Asymmetric synthesis of corsifuran A by an enantioselective reduction

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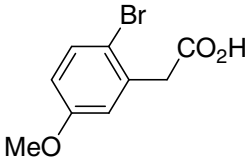
Supplementary Information

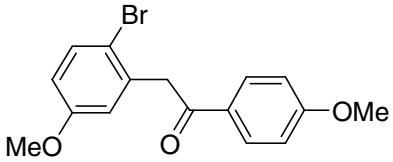
Contents

Preparation of 2-bromo-5-methoxyphenylacetic acid 4[1]



Bromine (4.31 g, 1.39 mL, 27 mmol) was added at 0 ˚C to a solution of 3-methoxyphenylacetic acid (4.50 g, 27 mmol) in dichloromethane (25 mL) and stirred at room temperature for 24 hrs. The dark red solution was discoloured with sodium thiosulfate solution (1M, 15 mL), washed with water (20 mL) and separated. The aqueous layer was extracted into dichloromethane (2 × 25 mL) and the combined organic layers dried over $MgSO_4$, filtered and evaporated to dryness to give compound **4** (6.62 g, 100%) as a white solid. mp 114 ^oC (lit.[1] 114 – 115 ^oC); IR 2974 (brd), 1690, 1596, 1571, 1473 cm^{-1}; ^1H NMR (250 MHz; $CDCl_3$) δ_H 7.75 – 8.30 (1H, brd), 7.45 (1H, d, *J* 9.0), 6.83 (1H, d, *J* 3.0), 6.70 (1H, dd, *J* 9.0, 3.0), 3.55 (3H, s), 3.50 (2H, s); ^{13}C NMR (63 MHz; $CDCl_3$) δ_C 177.1, 158.9, 134.3, 133.4, 117.3, 115.1, 114.8, 55.5, 41.6; HRMS calcd for $C_9H_9O_3{}^{79}Br$ [M^+] 243.9735, found 243.9739; MS (EI) 246 (25), 244 (25), 201 (20), 199 (20), 166 (18), 165 (100), 121 (20), 109 (15).

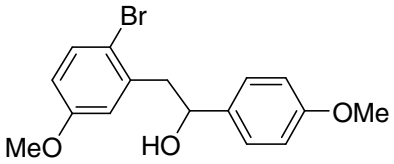
Preparation of 1-(4-methoxyphenyl)-2-(2-bromo-4-methoxyphenyl)ethanone 3[2]



2-Bromo-5-methoxyphenyl acetic acid **4** (312 mg, 1.3 mmol) and thionyl chloride (155 mg, 1.3 mmol) were combined in a round-bottomed flask and refluxed for 30 mins. The reaction was then evaporated under a reduced pressure to dryness, re-dissolved in dichloromethane (10 mL) and cooled to -5 ˚C. Anisole (140 mg, 1.3 mmol) was added followed by the addition of powdered aluminium trichloride (173 mg, 1.3 mmol) portion-wise. The reaction was stirred for 30 mins and then poured into a small beaker of ice, the organic layer separated and the aqueous layer extracted with dichloromethane (2 × 10 mL). The combined organic fractions were washed with 1M HCl (30 mL) and dried over $MgSO_4$. After purification by chromatography on silica gel, eluting with 20% EtOAc / 80% petroleum ether 40 – 60), compound **3** was obtained as an off white solid (0.380 g; 89%); mp 113 – 114 ^oC (lit.[2] 114 ^oC); IR 1672, 1596, 1574, 1459 cm^{-1}; ^1H NMR (250 MHz; $CDCl_3$) δ_H 8.08 – 8.00 (2H, m),

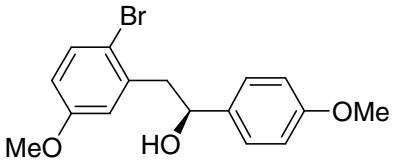
7.48 (1H, d, *J* 8.8), 6.99 – 6.93 (2H, m) 6.78 (1H, d, *J* 2.8), 6.41 (1H, dd, *J* 8.0, 2.8), 4.38 (2H, s), 3.90 (3H, s) 3.78 (3H, s); ^{13}C NMR (63 MHz; $CDCl_3$) δ_C 194.9, 163.7, 158.9, 136.2, 133.2, 130.7, 129.6, 117.2, 115.4, 114.5, 113.9, 55.5, 55.4, 45.6; HRMS calcd for $C_{16}H_{15}O_3{}^{79}Br$ [M^+] 334.0205, found 334.0207; MS (EI) 336 (1), 334 (1), 255 (32), 135 (100), 107 (6), 92 (15), 77 (24), 64 (6).

Preparation of racemic 1-(2-bromo-5-methoxy-phenyl)-2-(3-methoxy-phenyl)-ethanol 2



Sodium borohydride (0.33 g, 8.9 mmol) was added to ethanol (25 mL) and stirred at room temperature. Ketone **3** (1.50 g, 4.4 mmol) was added drop-wise with stirring and the reaction was left for 1 h at room temperature. Water (10 mL) was added, the mixture extracted with CH_2Cl_2 (3 × 30 cm^3), the organic layer washed with water (2 × 200 mL) and dried over $MgSO_4$. The solution was filtered and concentrated under reduced pressure to provide the desired product **2** (1.38 g, 92%); Analytical data was as reported below.

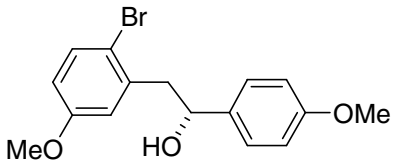
Preparation of 2*S*-(2-bromo-5-methoxy-phenyl)-1-(4-methoxy-phenyl)-ethanol 2



Trimethyl borate (0.2 5 mL, 0.2 mmol) was added to a solution o f the (1*R*, 2*S*)-1-amino-indan-2-ol (3 mg, 0.2 mmol) in THF (3 mL) and stirred at room temperature under a nitrogen atmosphere for 30 min. BH_3.DMS complex (0.17 mL, 2.0 mmol) was added, the reaction stirred for 30 min, then ketone **3** (0.737 g, 2.19 mmol) in THF (2 mL) added via cannula. The reaction mixture was stirred for a further 30 min at room temperature then quenched with methanol (5 mL). Water (10 mL) was added and the solvent removed under reduced pressure. The mixture was extracted with CH_2Cl_2 (3 × 10 mL), the organic phase washed with 1M HC1 (30 mL), water (30 mL) and dried over $MgSO_4$. Filtration, and removal of the solvent under reduced pressure

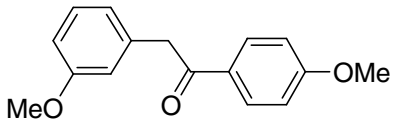
produced the crude product (*S*)-**2** in 76% ee. The alcohol was recrystallised to 99% ee from petroleum ether (40 – 60) / CH_2Cl_2; mp 76-77 ºC, $[\alpha]_D$ -11.2 (*c* 1, $CHCl_3$); ee 99% [Kromasil 3-CelluCoat 8% iso-propanol in hexane @ 1.00 mL / min, 10.04 and 15.36 mins (major)]; IR 3361, 2931, 1614, 1571, 1514, 1454 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ_H 7.45 (1H, d, *J* 9.1), 7.36 – 7.29 (2H, m), 6.90 – 6.82 (2H, m), 6.71 (1H, d, *J* 2.8), 6.67 (1H, dd, *J* 9.1, 2.8), 4.95 (1H, dd, *J* 4.9, 8.5), 3.80 (3H, s), 3.72 (3H, s), 3.13 (1H, dd, *J* 4.9, 13.7), 3.02 (1H, dd, *J* 8.5, 13.7), 1.95 (1H, brd s); ^{13}C NMR (125 MHz; $CDCl_3$) δ_C 159.1, 158.7, 138.6, 136.0, 133.3, 127.0, 117.4, 115.2, 114.1, 113.8, 73.1, 55.4, 55.3, 46.4; HRMS calcd for $C_{16}H_{17}O_3{}^{79}Br$ [M^+] 336.0361, found 336.0367; MS (EI) 338 (1), 336 (1), 202 (13), 200 (14), 137 (100), 109 (20), 94 (15), 77 (30).

Preparation of 2*R*-(2-bromo-5-methoxy-phenyl)-1-(4-methoxy-phenyl)-ethanol 2.



Using the above method with (1*S*, 2*R*)-catalyst with ketone **3** (2.66 g, 7.92 mmol), gave alcohol (*R*)-**2** (2.38 g, 89%) in 78% ee**.** The ee was increased to 99% by repeated recrystallisation from petroleum ether (40 – 60) / CH_2Cl_2; mp 77-78 ºC, $[\alpha]_D$ +15.9 (*c* 1, $CHCl_3$); ee 99% [Kromasil 3-CelluCoat 8% iso-propanol in hexane @ 1.00 mL / min, 9.90 (major) and 15.84 mins]; remainder of analytical data the same as for (*S*)-**2**.

