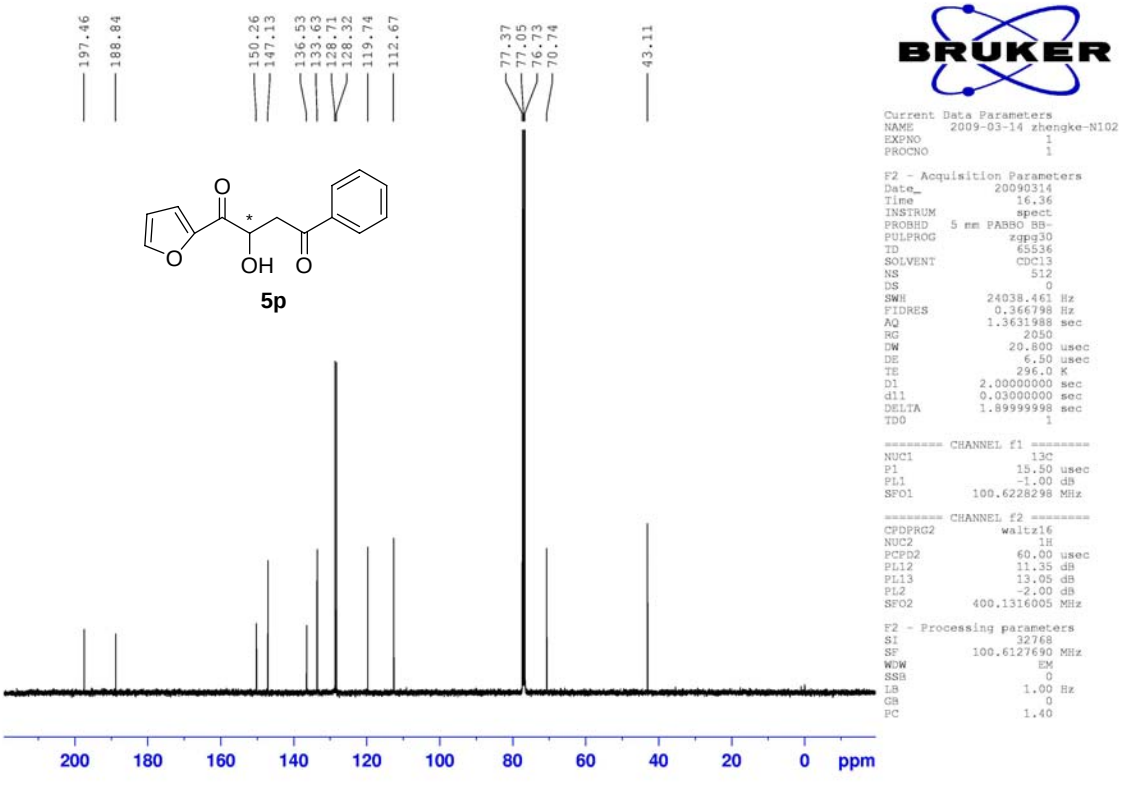

199.36
198.05
150.48
139.23
136.19
134.00
130.03
128.81
128.33
123.90
77.34
77.02
76.70
71.31
42.19
O
OH
O
O_2N
5n
200
180
160
140
120
100
80
60
40
20
0
ppm

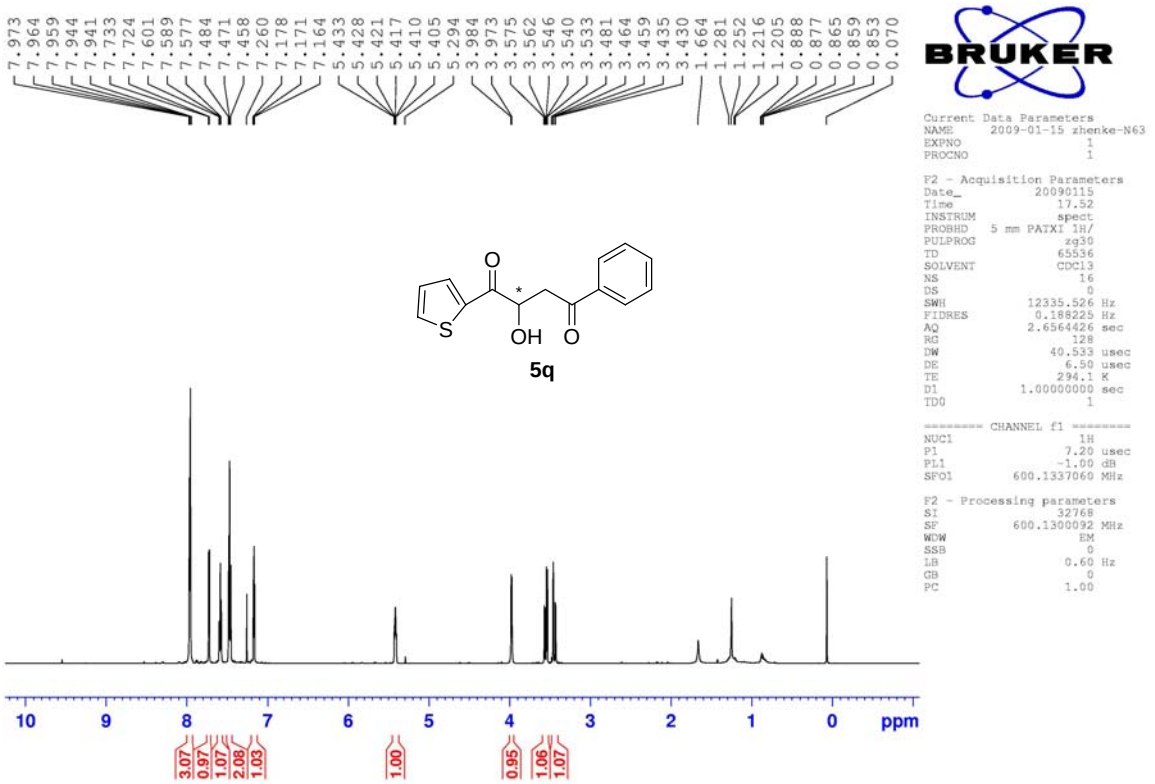

BRUKER
Current Data Parameters
NAME 2009-03-18 zhengke-N104
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090318
Time 11.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

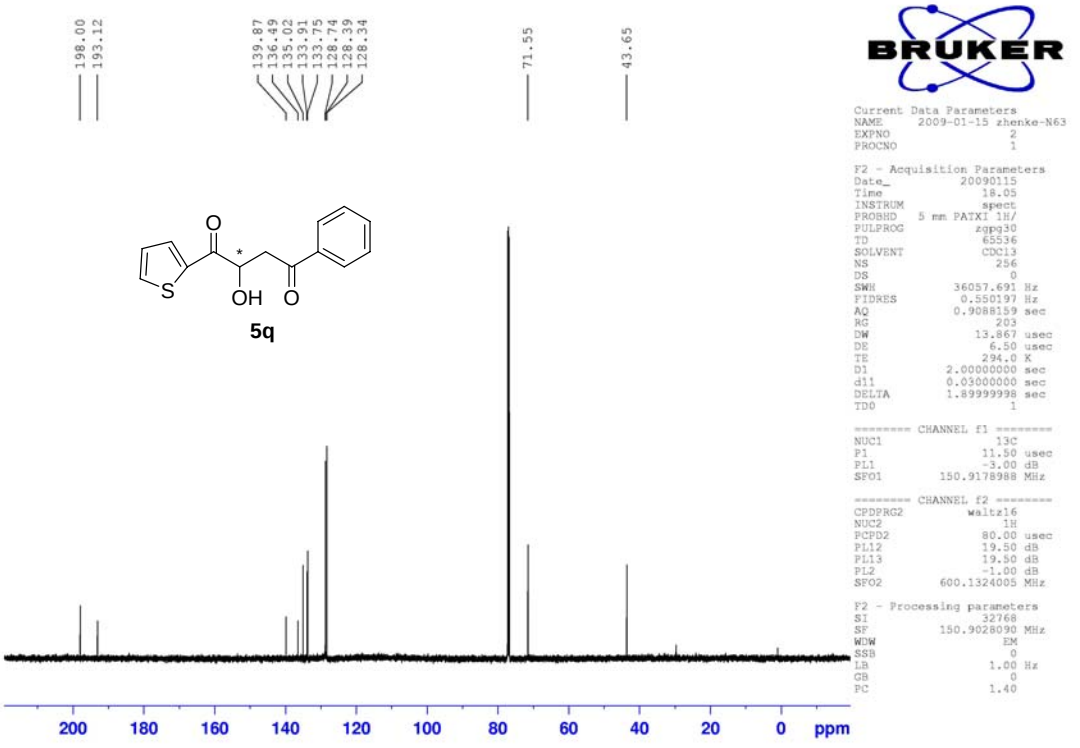

7.895
7.882
7.645
7.643
7.632
7.620
7.618
7.589
7.588
7.581
7.576
7.569
7.565
7.557
7.465
7.452
7.439
7.260
5.852
5.846
5.840
5.838
5.832
5.825
5.297
4.100
4.090
3.511
3.499
3.483
3.470
3.460
3.454
3.431
3.426
0.073
O
OH
O
5o
BRUKER
Current Data Parameters
NAME 2009-01-15 zhenke-N62
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 17.26
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 181
DW 40.533 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.20 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 32768
SF 600.1300096 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.01
1.01
1.04
3.01
1.05
1.06
2.07
2.11
1.00
0.93
2.06

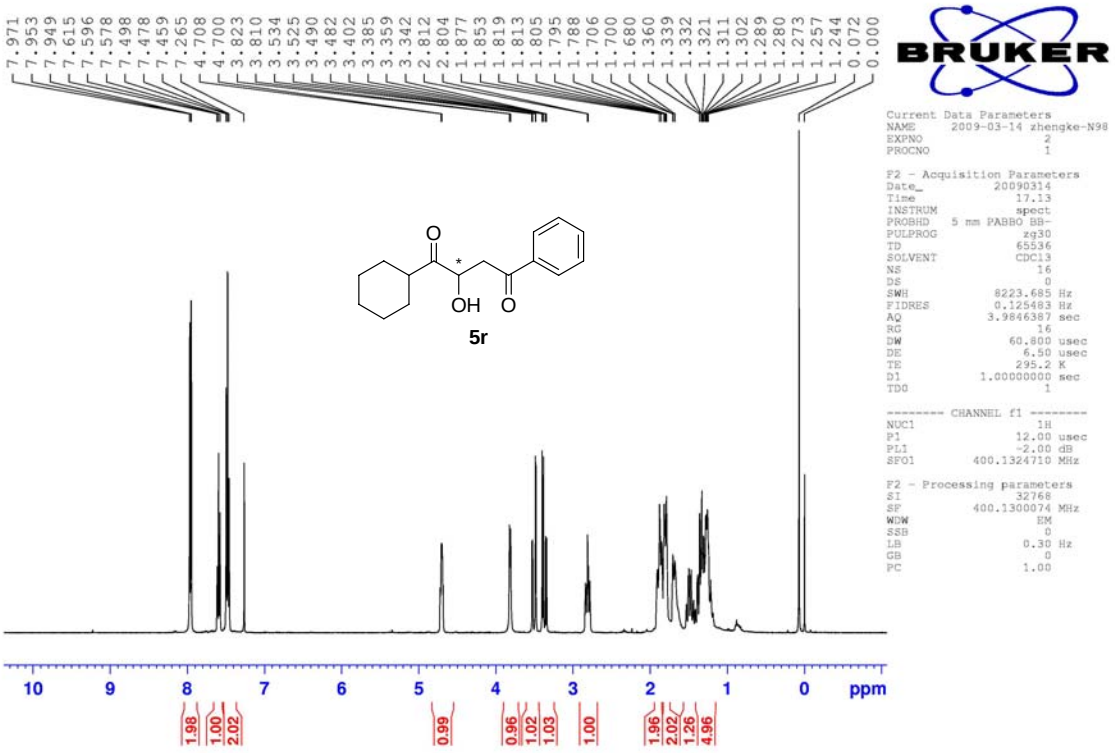

200.74
197.27
136.62
135.94
133.62
132.42
130.85
130.64
129.74
129.04
128.94
128.70
128.35
127.86
127.12
124.08
77.24
77.03
76.82
70.17
43.79
1.03
O
OH
O
5o
BRUKER
Current Data Parameters
NAME 2009-01-15 zhenke-N62
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 17.39
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.50 usec
PL1 -3.00 dB
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.50 dB
PL13 19.50 dB
PL2 -1.00 dB
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