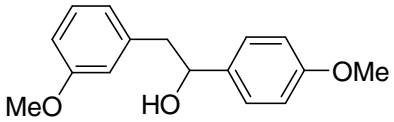
Preparation of 3, 4-dimethoxydeoxybenzoin 8[3]



3-Methoxyphenylacetic acid (4.00 g, 24 mmol) and thionyl chloride (2.86 g, 23 mmol) were combined in a round-bottomed flask and refluxed for 30 mins. The reaction was then evaporated under a reduced pressure to dryness, re-dissolved in dichloromethane (40 mL) and cooled to -5 ˚C. Anisole (2.60 g, 24 mmol) was added followed by the addition of powdered aluminium trichloride (3.20 g, 24 mmol) portion-wise. The reaction was stirred for 30 mins and then poured into a small beaker of ice, the organic layer separated and the aqueous layer extracted with

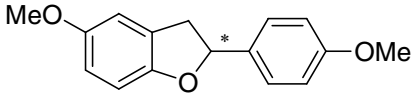
dichloromethane (2 × 40 mL). The combined organic fractions were washed with 1M HCl (40 mL) and dried over $MgSO_4$. After purification by chromatography on silica gel, eluting with 20% EtOAc / 80% petroleum ether 40 – 60), compound **8** (3.90 g; 65%) was obtained as an off white solid; mp 44 – 45 ^oC (lit.[3] 38 – 40 ^oC); IR 1672, 1592, 1492 cm^{-1}; ^1H NMR (250 MHz; $CDCl_3$) δ_H 8.02 – 7.95 (2H, m), 7.25 – 7.17 (1H, m), 6.95 – 6.74 (5H, m), 4.22 (2H, s), 3.85 (3H, s) 3.80 (3H, s); ^{13}C NMR (125 MHz; $CDCl_3$) δ_C 195.9, 163.3, 159.6, 136.3, 130.8, 129.4, 121.6, 114.9, 113.6, 112.1, 55.2, 54.9, 45.1; HRMS calcd for $C_{16}H_{16}O_3$ [M^+], 256.1099, found 256.1093; MS (EI) 256 (7), 136 (11), 135 (100), 121 (3), 107 (6), 92 (11), 77 18).

Preparation of 1-(4-methoxy-phenyl)-2-(3-methoxy-phenyl)-ethanol 7



Sodium borohydride (0.73 g, 19 mmol) was added to ethanol (30 mL) and stirred at room temperature. Ketone **9** (2.50 g, 9.7 mmol) was added drop-wise with stirring and the reaction was left for 1 h at room temperature. Water (10 mL) was added, the mixture extracted with CH_2Cl_2 (3 × 30 mL), the organic layer washed with water (2 × 200 mL) and dried over $MgSO_4$. The solution was filtered and concentrated under reduced pressure to provide the desired product **7** (2.20 g, 88%); Found: C, 74.03; H, 7.16%. $C_{16}H_{18}O_3$ requires C, 64.39, H, 7.02; IR 3425, 2936, 1610, 1584, 1511 cm^{-1}; ^1H NMR (250 MHz; $CDCl_3$) δ_H 7.35 –7.21 (3H, m), 6.95 – 6.85 (2H, m), 6.81 – 6.72 (4H, m), 4.85 (1H, td, *J* 4.7, 2.1), 3.80 (3H, s), 3.75 (3H, s), 3.05 – 2.91 (2H, m), 2.63 (1H, d, *J* 2.1); ^{13}C NMR (63 MHz, $CDCl_3$) δ_C 159.7, 159.1, 139.8, 136.0, 129.5, 127.2, 121.8, 115.1, 113.8, 112.1, 74.9, 55.3, 55.2, 46.0; HRMS calcd for $C_{16}H_{18}O_3$ [M^+] 258.1256, found 258.1252; MS (EI) 258 (4), 241 (15), 240 (23), 165 (10), 122 (100), 121 (55), 109 (55), 94 (47), 91 (52), 79 (66), 77 (95).

General procedure for preparation of Corsifuran A 1 by palladium-catalyzed intramolecular C-O bond formation



2-Bromo-5-methoxy-phenyl-1-(4-methoxy-phenyl)-ethanol **2** (400 mg, 1.8 mmol) and base (1.5 equiv.), in dry toluene (5 mL) were added to a flame dried 50 cm^3 two-necked round bottomed flask fitted with a condenser under a N_2 atmosphere, containing the palladium source (3 mol %) and ligand (3.5 mol %) in toluene (5 mL). The reaction was refluxed for 24 h then cooled to room temperature, diluted with diethyl ether (15 mL) and filtered through Celite. The solvent was evaporated under a reduced atmosphere to give a dark yellow solution. Column chromatography on silica gel eluting with 20 % EtOAc / 80 % petroleum ether 40 – 60 gave natural product **1** as the first fraction, inseparable alcohols **2** and **7** as the second fraction, and inseparable ketones **3** and **8** as the third fraction. Conversion and yields were as reported in the manuscript table. Analytical data was as reported below.

Preparation of (*R*)-Corsifuran A 1 by copper-catalyzed intramolecular C-O bond formation

(*R*)-2-Bromo-5-methoxy-phenyl-1-(4-methoxy-phenyl)-ethanol **2** (500 mg, 1.48 mmol, 100% ee) was added to a stirred solution of sodium hydride (60% dispersion in mineral oil; pre-washed with petroleum ether; 44 mg, 1.85 mmol) in toluene (2 mL). The solution was heated to 40 ˚C for 15 mins and cooled back to room temperature. Copper^{I} chloride[1] (7 mg, 0.074 mmol) and ethyl acetate (1-2 drops) in toluene (2 mL) were added and the reaction was heated at reflux for 24 h. After cooling to room temperature, the solution was quenched with water (20 mL) and filtered through Celite with ether washings (2 × 10 mL). The organic layer was separated, the aqueous layer extracted with ether (2 × 20 mL) and the combined organic fractions washed sequentially with 1 M HCl (30 mL), saturated $NaHCO_3$ (30 mL), saturated NaCl (30 mL) and water (30 mL) and dried over $NaSO_4$. After flash chromatography on silica gel, eluting with 15 % EtOAc / 75 % petroleum ether 40-60, (*R*)-corsifuran A **1** was obtained in a 0.288 g, 76% yield, >99 % ee; (5 m octakis(2, 6-O-Me-3-O-Pe)-gamma-cyclodextrin capillary, hydrogen as the carrier gas at 2 mL per minute; Injector temp 250 ºC, flame ionisation detector at 250 ºC, isothermal at 160 ºC; mp 61 ºC; $[\alpha]_D$ -11.5 (*c* 1, $CHCl_3$) [lit.[4] -11.1 (*c* 0.1 $CDCl_3$)]; IR 2937, 1590, 1486 cm^{-1}; ^{1}H NMR (250 MHz; $CDCl_3$) δ_H 7.33 – 7.22 (2H, m), 6.86 – 6.78 (2H, m), 6.73 – 6.56 (4H, m), 5.62 (1H, app t, *J* 8.8), 3.75 (3H, s), 3.70 (3H, s), 3.45 (1H, dd, *J* 8.8, 16.5), 3.10 (1H, dd, *J*

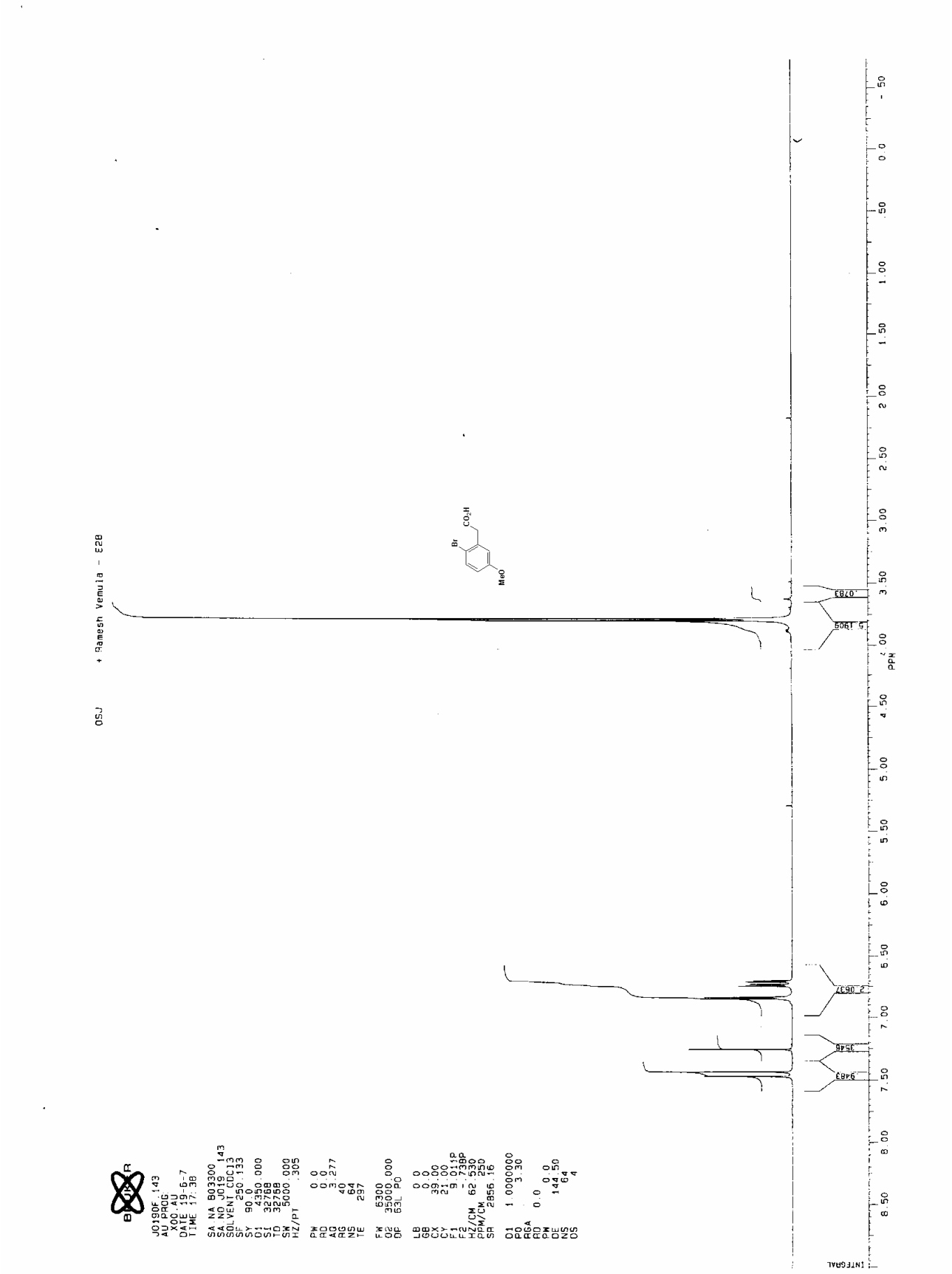
[1] Purified reagent plus, greater than or equal to 99%, Sigma Aldrich.

8.8, 16.5); ^{13}C NMR (63 MHz; $CDCl_3$) δ_C 159.4, 154.2, 153.7, 133.9, 127.7, 127.3, 113.9, 113.0, 111.1, 109.2, 84.2, 56.0, 55.3, 38.7; HRMS calcd for $C_{16}H_{16}O_3$ $[M^+]$ 256.1099, found 256.1103; MS (EI) 256 (26), 241 (6), 213 (6), 198 (7), 181 (11), 152 (16), 115 (26).

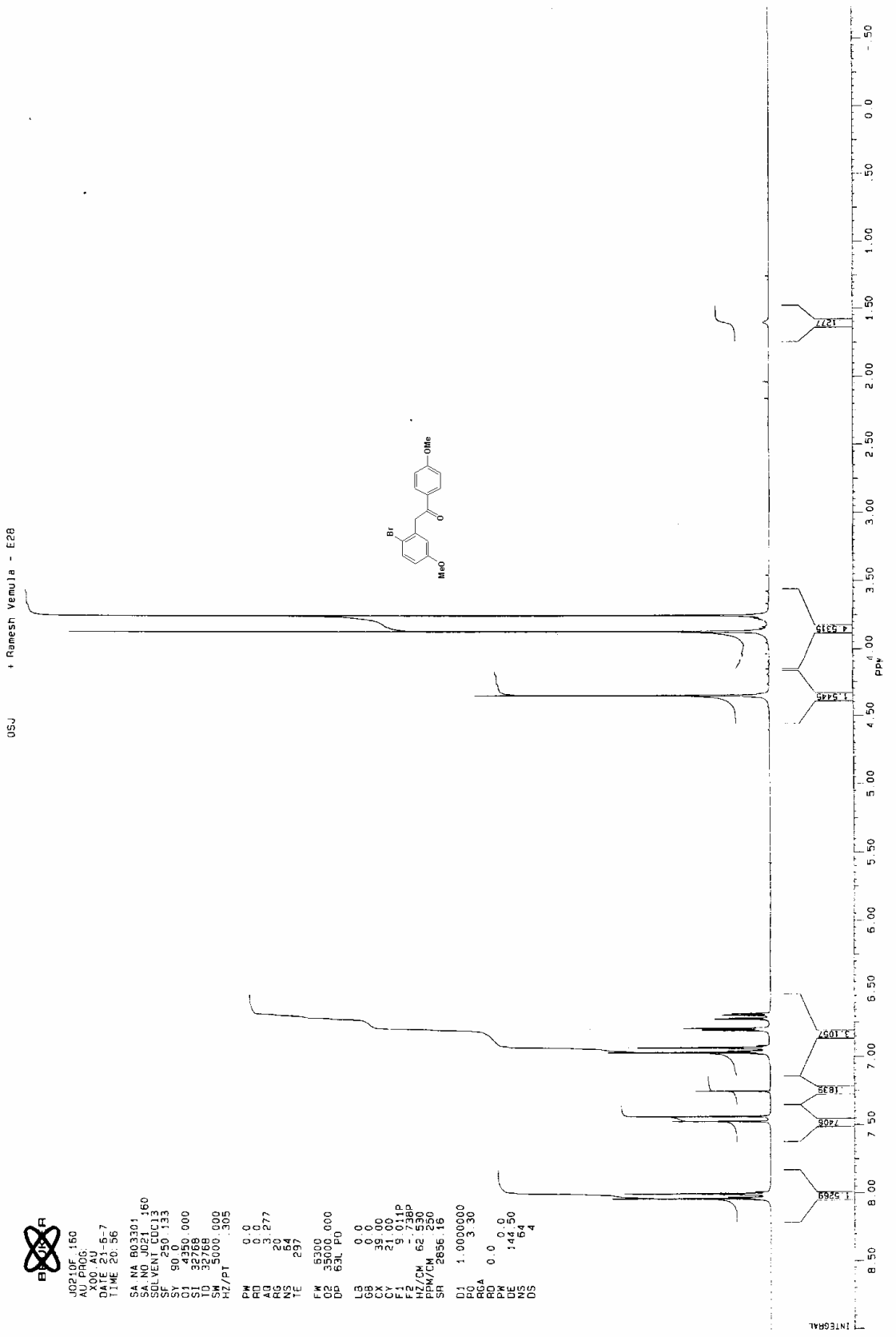
(*S*)-Corsifuran A **1; >**99% ee, (5 m octakis(2, 6-O-Me-3-O-Pe)-gamma-cyclodextrin capillary, hydrogen as the carrier gas at 2 mL per minute; Injector temp 250 ºC, flame ionisation detector at 250 ºC, isothermal at 160 ºC; mp 61 – 62 ºC; $[\alpha]_D$ +15.5 (*c* 1, $CHCl_3$).

Racemic corsifuran was prepared in an identical manner starting from racemic alcohol **2**.