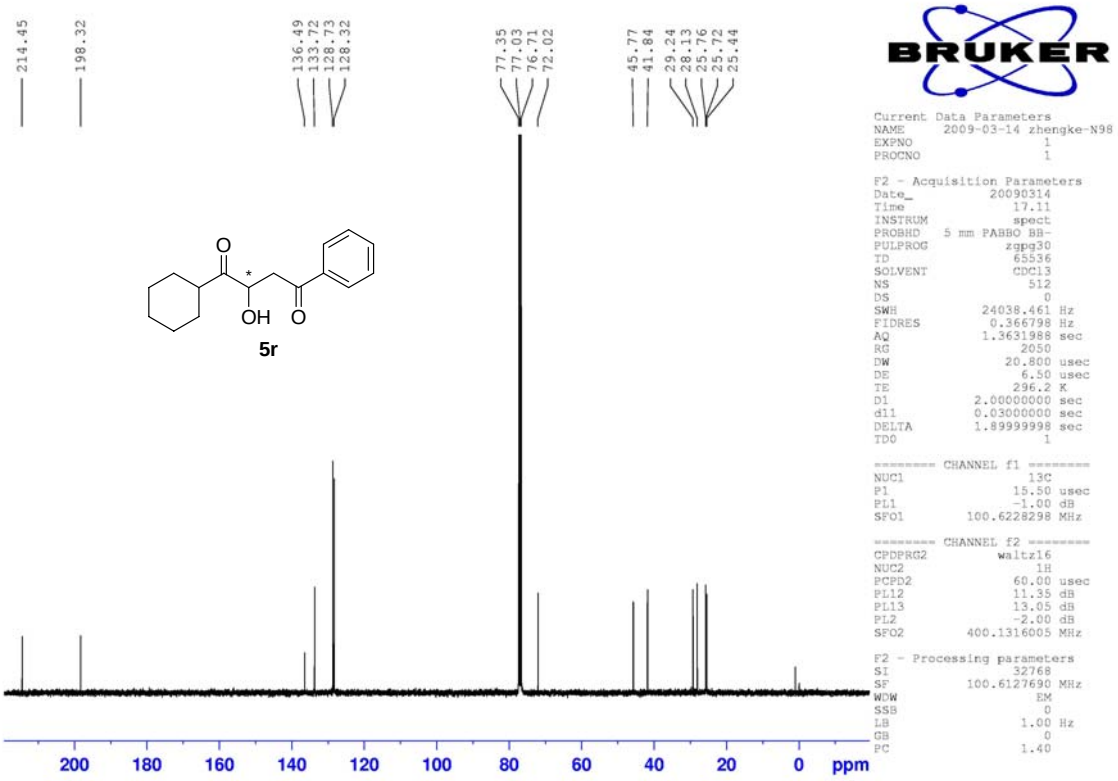

7.979
7.976
7.963
7.958
7.955
7.632
7.630
7.628
7.626
7.608
7.605
7.602
7.591
7.587
7.582
7.571
7.568
7.565
7.494
7.490
7.486
7.476
7.474
7.470
7.467
7.465
7.456
7.452
7.260
6.604
6.599
6.594
6.590
5.341
5.332
5.324
5.315
5.309
5.300
5.297
3.900
3.885
3.600
3.590
3.502
3.485
3.467
BRUKER
Current Data Parameters
NAME 2009-03-10 zhengke N94
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090310
Time 11.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300093 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
O
OH
O
5p
9
8
7
6
5
4
3
2
1
0
ppm
1.95
1.09
1.00
2.91
1.00
1.05
1.03
1.98

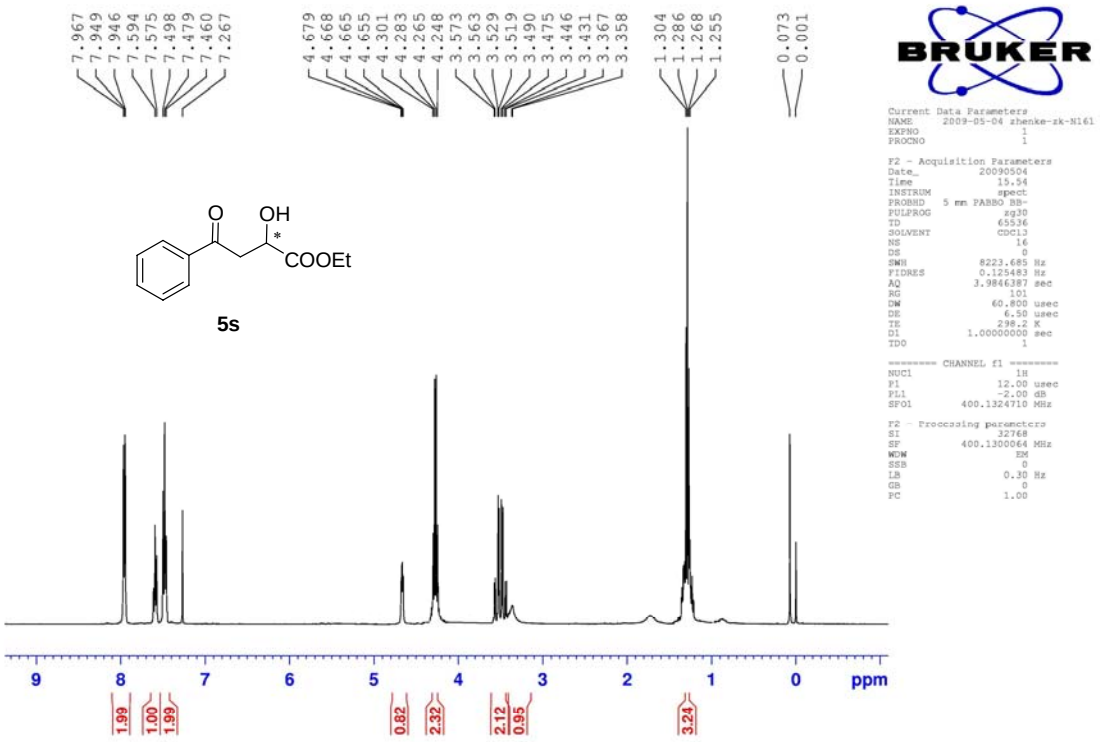

197.46
188.84
150.26
147.13
136.53
133.63
128.71
128.32
119.74
112.67
77.37
77.05
76.73
70.74
43.11
BRUKER
Current Data Parameters
NAME 2009-03-14 zhengke-N102
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090314
Time 16.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OH
O
5p
200
180
160
140
120
100
80
60
40
20
0
ppm


7.973
7.964
7.959
7.944
7.941
7.733
7.724
7.601
7.589
7.577
7.484
7.471
7.458
7.260
7.178
7.171
7.164
5.433
5.428
5.421
5.417
5.410
5.405
5.294
3.984
3.973
3.575
3.562
3.546
3.540
3.533
3.481
3.464
3.459
3.435
3.430
1.664
1.281
1.252
1.216
1.205
0.888
0.877
0.865
0.859
0.853
0.070
BRUKER
Current Data Parameters
NAME 2009-01-15 zhenke-N63
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 17.52
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 128
DW 40.533 usec
DE 6.50 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.20 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 32768
SF 600.1300092 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
O
S
OH
O
5q
10
9
8
7
6
5
4
3
2
1
0
ppm
3.07
0.97
1.07
2.08
1.03
1.00
0.95
1.06
1.07


198.00
193.12
139.87
136.49
135.02
133.91
133.75
128.74
128.39
128.34
71.55
43.65
BRUKER
Current Data Parameters
NAME 2009-01-15 zhenke-N63
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 18.05
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 6.50 usec
TE 294.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.50 usec
PL1 -3.00 dB
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.50 dB
PL13 19.50 dB
PL2 -1.00 dB
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
S
OH
O
5q
200
180
160
140
120
100
80
60
40
20
0
ppm


7.971
7.953
7.949
7.615
7.596
7.578
7.498
7.478
7.459
7.265
4.708
4.700
3.823
3.810
3.534
3.525
3.490
3.482
3.402
3.385
3.359
3.342
2.812
2.804
1.877
1.853
1.819
1.813
1.805
1.795
1.788
1.706
1.700
1.680
1.360
1.339
1.332
1.321
1.311
1.302
1.289
1.280
1.273
1.257
1.244
0.072
0.000
BRUKER
Current Data Parameters
NAME 2009-03-14 zhengke-N98
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090314
Time 17.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OH
O
5r
10
9
8
7
6
5
4
3
2
1
0
ppm
1.98
1.00
2.02
0.99
0.96
1.02
1.03
1.00
1.96
2.02
1.26
4.96


214.45
198.32
136.49
133.72
128.73
128.32
77.35
77.03
76.71
72.02
45.77
41.84
29.24
28.13
25.76
25.72
25.44
BRUKER
Current Data Parameters
NAME 2009-03-14 zhengke-N98
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090314
Time 17.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OH
O
5r
200
180
160
140
120
100
80
60
40
20
0
ppm


7.967
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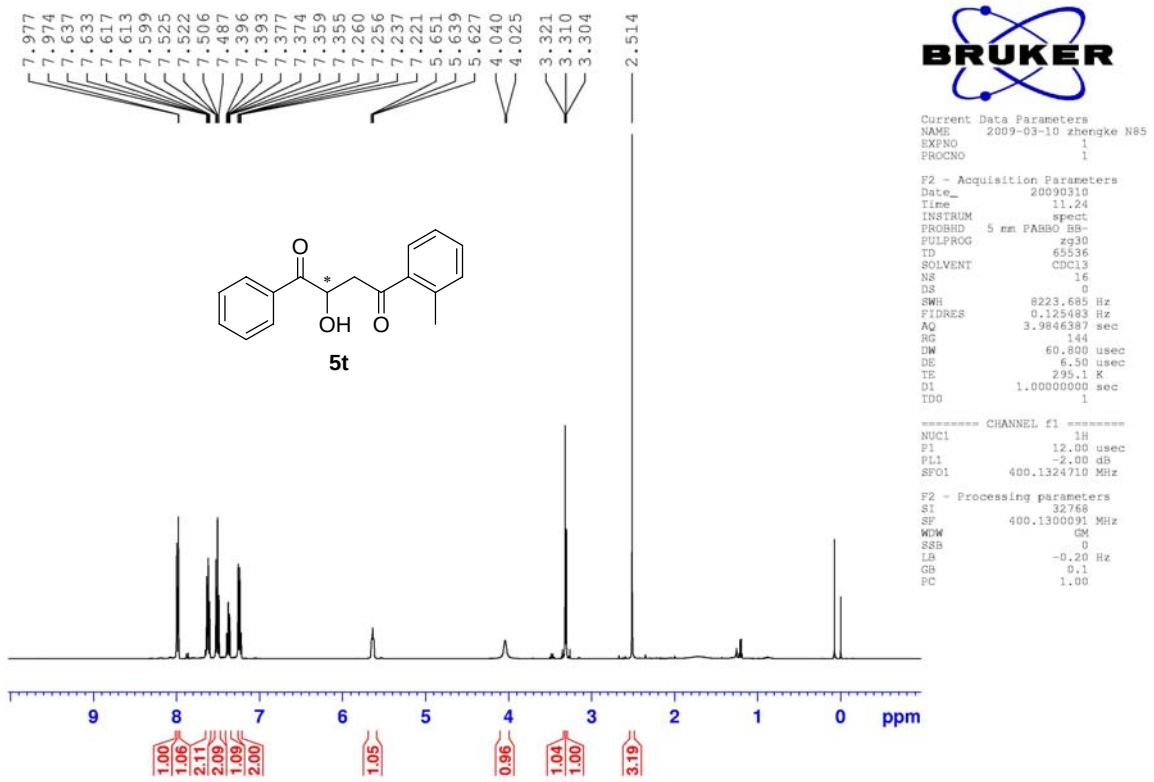
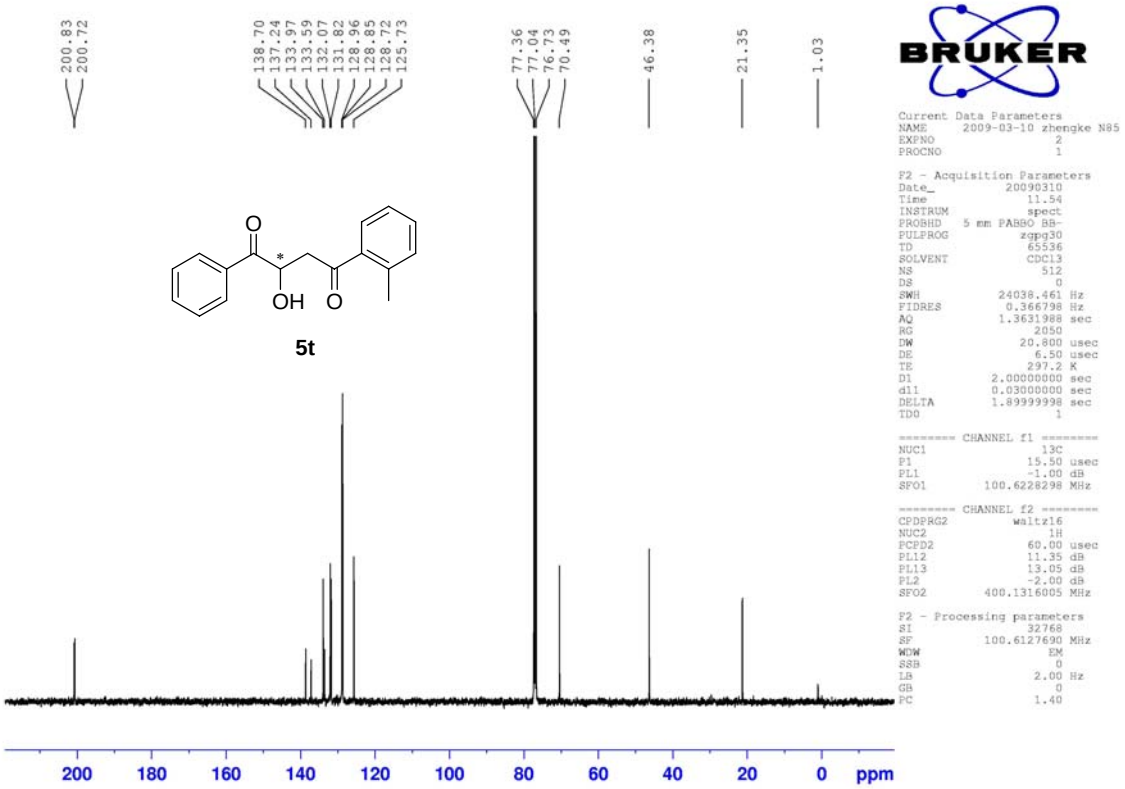
7.946
7.594
7.575
7.498
7.479
7.460
7.267
4.679
4.668
4.665
4.655
4.301
4.283
4.265
4.248
3.573
3.563
3.529
3.519
3.490
3.475
3.446
3.431
3.367
3.358
1.304
1.286
1.268
1.255
0.073
0.001
O
OH
*
COOEt
5s
BRUKER
Current Data Parameters
NAME 2009-05-04 zhenke-zk-N161
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090504
Time 15.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.99
1.00
1.99
0.82
2.32
2.12
0.95
3.24