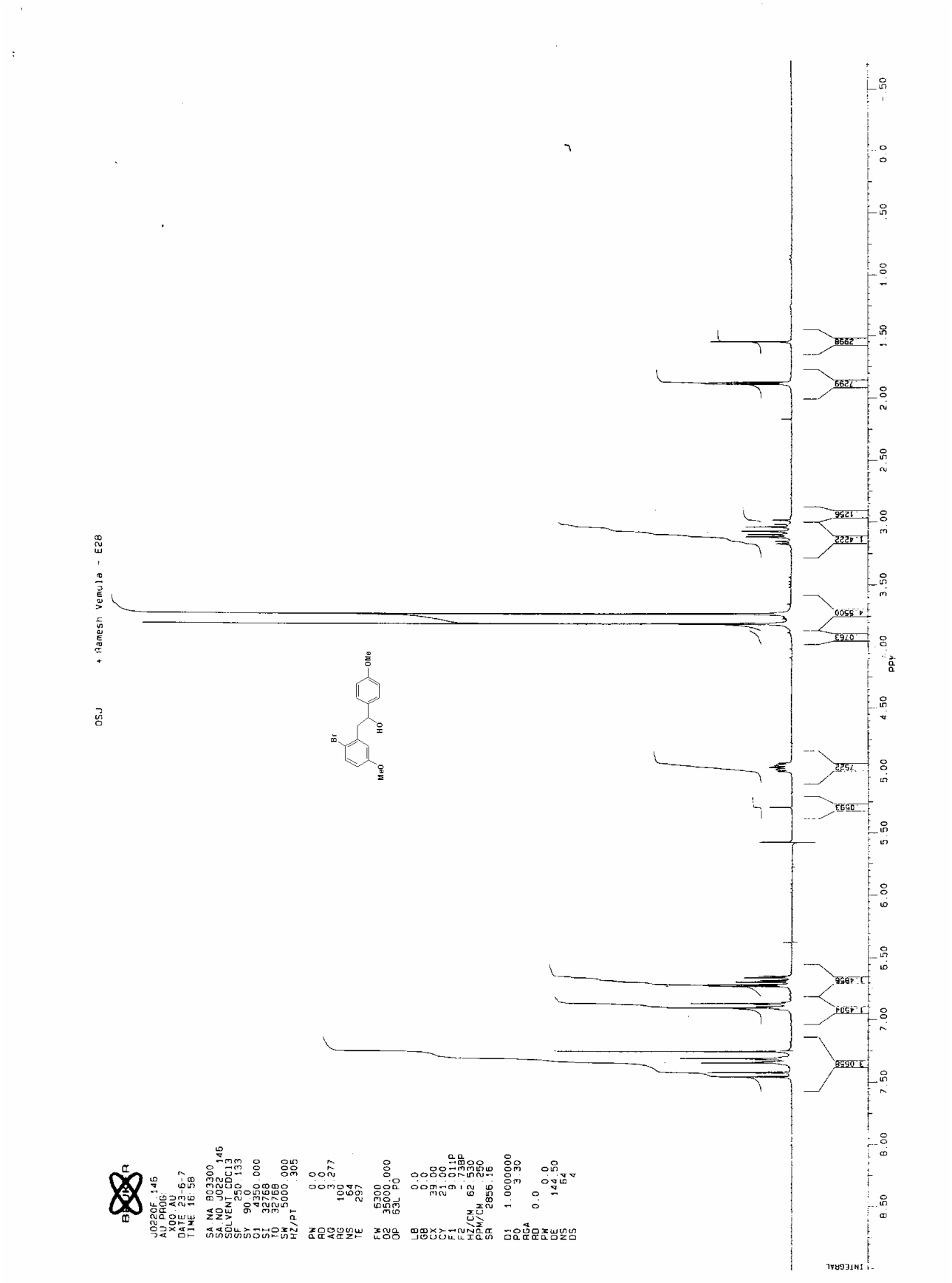
^{1}H NMR Spectrum of compound 4



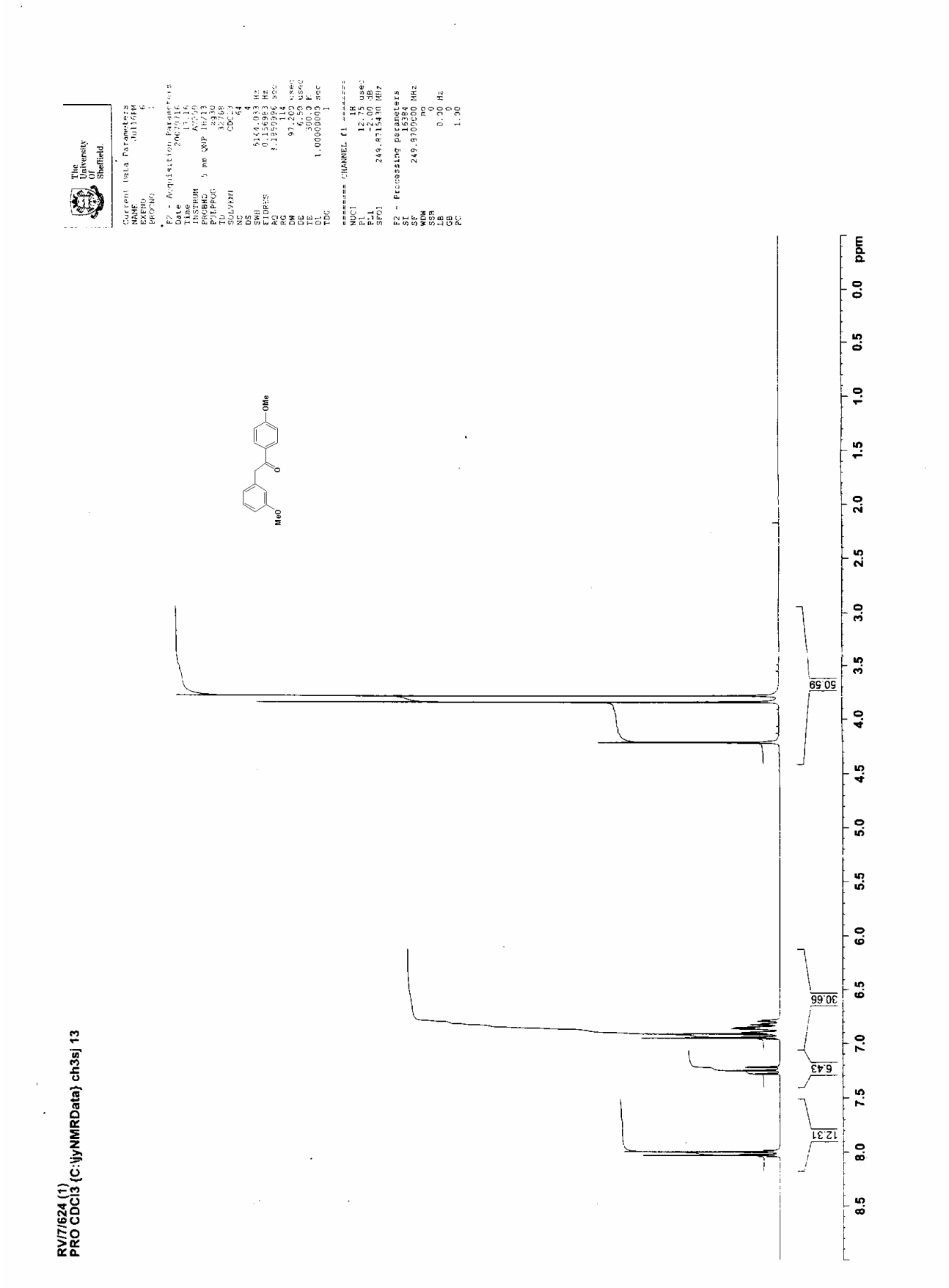
^{1}H NMR Spectrum of compound 3



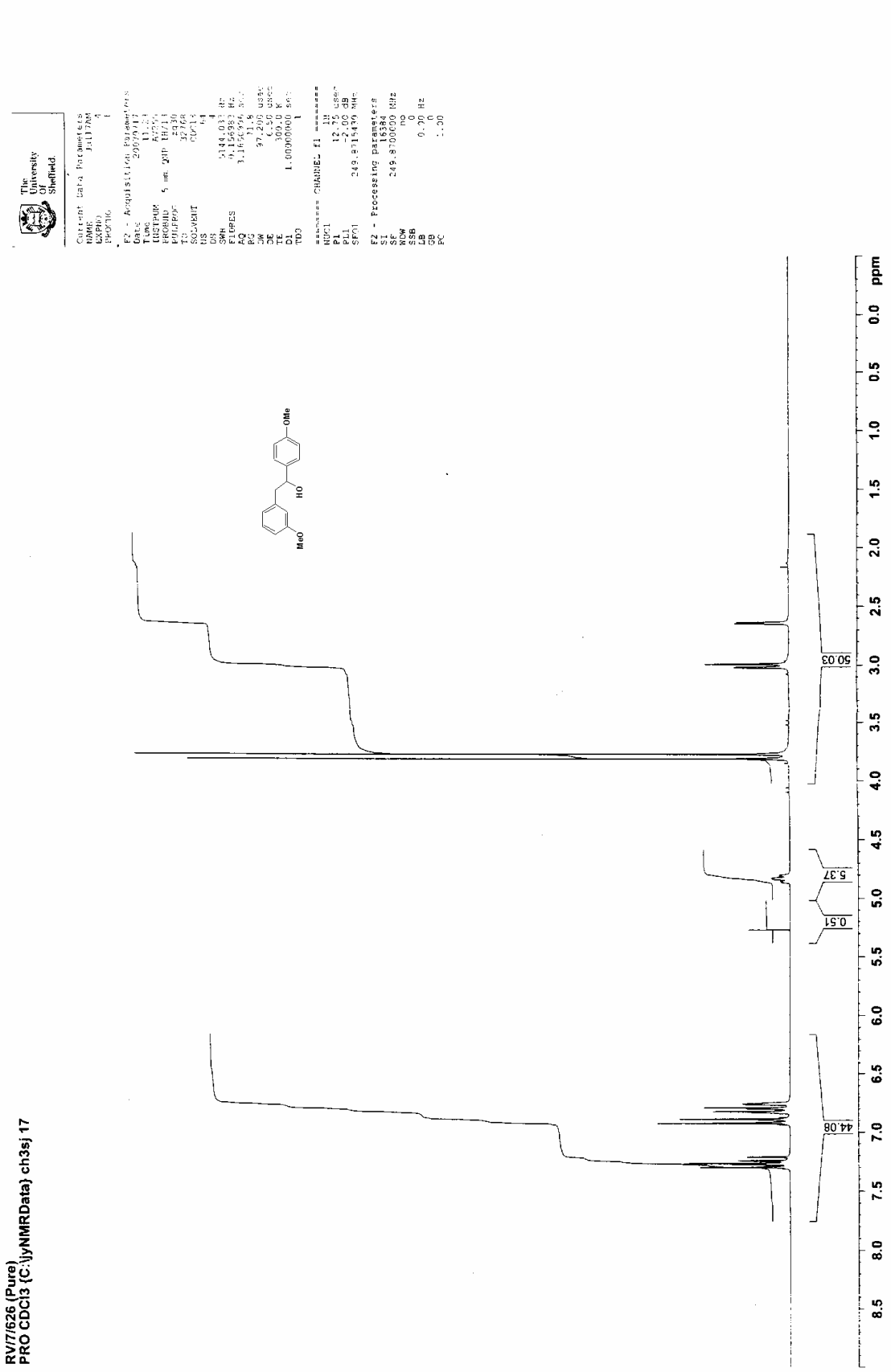
^{1}H NMR Spectrum of compound 2



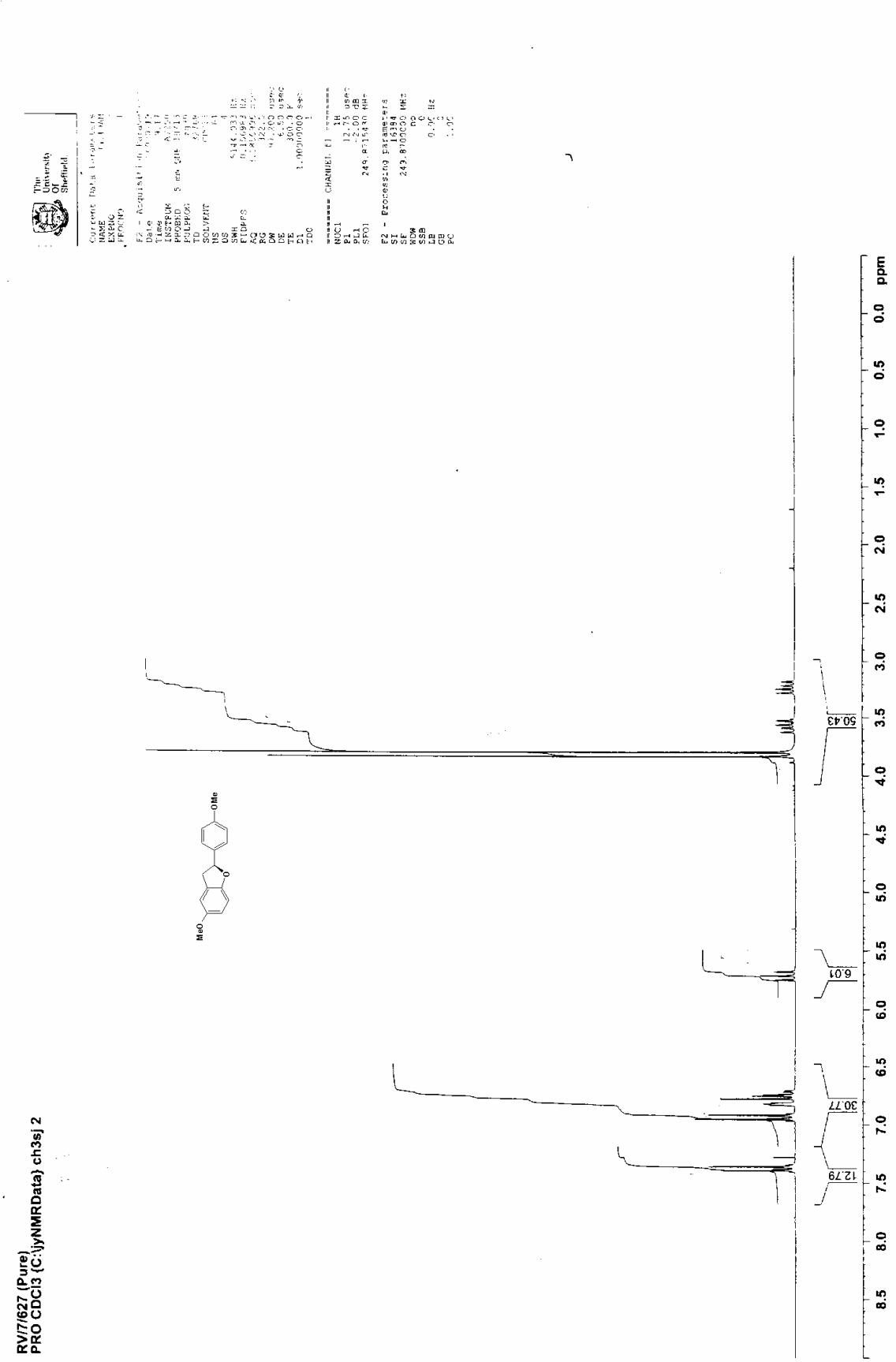
^{1}H NMR Spectrum of compound 8



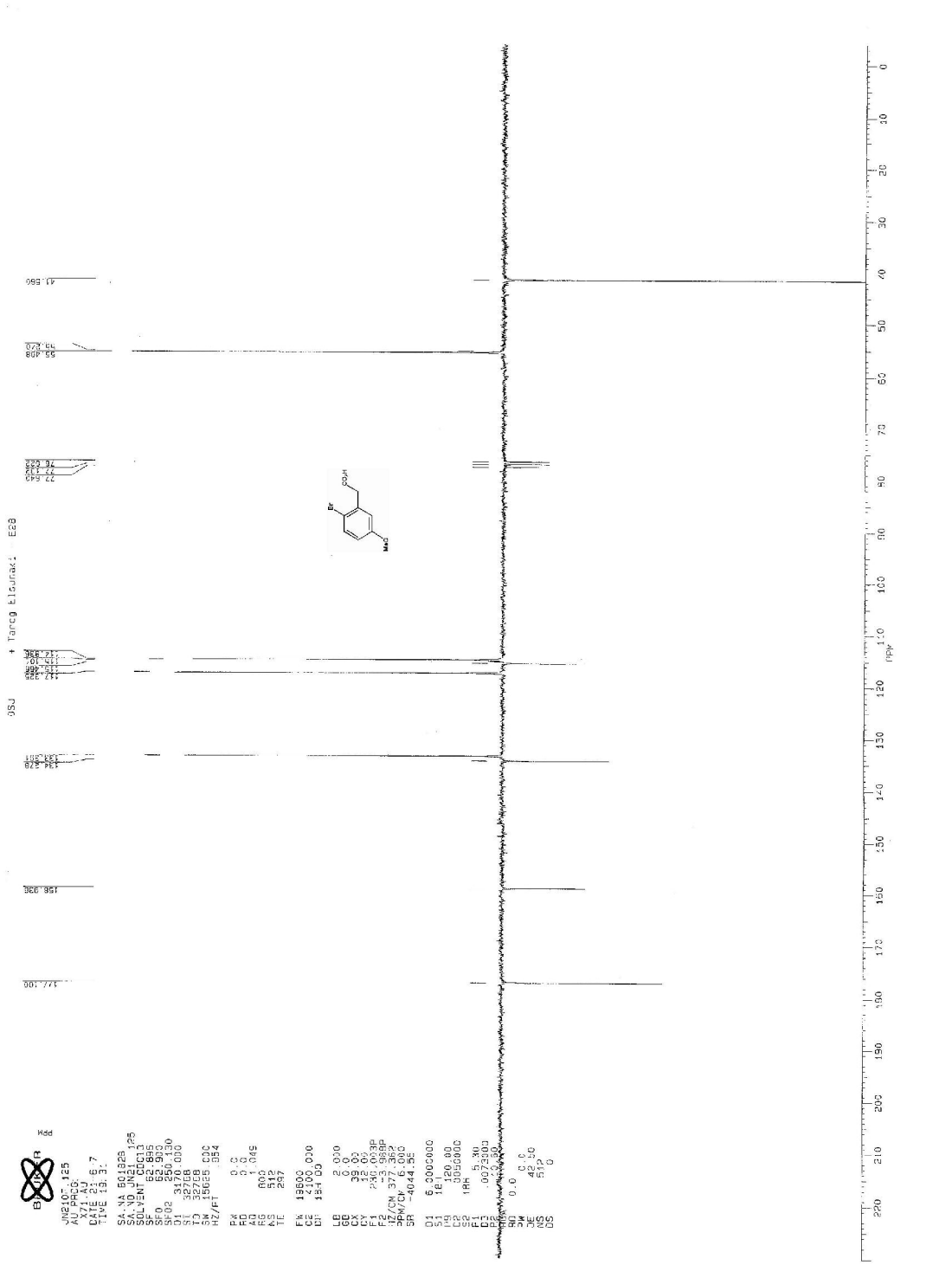
^{1}H NMR Spectrum of compound 7



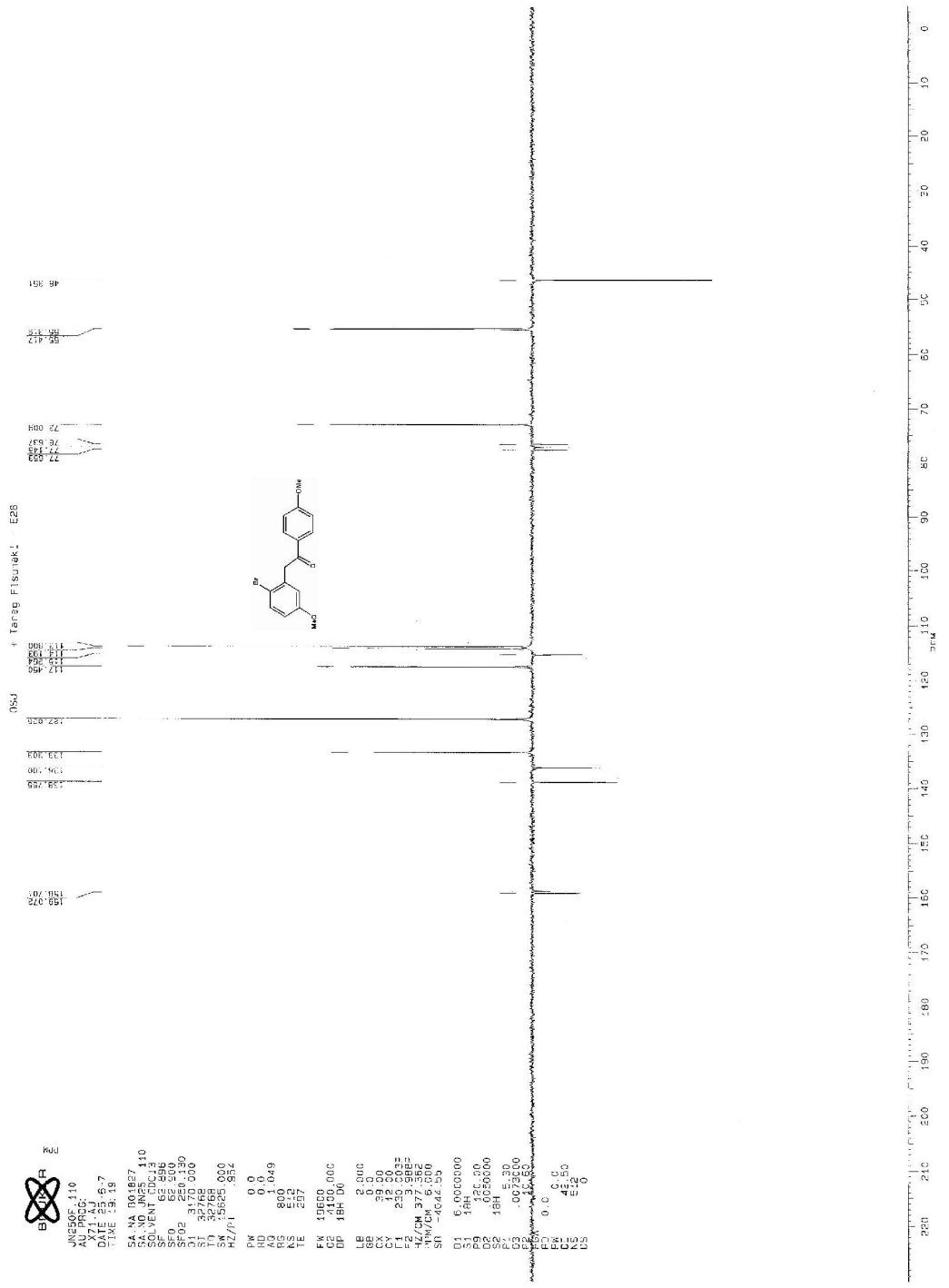
^{1}H NMR Spectrum of compound 1



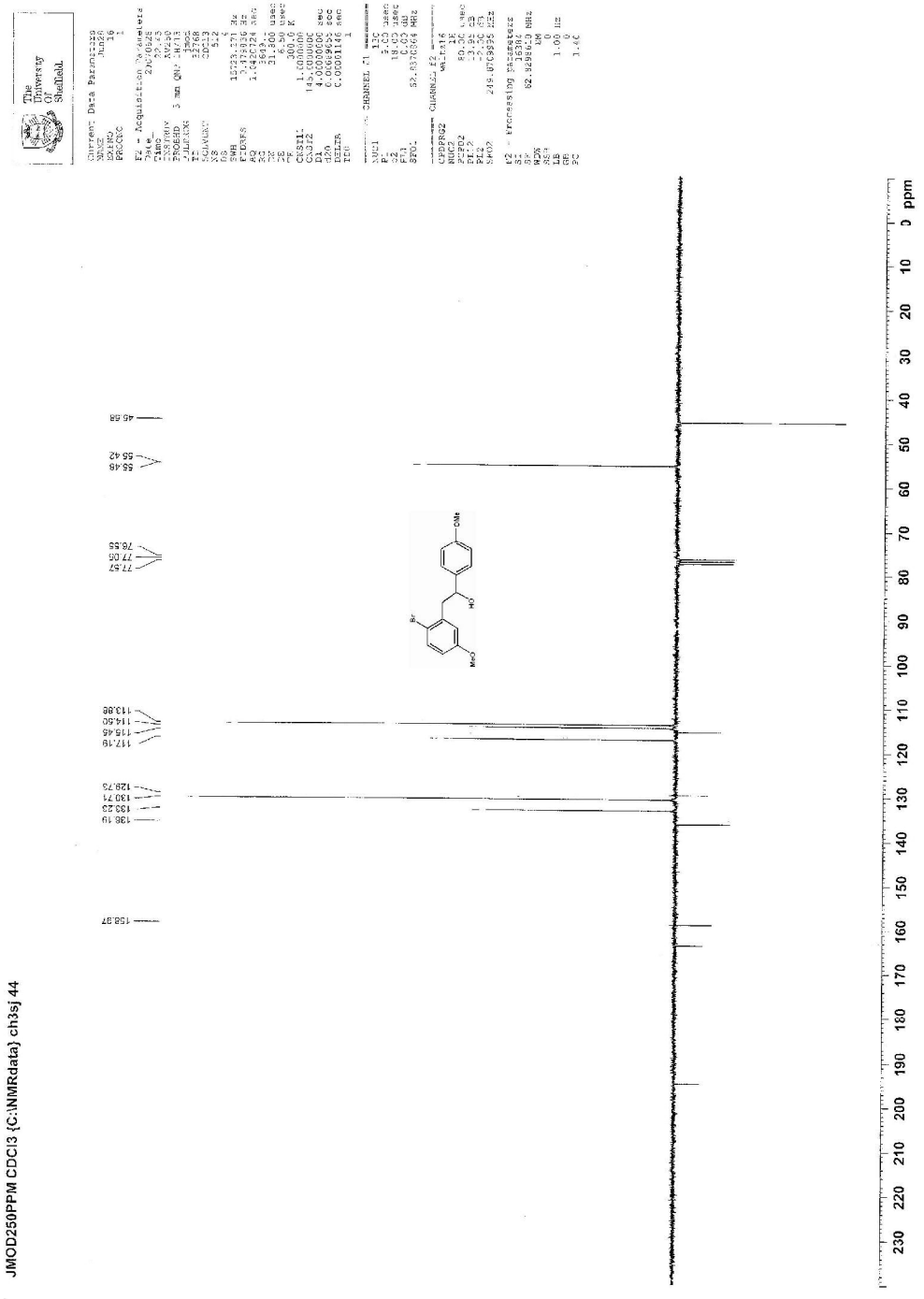
^{13}C NMR Spectrum of compound 4



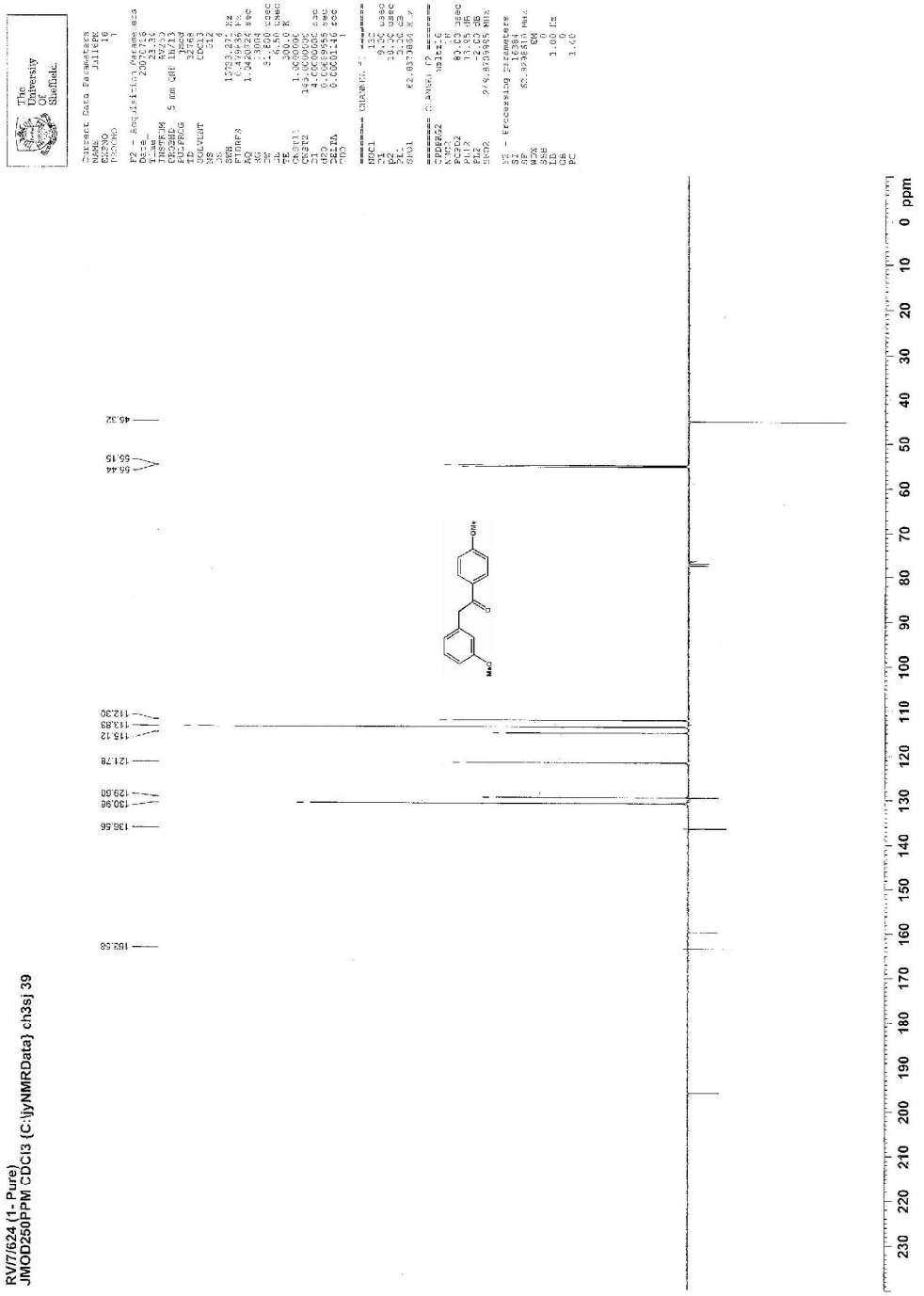
^{13}C NMR Spectrum of compound 3