7.977
7.974
7.637
7.633
7.617
7.613
7.599
7.525
7.522
7.506
7.487
7.396
7.393
7.377
7.374
7.359
7.355
7.260
7.256
7.237
7.221
5.651
5.639
5.627
4.040
4.025
3.321
3.310
3.304
2.514
O
*
OH
O
5t
BRUKER
Current Data Parameters
NAME 2009-03-10 zhengke N85
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090310
Time 11.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300091 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.06
2.11
2.09
1.09
2.00
1.05
0.96
1.04
1.00
3.19



5t

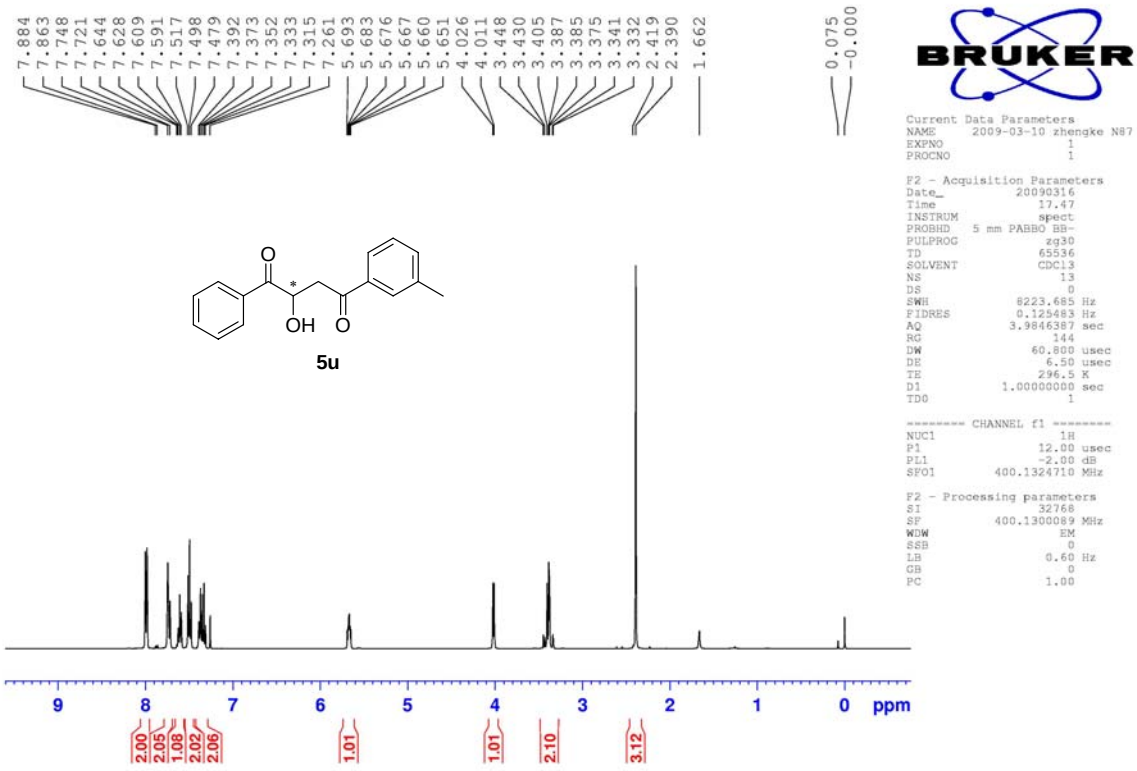
Current Data Parameters
NAME 2009-03-10 zhengke N85
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090310
Time 11.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



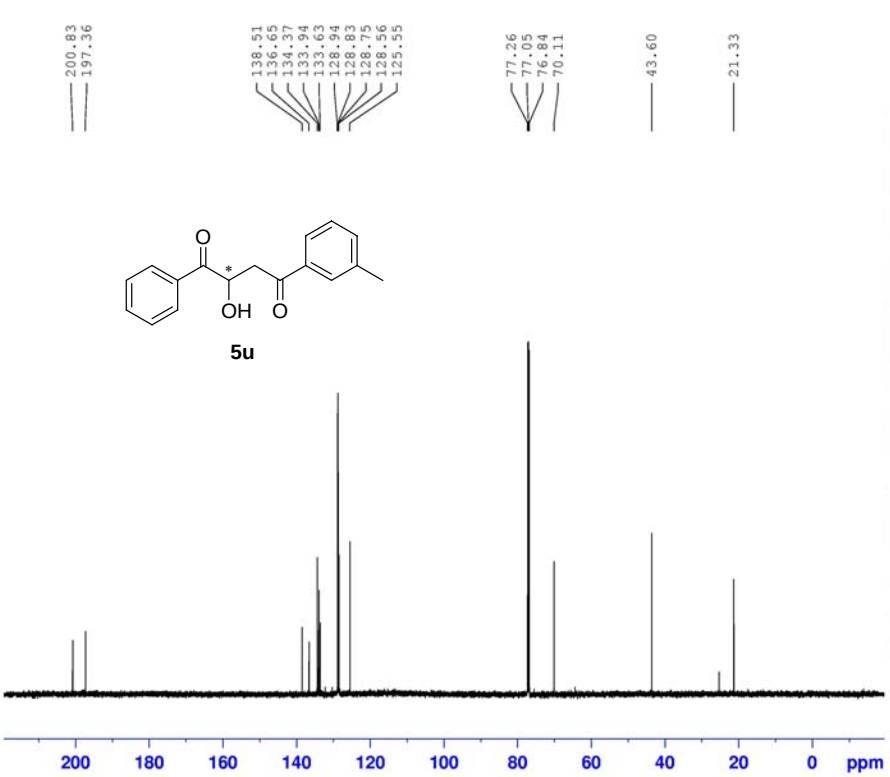
5u

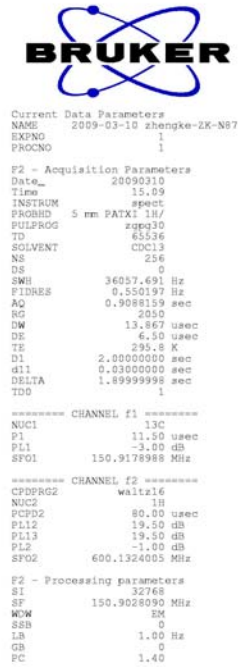
Current Data Parameters
NAME 2009-03-10 zhengke N87
EXPNO 1
PROCNO 1

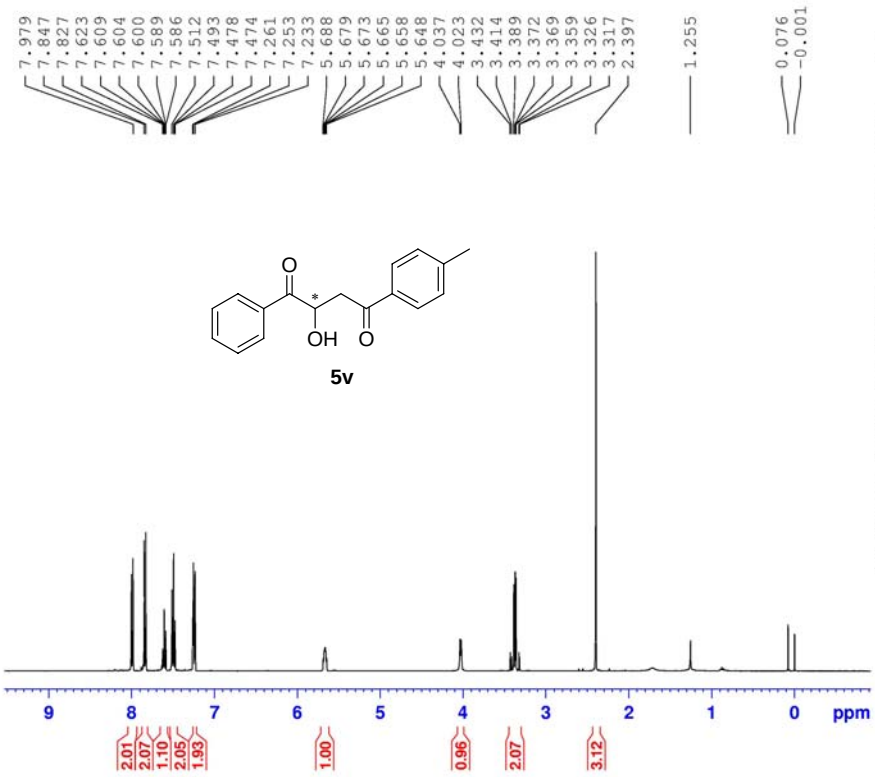
F2 - Acquisition Parameters
Date_ 20090316
Time 17.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

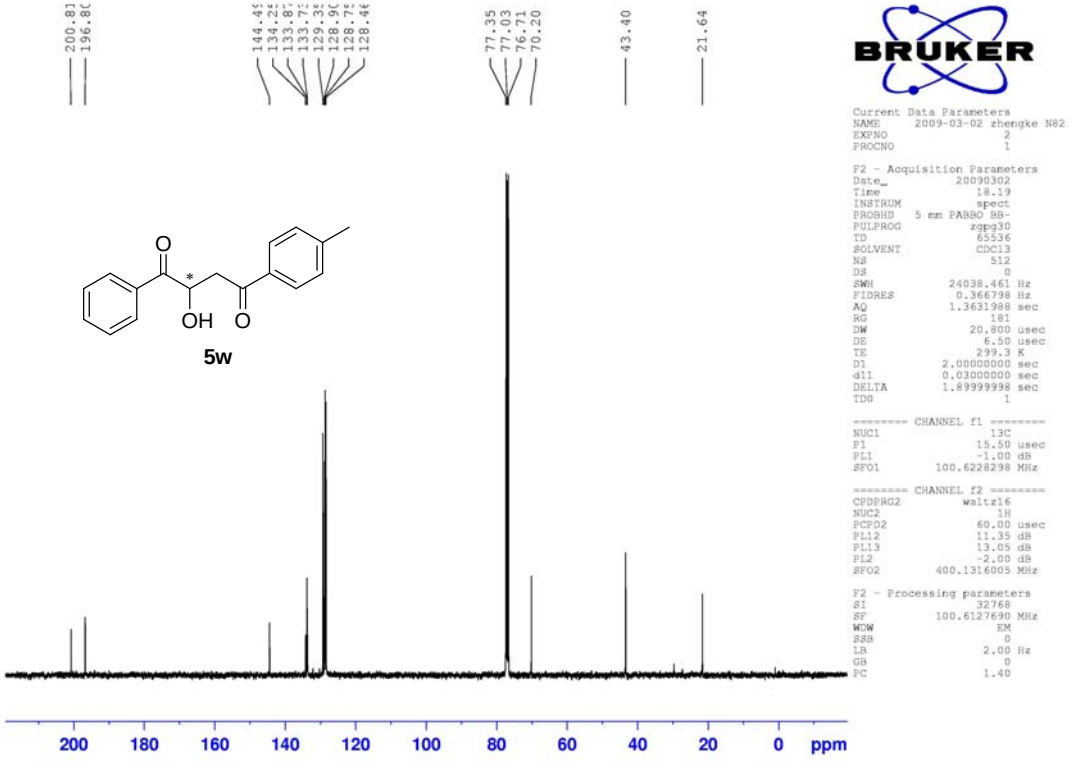
F2 - Processing parameters
SI 32768
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

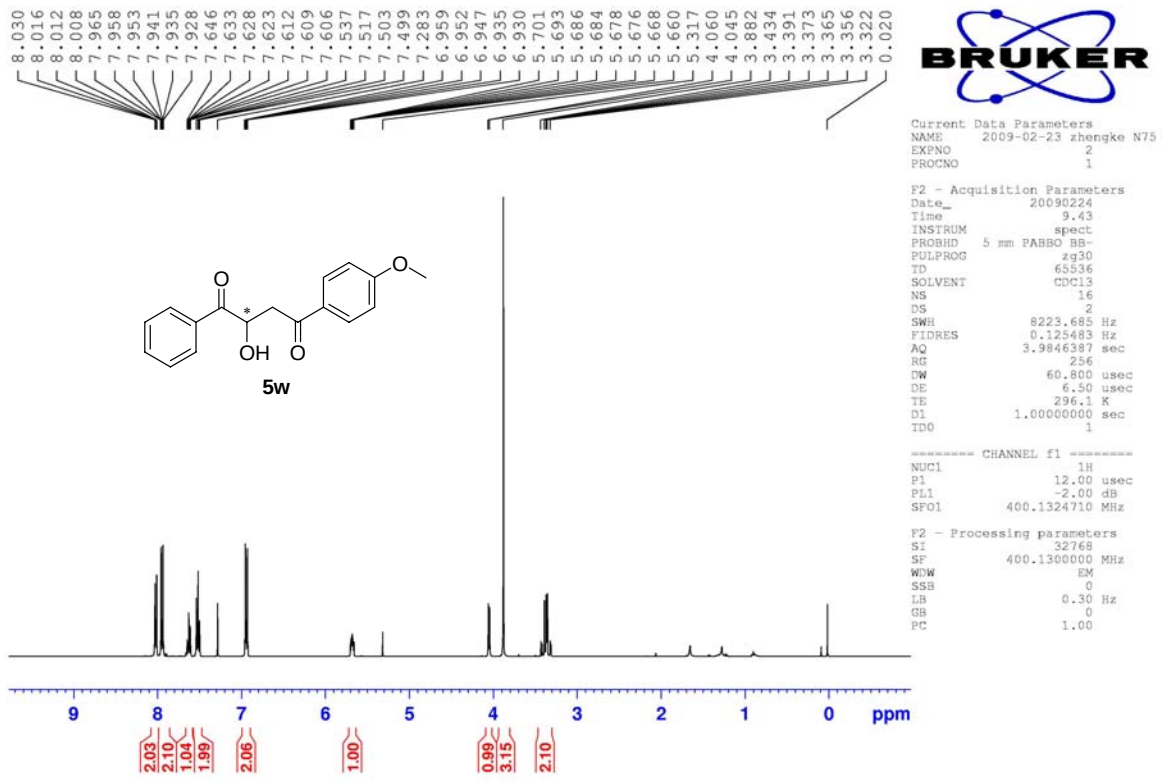

200.83
197.36
138.51
136.65
134.37
133.94
133.63
128.94
128.83
128.75
128.56
125.55
77.26
77.05
76.84
70.11
43.60
21.33
O
OH
O
5u
200
180
160
140
120
100
80
60
40
20
0
ppm

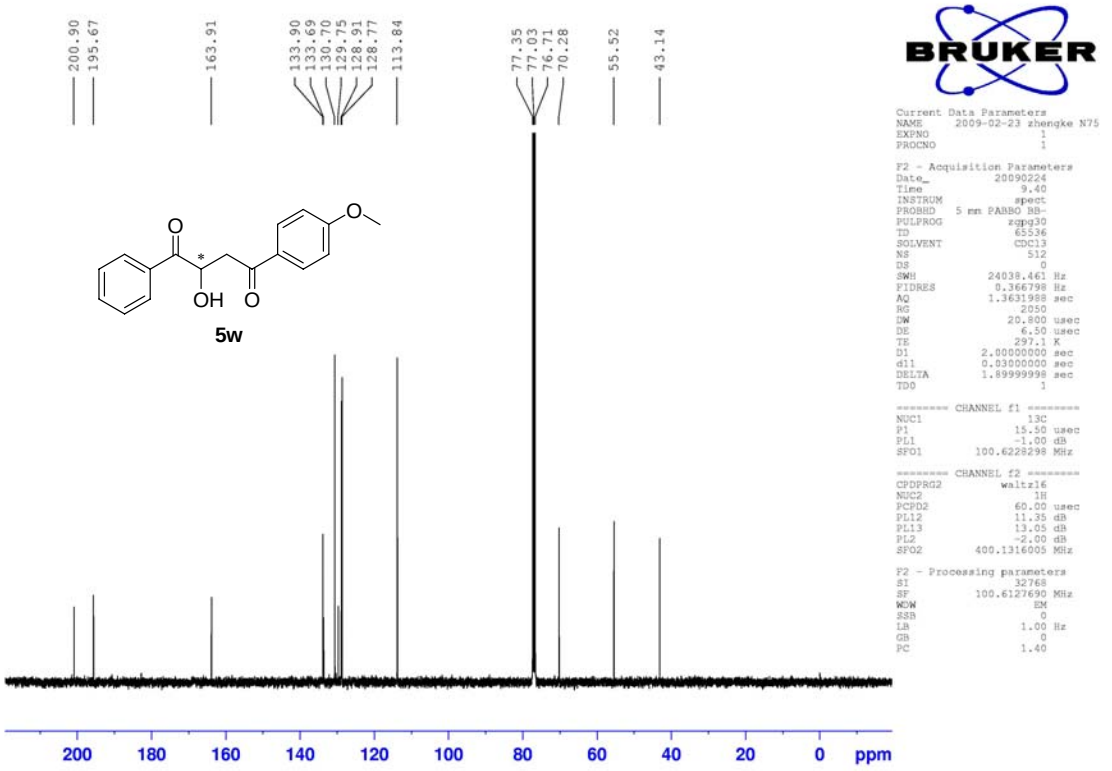

BRUKER
Current Data Parameters
NAME 2009-03-10 zhengke-ZK-N87
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090310
Time 15.09
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 2050
DW 13.867 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.50 usec
PL1 -3.00 dB
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.50 dB
PL13 19.50 dB
PL2 -1.00 dB
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

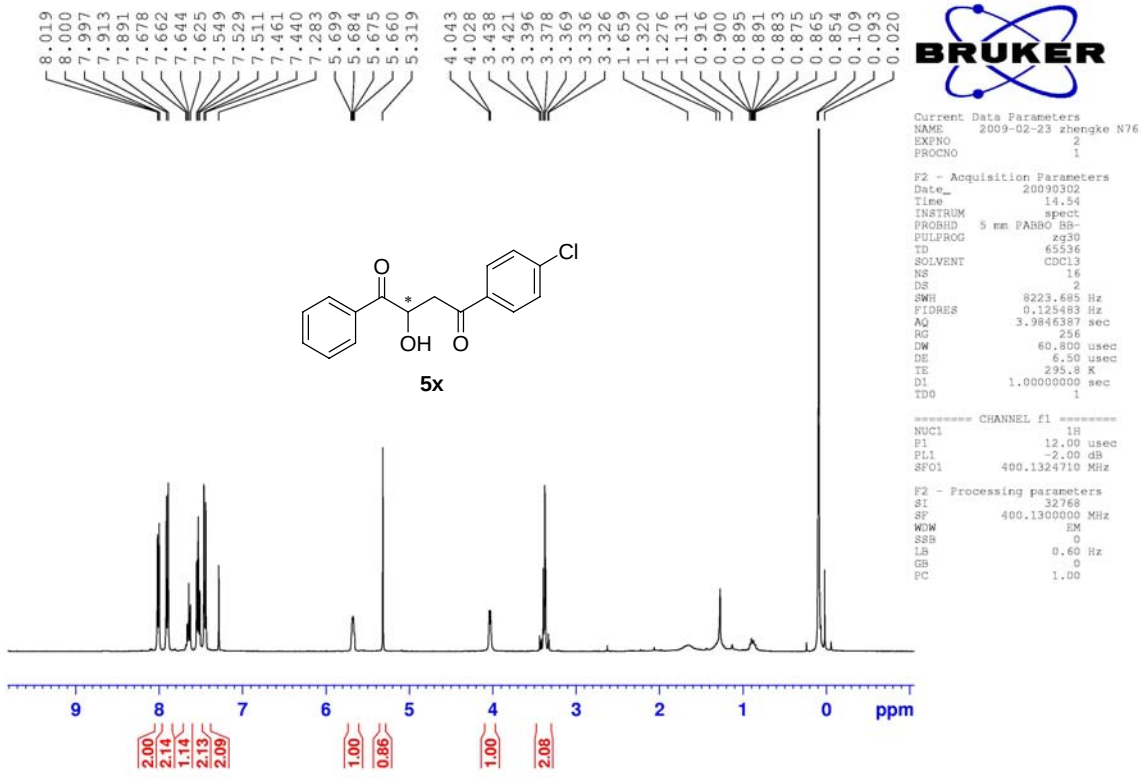

7.979
7.847
7.827
7.623
7.609
7.604
7.600
7.589
7.586
7.512
7.493
7.478
7.474
7.261
7.253
7.233
5.688
5.679
5.673
5.665
5.658
5.648
4.037
4.023
3.432
3.414
3.389
3.372
3.369
3.359
3.326
3.317
2.397
1.255
0.076
-0.001
O
OH
O
5v
9
8
7
6
5
4
3
2
1
0
ppm
2.01
2.07
1.10
2.05
1.93
1.00
0.96
2.07
3.12

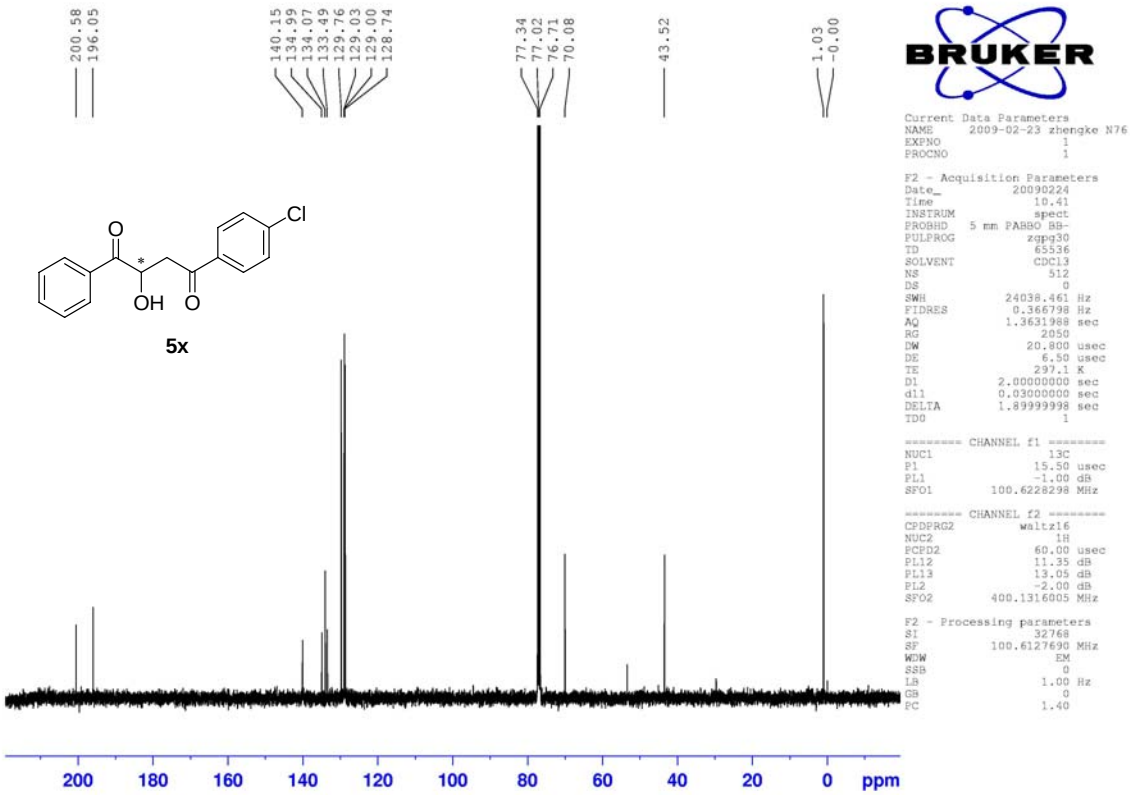

BRUKER
Current Data Parameters
NAME 2009-03-02 zhengke N82
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090302
Time 17.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846367 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300088 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00