^{13}C NMR Spectrum of compound 2



^{13}C NMR Spectrum of compound 8



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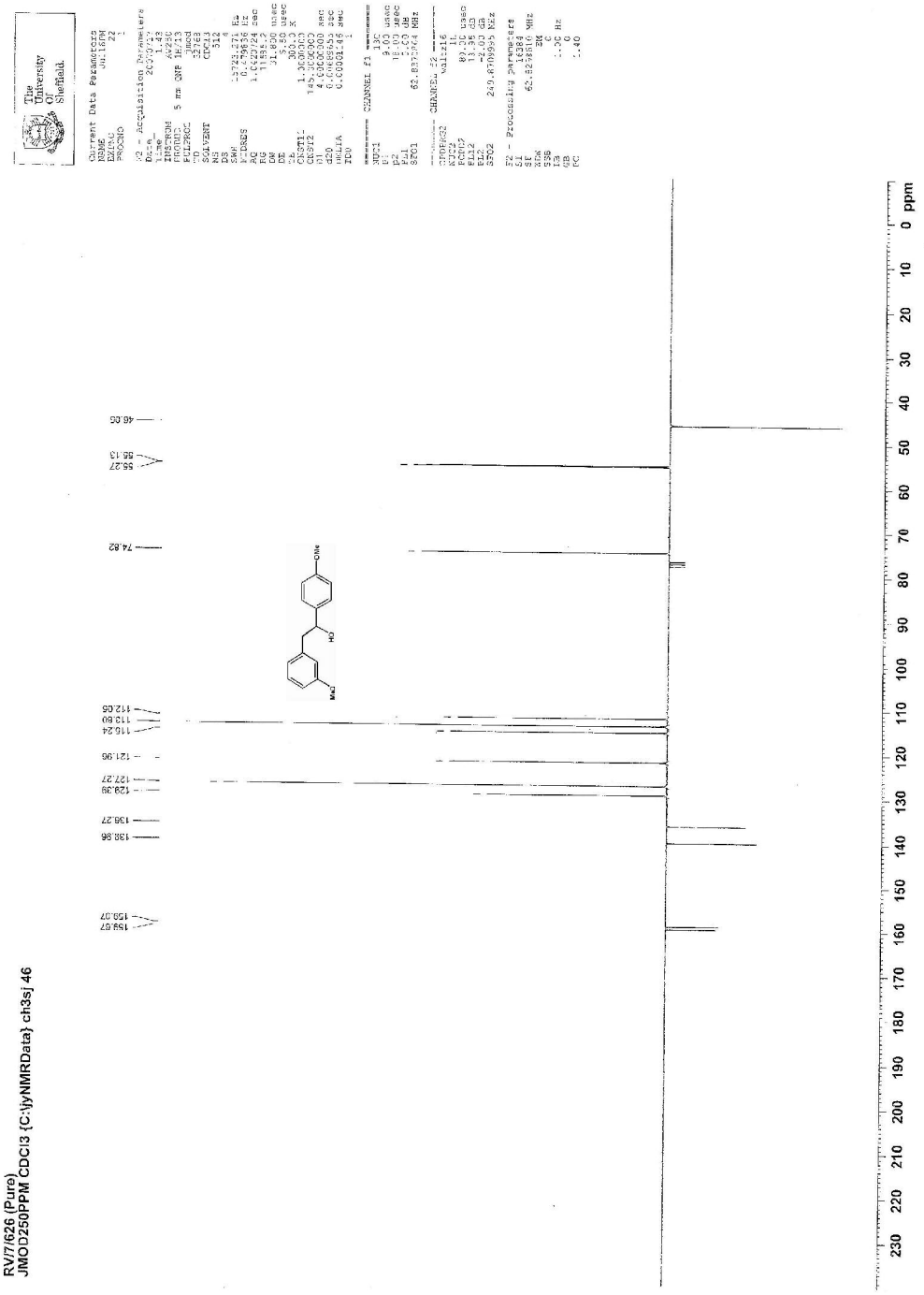
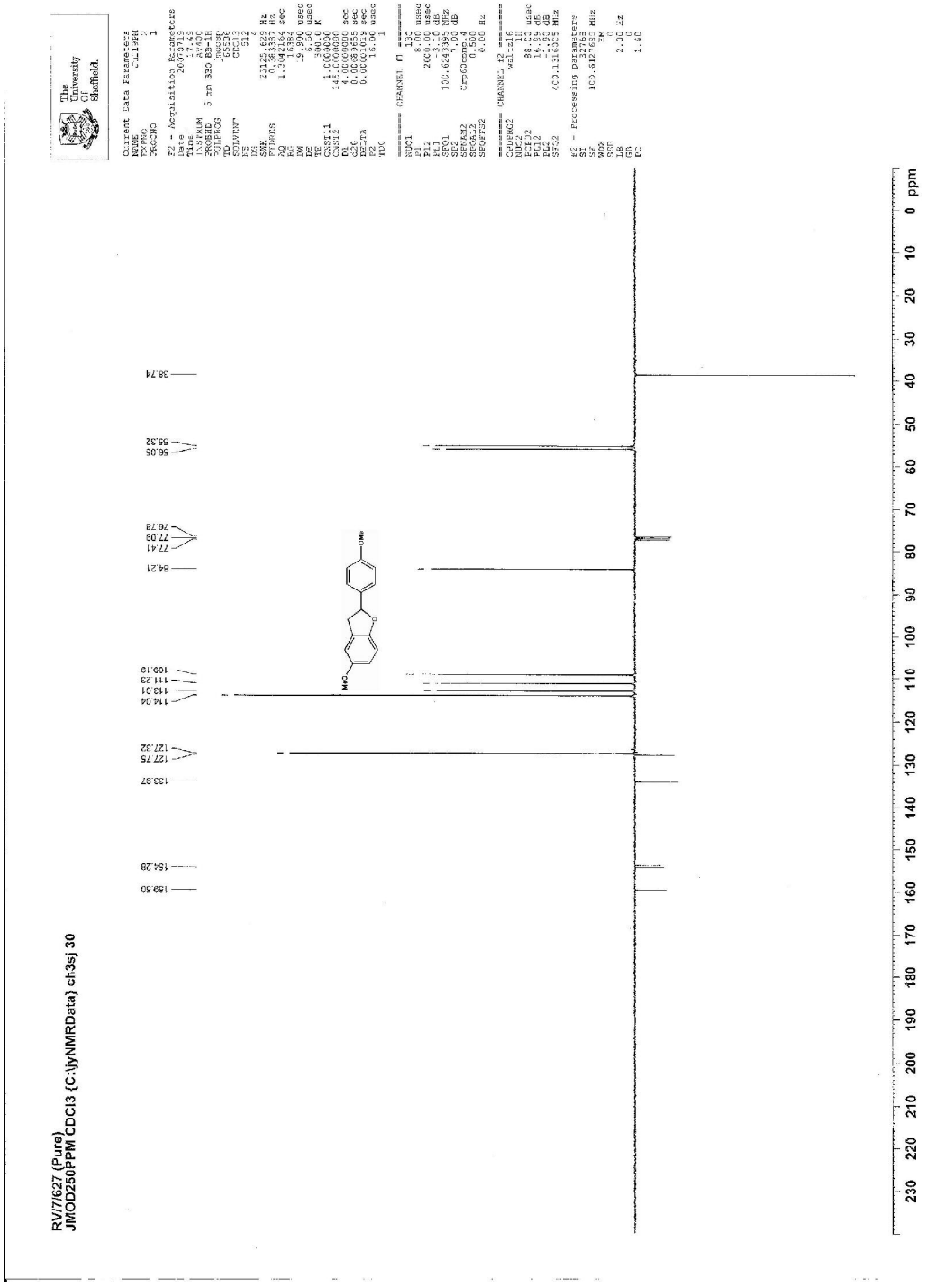


^{13}C NMR Spectrum of compound 1



HPLC Chromatogram of (*S*)-2 crude reaction mixture