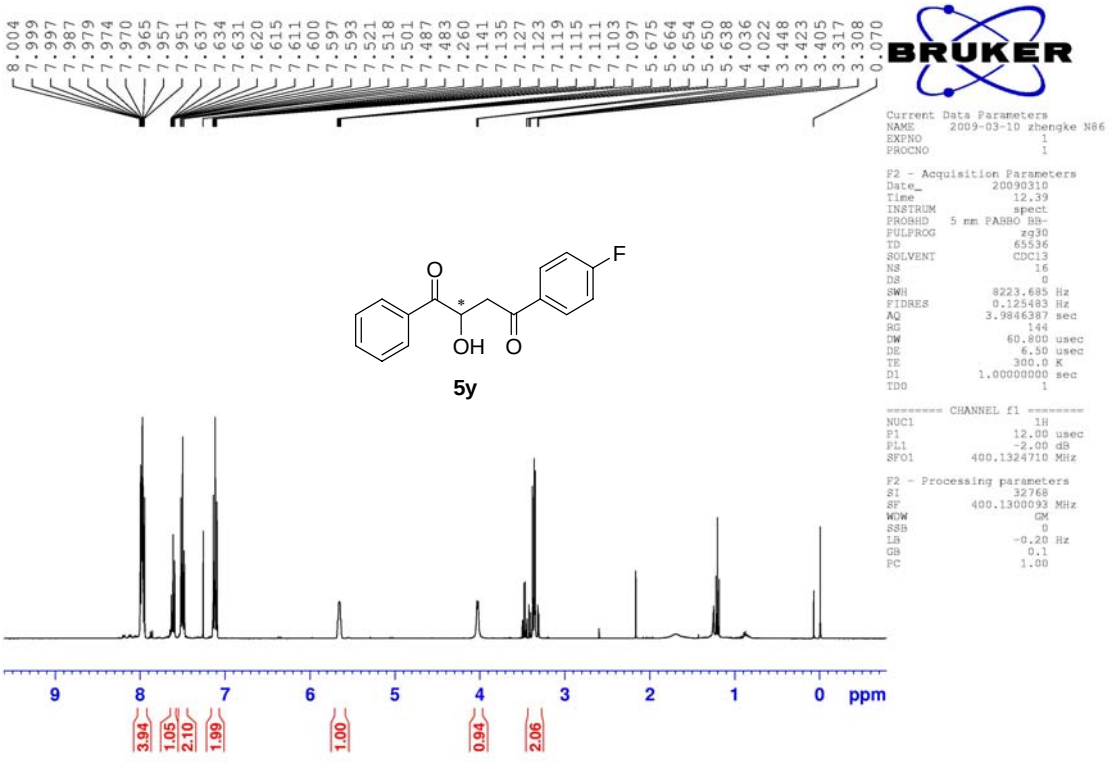

200.81
196.80
144.45
134.25
133.87
133.72
129.38
128.90
128.75
128.46
77.35
77.03
76.71
70.20
43.40
21.64
O
*
OH
O
5w
BRUKER
Current Data Parameters
NAME 2009-03-02 zhengke N82
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090302
Time 18.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

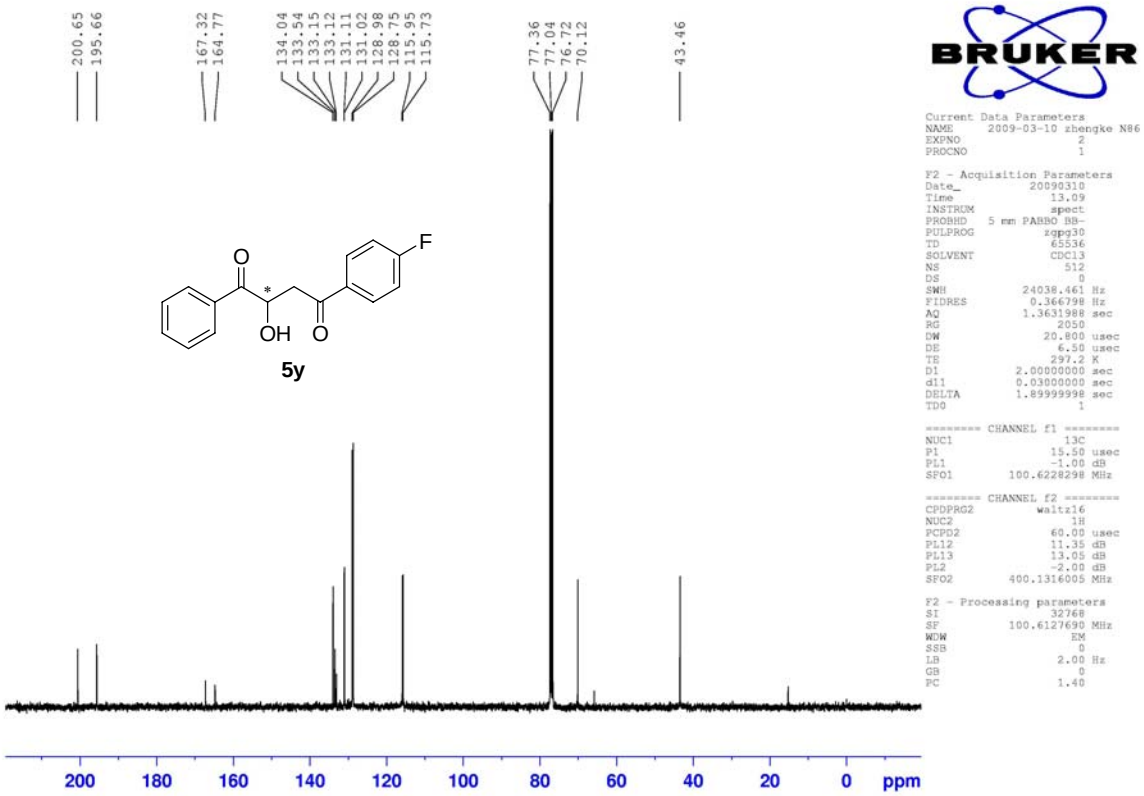

8.030
8.016
8.012
8.008
7.965
7.958
7.953
7.941
7.935
7.928
7.646
7.633
7.628
7.623
7.612
7.609
7.606
7.537
7.517
7.503
7.499
7.283
6.959
6.952
6.947
6.935
6.930
5.701
5.693
5.686
5.684
5.678
5.676
5.668
5.660
5.317
4.060
4.045
3.882
3.434
3.391
3.373
3.365
3.356
3.322
0.020
O
*
OH
O
O
5w
BRUKER
Current Data Parameters
NAME 2009-02-23 zhengke N75
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090224
Time 9.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.10
1.04
1.99
2.06
1.00
0.99
3.15
2.10

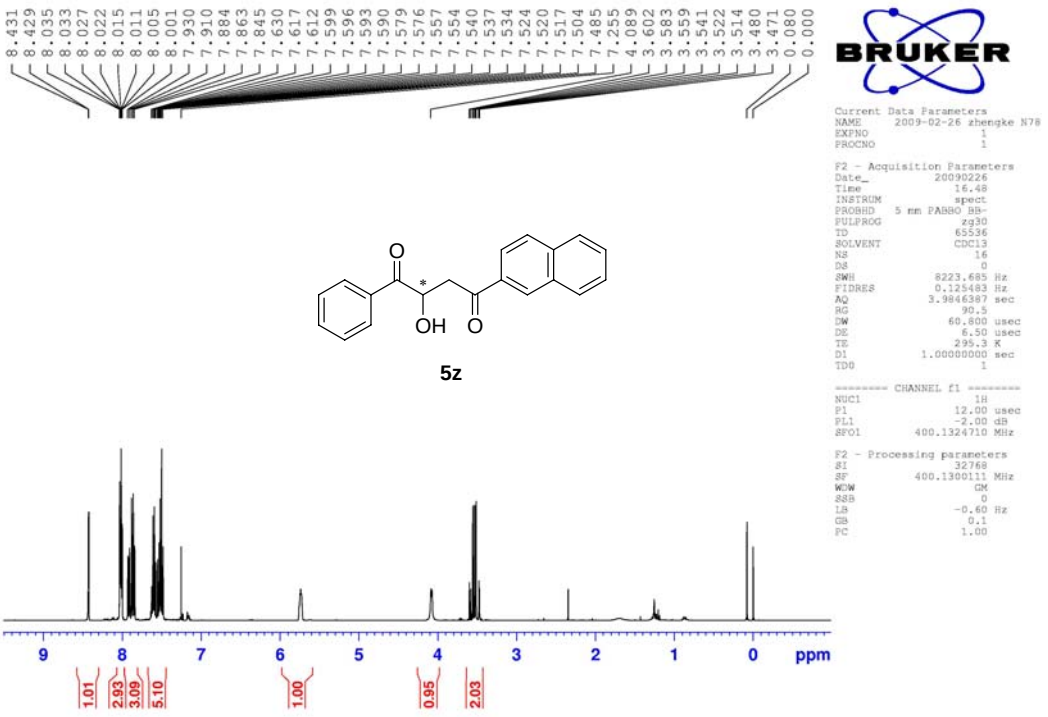

200.90
195.67
163.91
133.90
133.69
130.70
129.75
128.91
128.77
113.84
77.35
77.03
76.71
70.28
55.52
43.14
O
O
OH
O
5w
BRUKER
Current Data Parameters
NAME 2009-02-23 zhengke N75
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090224
Time 9.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

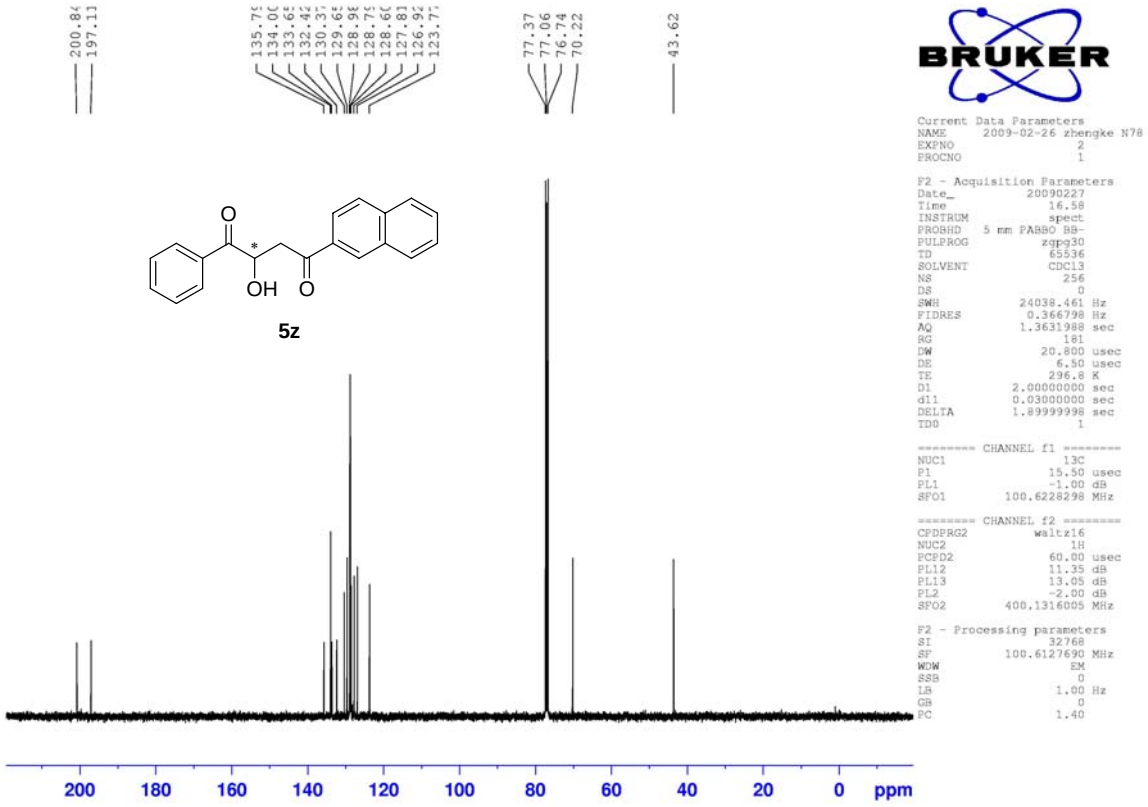

8.019
8.000
7.997
7.913
7.891
7.678
7.662
7.644
7.625
7.549
7.529
7.511
7.461
7.440
7.283
5.699
5.684
5.675
5.660
5.319
4.043
4.028
3.438
3.421
3.396
3.378
3.369
3.336
3.326
1.659
1.320
1.276
1.131
0.916
0.900
0.895
0.891
0.883
0.875
0.865
0.854
0.109
0.093
0.020
O
O
OH
O
Cl
5x
BRUKER
Current Data Parameters
NAME 2009-02-23 zhengke N76
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090302
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.14
1.14
2.13
2.09
1.00
0.86
1.00
2.08


200.58
196.05
140.15
134.99
134.07
133.49
129.76
129.03
129.00
128.74
77.34
77.02
76.71
70.08
43.52
1.03
0.00
Cl
O
OH
O
5x
200
180
160
140
120
100
80
60
40
20
0
ppm


8.004
7.999
7.997
7.987
7.979
7.974
7.970
7.965
7.957
7.951
7.637
7.634
7.631
7.620
7.615
7.611
7.600
7.597
7.593
7.521
7.518
7.501
7.487
7.483
7.260
7.141
7.135
7.127
7.123
7.119
7.115
7.111
7.103
7.097
5.675
5.664
5.654
5.650
5.638
4.036
4.022
3.448
3.423
3.405
3.317
3.308
0.070
F
O
OH
O
5y
9
8
7
6
5
4
3
2
1
0
ppm
3.94
1.05
2.10
1.99
1.00
0.94
2.06


200.65
195.66
167.32
164.77
134.04
133.54
133.15
133.12
131.11
131.02
128.98
128.75
115.95
115.73
77.36
77.04
76.72
70.12
43.46
O
F
*
OH
O
5y
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME 2009-03-10 zhengke N86
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090310
Time 13.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40


8.431
8.429
8.035
8.033
8.027
8.022
8.015
8.011
8.005
8.001
7.930
7.910
7.884
7.863
7.845
7.630
7.617
7.612
7.599
7.596
7.593
7.590
7.579
7.576
7.557
7.554
7.540
7.537
7.534
7.524
7.520
7.517
7.504
7.485
7.255
4.089
3.602
3.583
3.559
3.541
3.522
3.514
3.480
3.471
0.080
0.000
O
*
OH
O
5z
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.93
3.09
5.10
1.00
0.95
2.03
BRUKER
Current Data Parameters
NAME 2009-02-26 zhengke N78
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090226
Time 16.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300111 MHz
WDW EM
SSB 0
LB -0.60 Hz
GB 0.1
PC 1.00



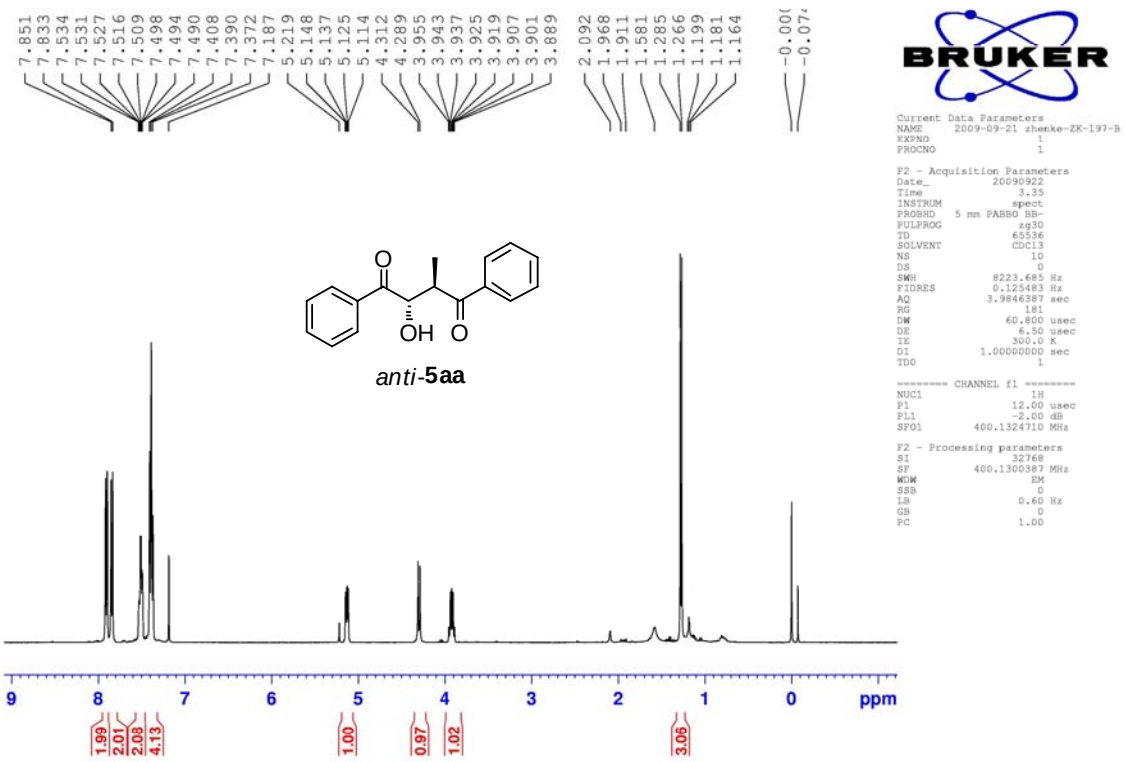
Current Data Parameters
NAME 2009-02-26 zhengke N78
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090227
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

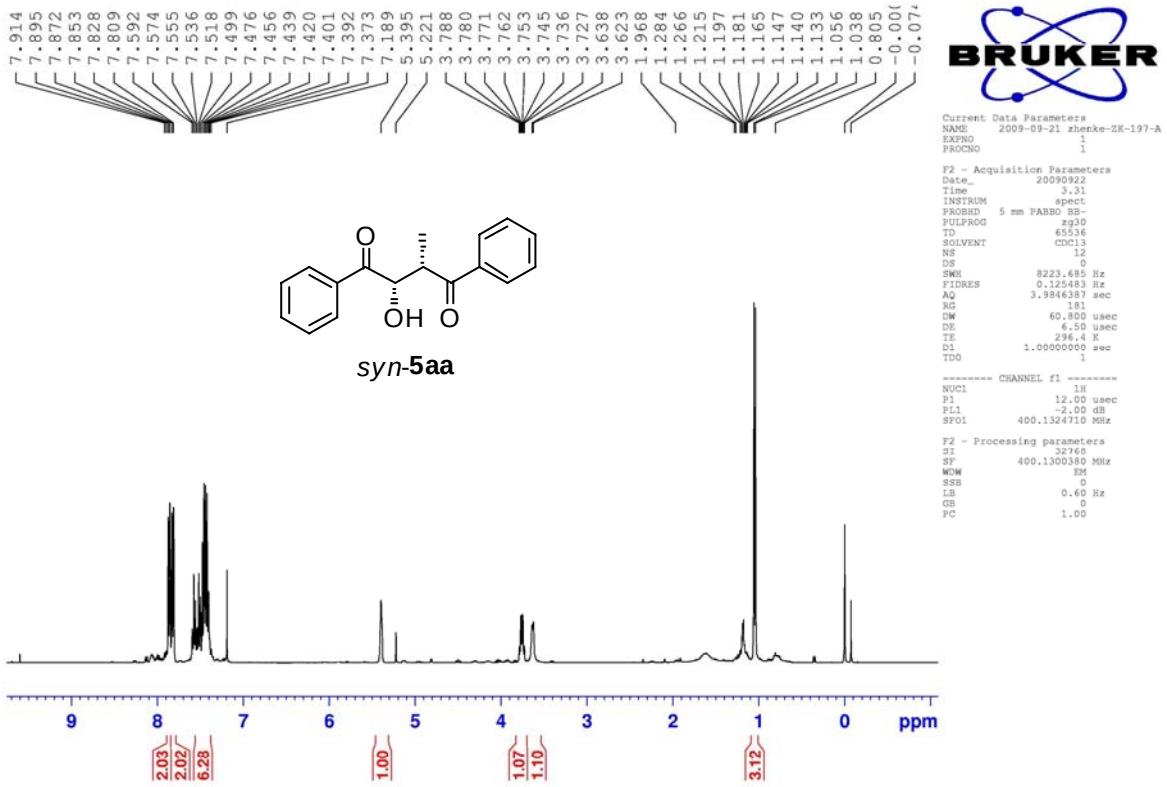


Current Data Parameters
NAME 2009-09-21 zhenke-ZK-197-B
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090922
Time 3.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300387 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00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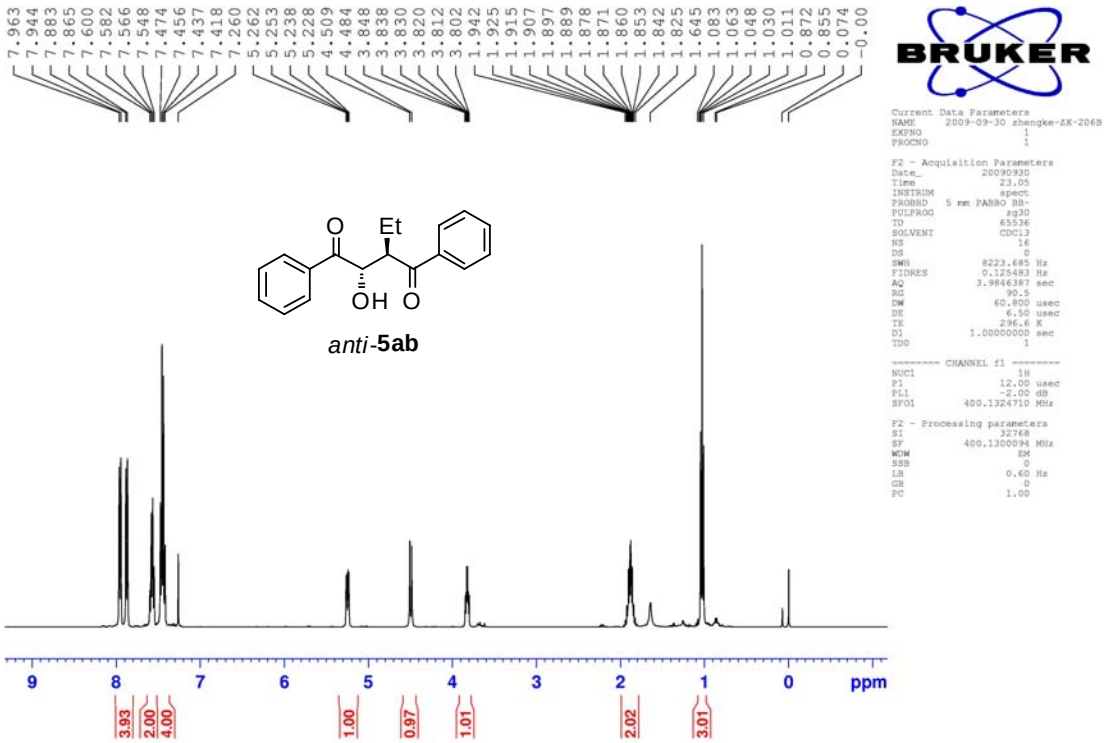
7.895
7.872
7.853
7.828
7.809
7.592
7.574
7.555
7.536
7.518
7.499
7.476
7.456
7.439
7.420
7.401
7.392
7.373
7.189
5.395
5.221
3.788
3.780
3.771
3.762
3.753
3.745
3.736
3.727
3.638
3.623
1.968
1.284
1.266
1.215
1.197
1.181
1.165
1.147
1.140
1.133
1.056
1.038
0.805
BRUKER
Current Data Parameters
NAME 2009-09-21 zhenke-ZK-197-A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090922
Time 3.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SF01 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300380 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
O
OH O
syn-5aa
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.02
6.28
1.00
1.07
1.10
3.12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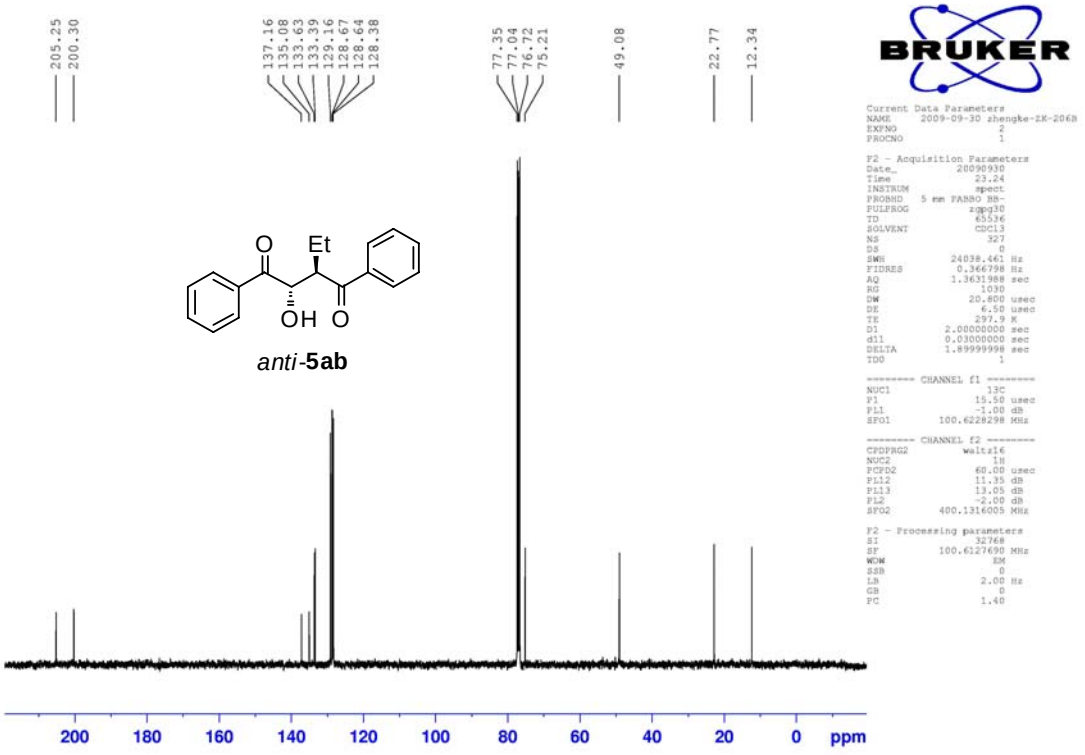
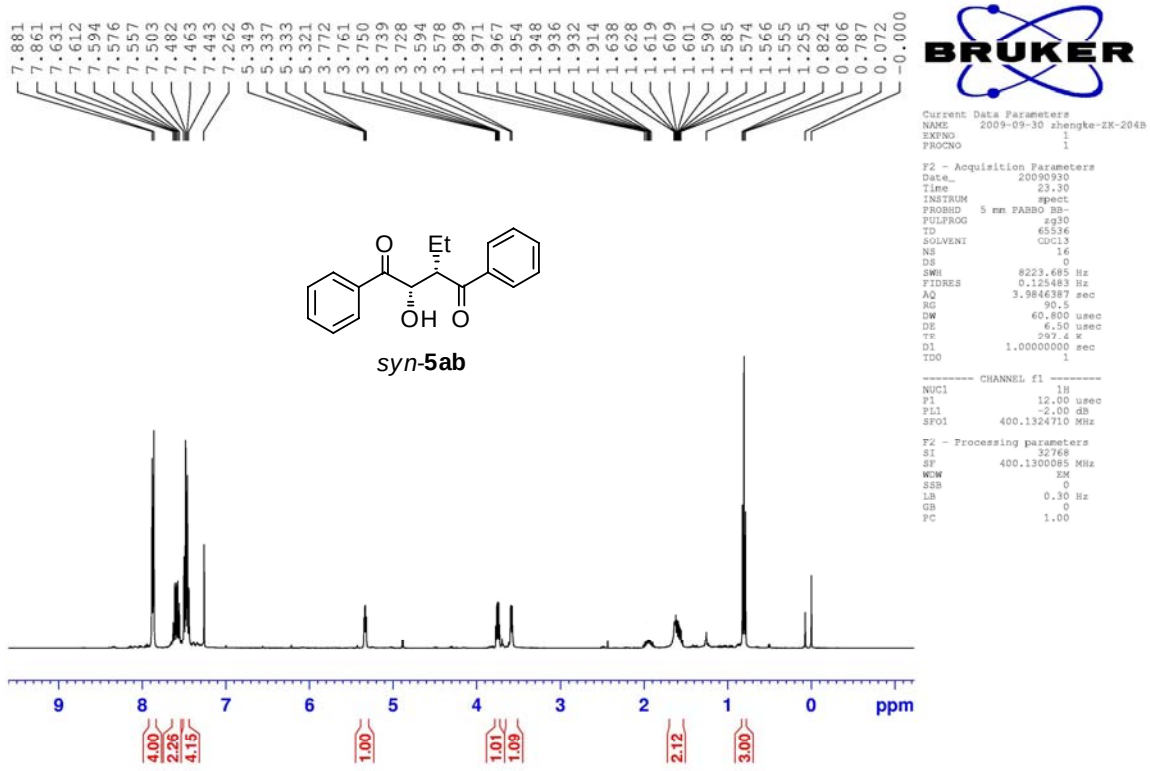
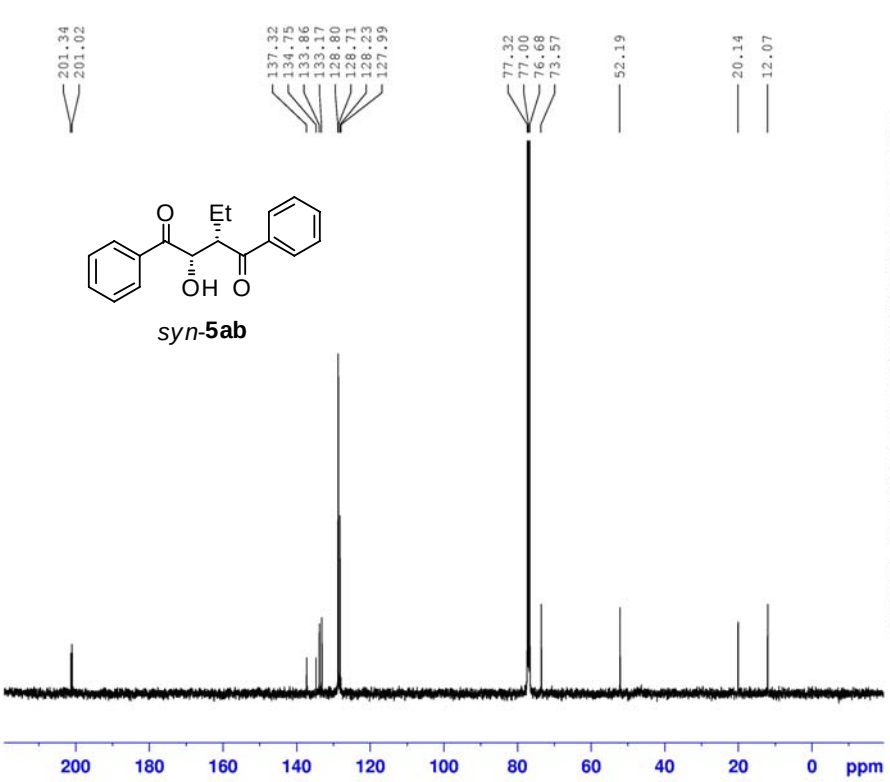
7.944
7.883
7.865
7.600
7.582
7.566
7.548
7.474
7.456
7.437
7.418
7.260
5.262
5.253
5.238
5.228
4.509
4.484
3.848
3.838
3.830
3.820
3.812
3.802
1.942
1.925
1.915
1.907
1.897
1.889
1.878
1.871
1.860
1.853
1.842
1.825
1.645
1.083
1.063
1.048
1.030
1.011
0.872
0.855
0.074
-0.00
BRUKER
Current Data Parameters
NAME 2009-09-30 zhengke-ZK-206B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090930
Time 23.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SF01 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
O
Et
OH O
anti-5ab
9
8
7
6
5
4
3
2
1
0
ppm
3.93
2.00
4.00
1.00
0.97
1.01
2.02
3.01