27/06/2007 16:16 Chromatogram RV-7-612(rc) Page 1 of 1

Created	: 27/06/2007 14:59:56	By	: Clarity
Project	: Work3	Report Style	: Full
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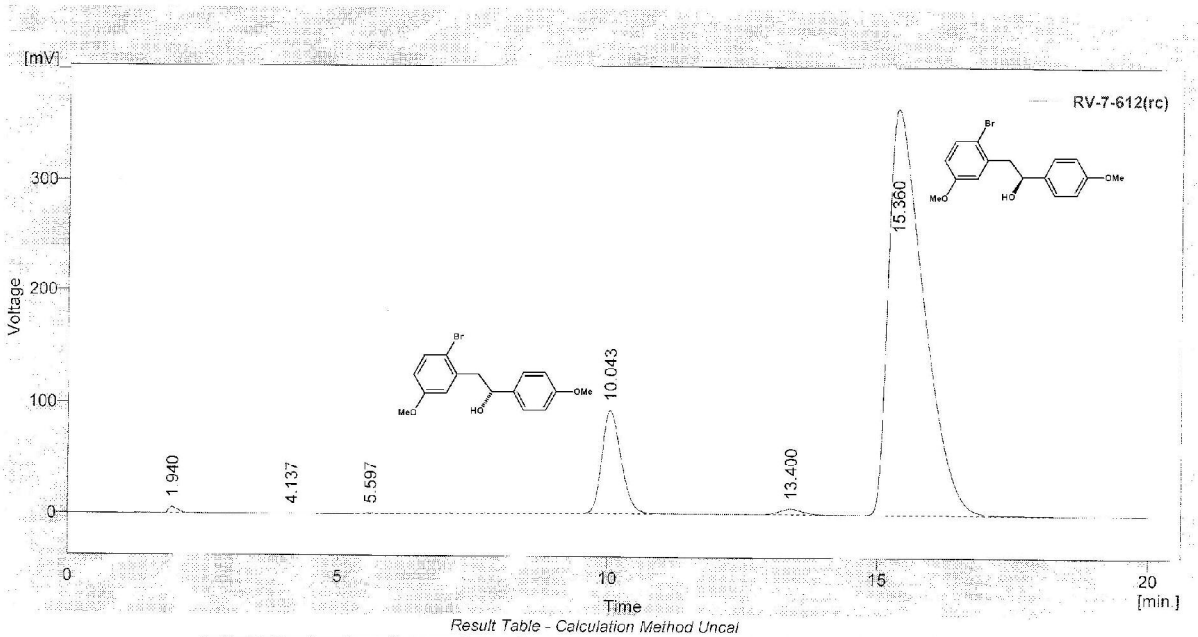
Calibration	: RV-7-612(rc)	Chromatogram	: DATA\RV-7-612(rc)