205.25
200.30
137.16
135.08
133.63
133.39
129.16
128.67
128.64
128.38
77.35
77.04
76.72
75.21
49.08
22.77
12.34
O
Et
OH O
anti-5ab
200
180
160
140
120
100
80
60
40
20
0
ppm


7.881
7.861
7.631
7.612
7.594
7.576
7.557
7.503
7.482
7.463
7.443
7.262
5.349
5.337
5.333
5.321
3.772
3.761
3.750
3.739
3.728
3.594
3.578
1.989
1.971
1.967
1.954
1.948
1.936
1.932
1.914
1.638
1.628
1.619
1.609
1.601
1.590
1.585
1.574
1.566
1.555
1.255
0.824
0.806
0.787
0.072
-0.000
O
Et
OH O
syn-5ab
9
8
7
6
5
4
3
2
1
0
ppm
4.00
2.26
4.15
1.00
1.01
1.09
2.12
3.00



syn-**5ab**

BRUKER

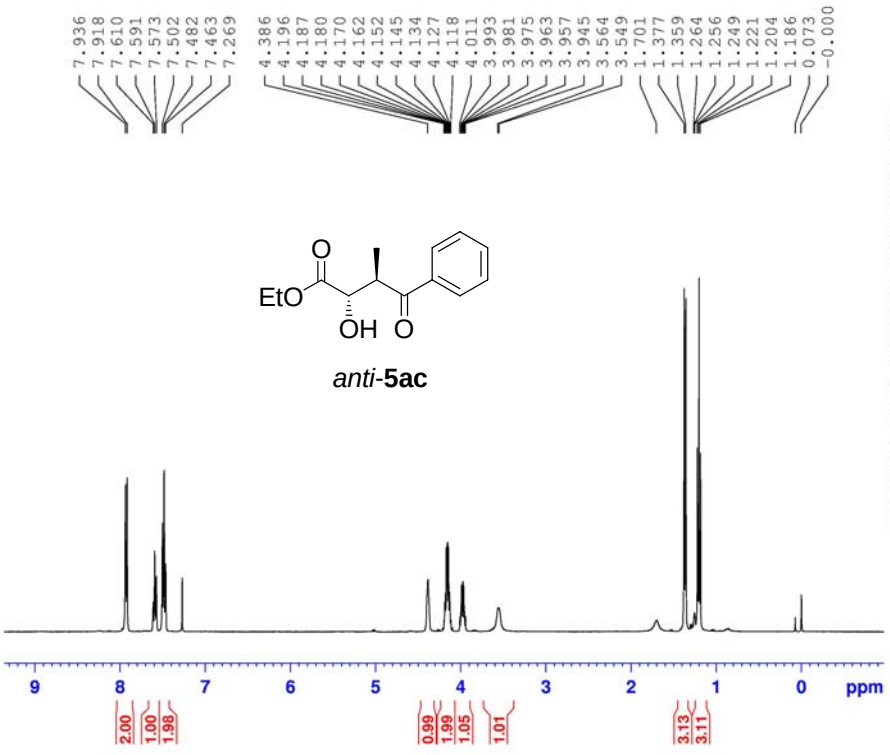
```
Current Data Parameters
NAME       2009-09-30 zhengke-ZK-204B
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20091001
Time               0.00
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                512
DS                  0
SWH         24038.461 Hz
FIDRES      0.366798 Hz
AQ          1.3631988 sec
RG               1030
DW             20.800 usec
DE              6.50 usec
TE             300.5 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                1

======== CHANNEL f1 ========
NUC1              13C
P1              15.50 usec
PL1             -1.00 dB
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2          60.00 usec
PL12           11.35 dB
PL13           13.05 dB
PL2            -2.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127690 MHz
WDW                EM
SSB                 0
LB               2.00 Hz
GB                  0
PC               1.40
```



anti-**5ac**

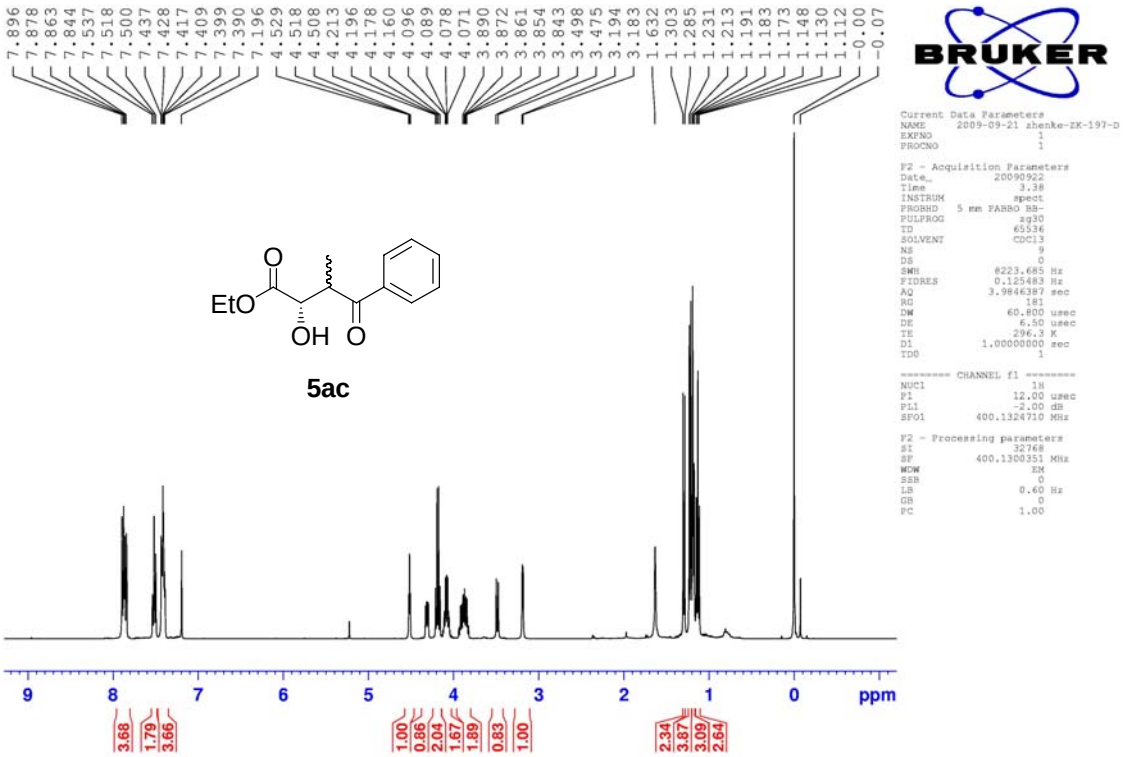
BRUKER

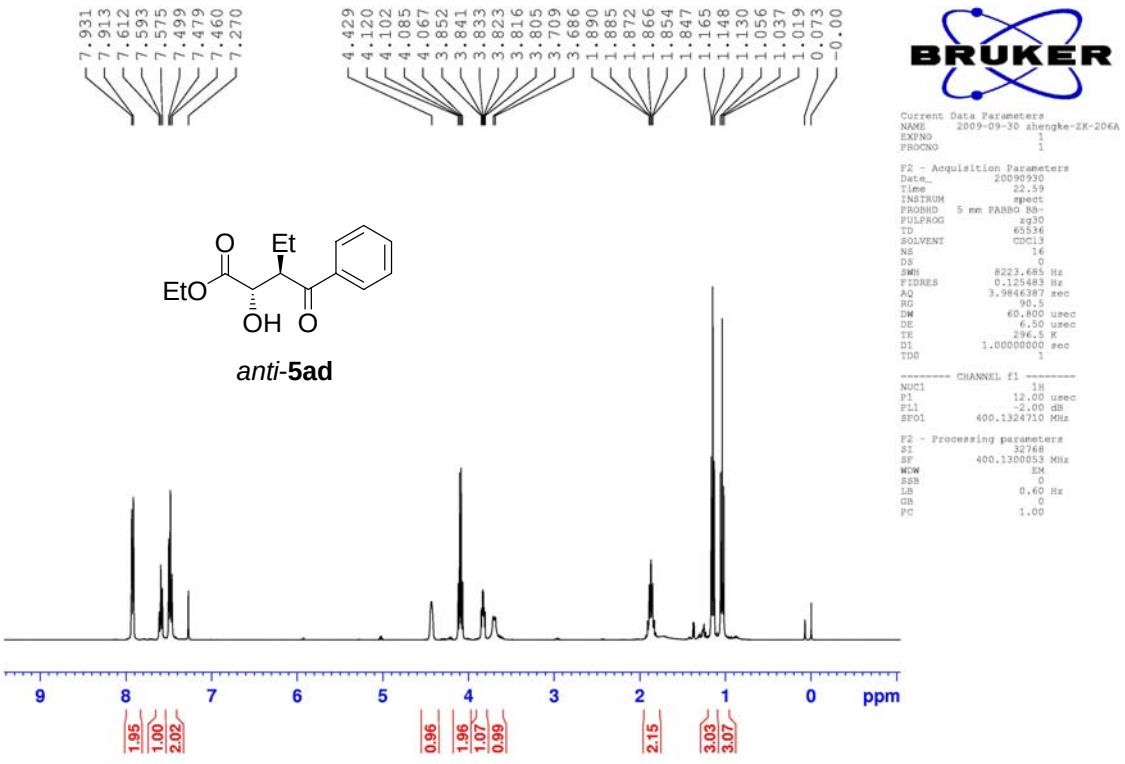
```
Current Data Parameters
NAME       2009-09-30 zhengke-ZK-203C
EXPNO                 1
PROCNO                1

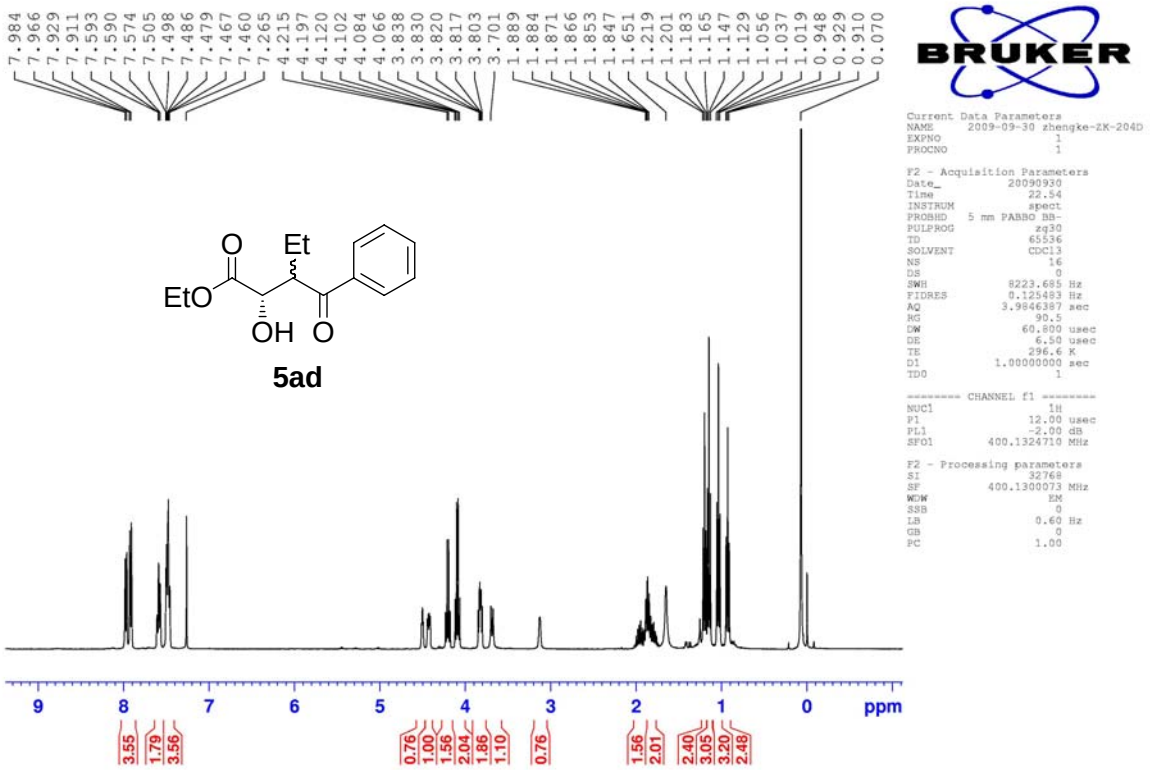
F2 - Acquisition Parameters
Date_          20090930
Time              22.49
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD              65536
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          8223.685 Hz
FIDRES      0.125483 Hz
AQ          3.9846387 sec
RG                90.5
DW             60.800 usec
DE              6.50 usec
TE             296.5 K
D1          1.00000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1               1H
P1              12.00 usec
PL1             -2.00 dB
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                32768
SF         400.1300057 MHz
WDW                EM
SSB                 0
LB               0.60 Hz
GB                  0
PC               1.00
```


7.896
7.878
7.863
7.844
7.537
7.518
7.500
7.437
7.428
7.417
7.409
7.399
7.390
7.196
4.529
4.518
4.508
4.213
4.196
4.178
4.160
4.096
4.089
4.078
4.071
3.890
3.872
3.861
3.854
3.843
3.498
3.475
3.194
3.183
1.632
1.303
1.285
1.231
1.213
1.191
1.183
1.173
1.148
1.130
1.112
-0.00
-0.07
BRUKER
Current Data Parameters
NAME 2009-09-21 zhenke-ZK-197-D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090922
Time 3.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300351 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
EtO
OH
O
O
5ac
9
8
7
6
5
4
3
2
1
0
ppm
3.68
1.79
3.66
1.00
0.86
2.04
1.67
1.89
0.83
1.00
2.34
3.87
3.09
2.64


7.931
7.913
7.612
7.593
7.575
7.499
7.479
7.460
7.270
4.429
4.120
4.102
4.085
4.067
3.852
3.841
3.833
3.823
3.816
3.805
3.709
3.686
1.890
1.885
1.872
1.866
1.854
1.847
1.165
1.148
1.130
1.056
1.037
1.019
0.073
-0.00
BRUKER
Current Data Parameters
NAME 2009-09-30 zhengke-ZK-206A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090930
Time 22.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300053 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
EtO
Et
OH
O
O
anti-5ad
9
8
7
6
5
4
3
2
1
0
ppm
1.95
1.00
2.02
0.96
1.96
1.07
0.99
2.15
3.03
3.07


7.984
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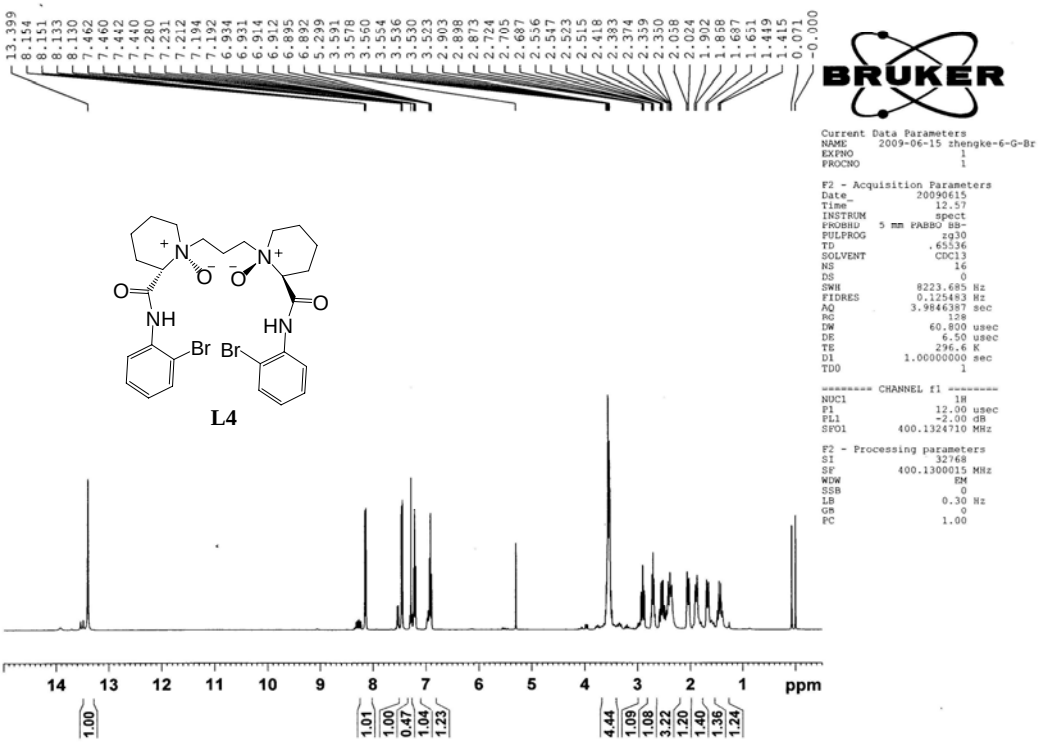
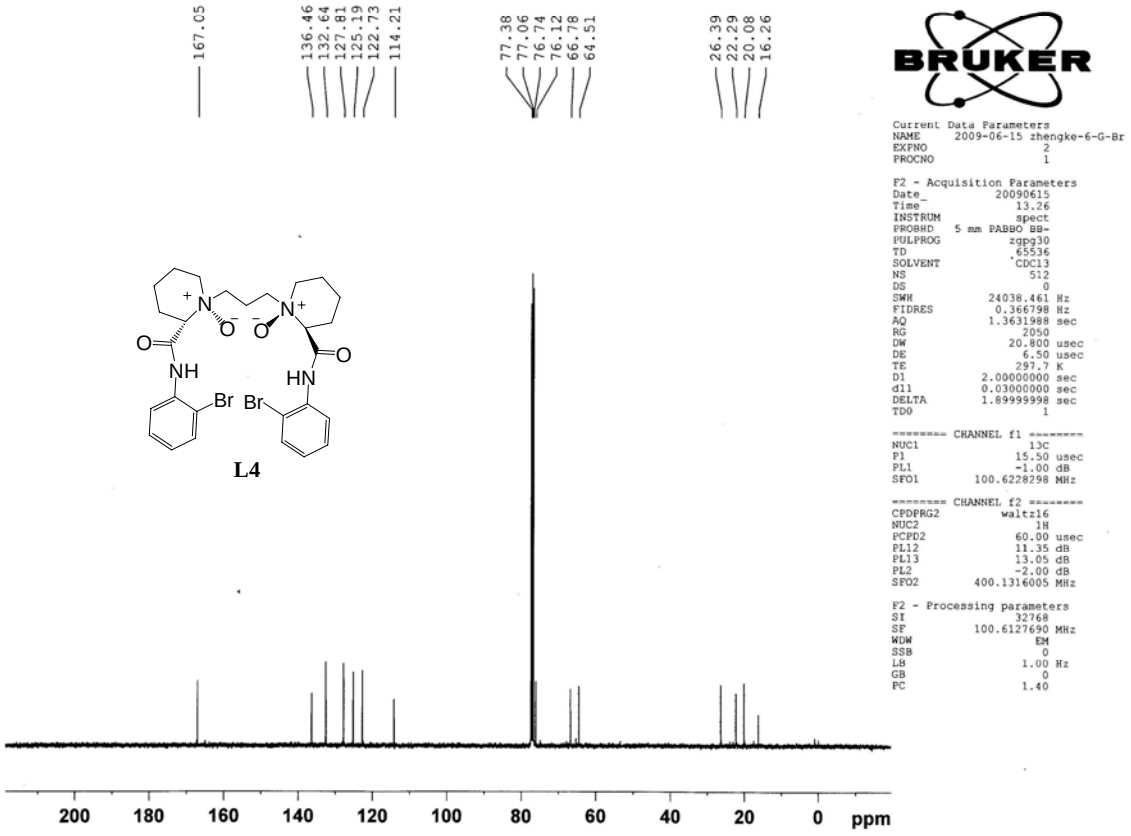
7.929
7.911
7.593
7.590
7.574
7.505
7.498
7.486
7.479
7.467
7.460
7.265
4.215
4.197
4.120
4.102
4.084
4.066
3.838
3.830
3.820
3.817
3.803
3.701
1.889
1.884
1.871
1.866
1.853
1.847
1.651
1.219
1.201
1.183
1.165
1.147
1.129
1.056
1.037
1.019
0.948
0.929
0.910
0.070
BRUKER
Current Data Parameters
NAME 2009-09-30 zhengke-ZK-204D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090930
Time 22.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300073 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
O
Et
EtO
OH
O
5ad
9
8
7
6
5
4
3
2
1
0
ppm
3.55
1.79
3.56
0.76
1.00
1.56
2.04
1.86
1.10
0.76
1.56
2.01
2.40
3.05
3.20
2.48


13.399
8.154
8.151
8.133
8.130
7.462
7.460
7.442
7.440
7.280
7.231
7.212
7.194
7.192
6.934
6.931
6.914
6.912
6.895
6.892
5.299
3.591
3.578
3.560
3.554
3.536
3.530
3.523
2.903
2.898
2.873
2.724
2.705
2.687
2.556
2.547
2.523
2.515
2.418
2.383
2.374
2.359
2.350
2.058
2.024
1.902
1.858
1.687
1.651
1.449
1.415
0.071
-0.000
BRUKER
Current Data Parameters
NAME 2009-06-15 zhengke-6-G-Br
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090615
Time 12.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300015 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
NH
Br
Br
HN
O
L4
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.00
0.47
1.04
1.23
4.44
1.09
1.08
3.22
1.20
1.40
1.36
1.24


167.05
136.46
132.64
127.81
125.19
122.73
114.21
77.38
77.06
76.74
76.12
66.78
64.51
26.39
22.29
20.08
16.26
BRUKER
Current Data Parameters
NAME 2009-06-15 zhengke-6-G-Br
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090615
Time 13.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

L4

(G) References

1. B. Qin, X. H. Liu, J. Shi, K. Zheng, H. T. Zhao, X. M. Feng, *J. Org. Chem.* 2007, **72**, 2374-2378, and references therein.

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