Method	: FMG	By	: Clarity
Description	:		
Created	: 24/08/2004 16:25	Modified	: 27/06/2007 14:59
Column	: Kromasil 3-CelluCoat	Detection	: UV 254nm
Mobile Phase	: 8% IPA : Hex	Temperature	: RT
Flow Rate	: 1.0 mL/min	Pressure	:
Note	:		

Autostop	: 20.00, min	External Start	: Start Only, Down
Detector 1	: Signal 1	Range 1	: Bipolar, 10000 mV, 10 Samp. per Sec.
Subtraction chromatogram	: (None)	Matching	: No Change



Result Table - Calculation Method Uncal

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2	4.137	8.736	0.212	4.6e-02	4.5e-02	0.18
3	5.597	11.128	0.604	0.1	0.1	0.29
4	10.043	2370.023	92.884	12.4	19.8	0.38
5	13.400	154.470	5.090	0.8	1.1	0.46
6	15.360	16535.640	365.795	86.4	77.8	0.70
	Total	19139.219	469.971	100.0	100.0	

HPLC Chromatogram of (*R*)-2 crude reaction mixture

26/06/2007 16:12 Chromatogram RV-7-612 Page 1 of 1

Created	: 26/06/2007 15:59:37	By	: Clarity
Project	: Work3	Report Style	: Full
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Calibration	: RV-7-612	Chromatogram	: DATA\RV-7-612
Method	: FMG	By	: Clarity
Description	:		
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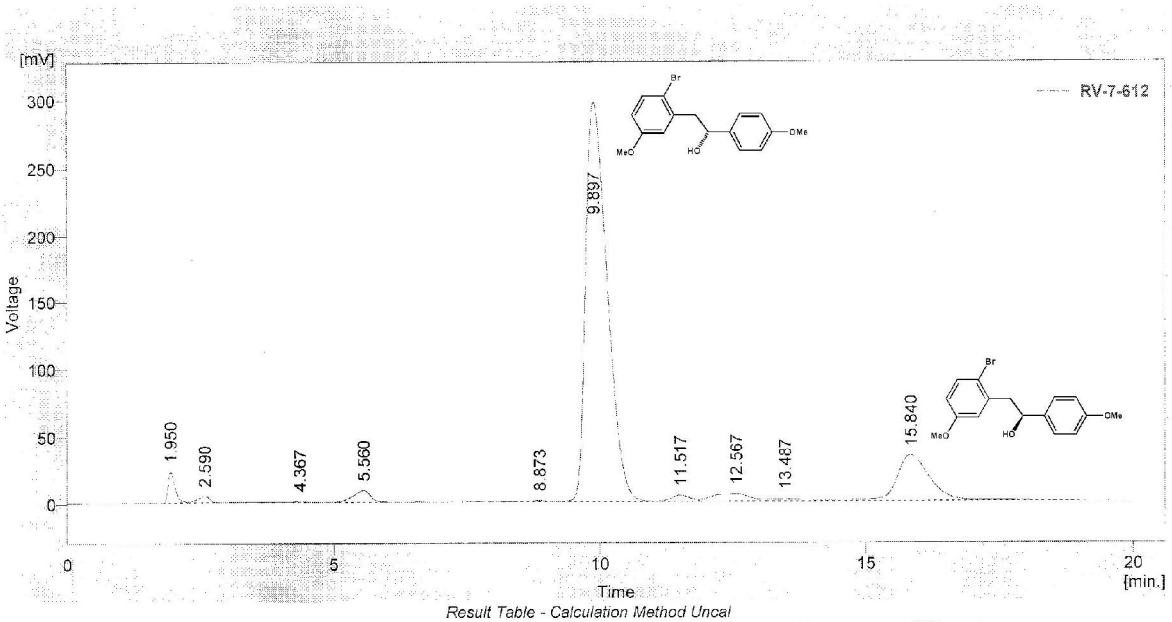
Column	: Kromasil 3-CelluCoat	Detection	: UV 254nm
Mobile Phase	: 8%IPA : Hex	Temperature	: RT
Flow Rate	: 1.0 mL/min	Pressure	:
Note	:		
Autostop	: 20.00, min	External Start	: Start Only, Down
Detector 1	: Signal 1	Range 1	: Bipolar, 10000 mV, 10 Samp. per Sec.
Subtraction chromatogram	: (None)	Matching	: No Change



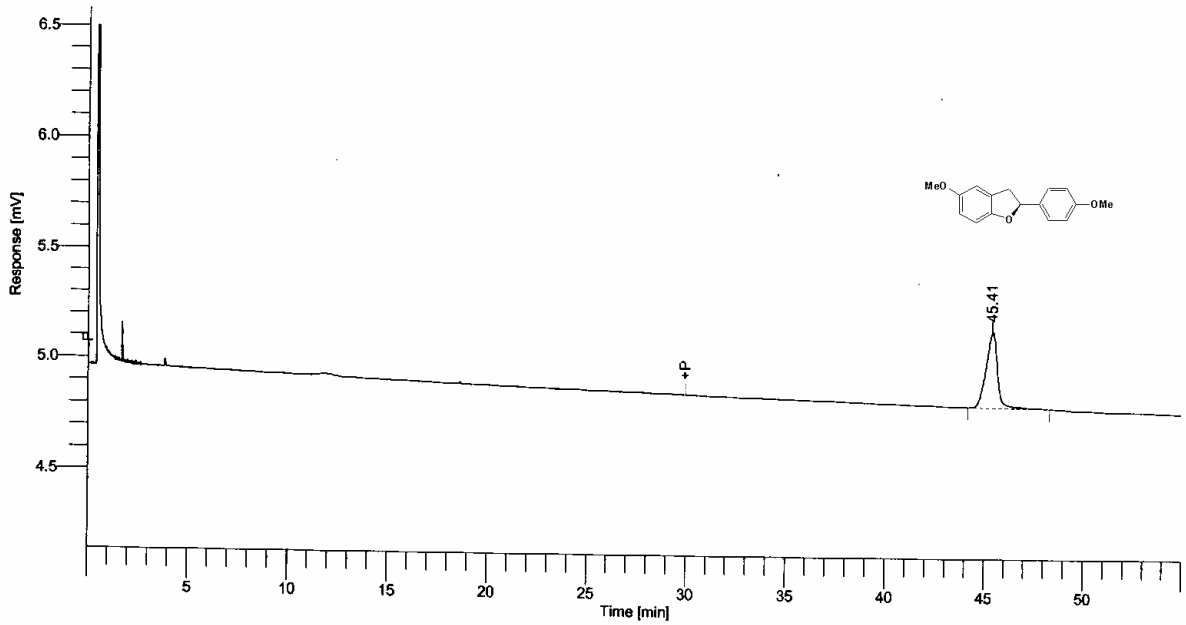
Result Table - Calculation Method Uncal

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	1.950	209.153	22.952	1.9	6.0	0.14
2	2.590	75.363	4.689	0.7	1.2	0.26
3	4.367	12.546	1.097	0.1	0.3	0.17
4	5.560	200.450	8.679	1.8	2.3	0.36
5	8.873	25.522	0.959	0.2	0.3	0.34
6	9.897	8331.482	296.745	76.8	77.9	0.45
7	11.517	116.325	4.587	1.1	1.2	0.40
8	12.567	271.861	5.559	2.5	1.5	0.81
9	13.487	87.075	1.749	0.8	0.5	0.92
10	15.840	1520.793	33.914	14.0	8.9	0.62
	Total	10850.569	380.939	100.0	100.0	

GC Chromatogram of Synthetic (*S*)-1

Software Version	: 6.2.0.0.0:B27	Date	: 1/21/08 3:49:00 PM
Reprocess Number	: gc_server: 13067		
Operator	: chp04rv	Sample Name	: RV/8/705
Sample Number	: 002	Study	:
AutoSampler	: BUILT-IN	Rack/Vial	: 0/2
Instrument Name	: AutoSystemFID	Channel	: A
Instrument Serial #	: None	A/D mV Range	: 1000
Delay Time	: 0.00 min	End Time	: 55.00 min
Sampling Rate	: 6.2500 pts/s		
Sample Volume	: 1.000000	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 1/21/08 2:53:53 PM	Cycle	: 2

Raw Data File : \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.raw
Result File : \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.rst
Inst Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.raw
Proc Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.rst
Calib Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.rst
Report Format File: \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H.rpt
Sequence File : \\gc_server\TCData\AutoSystemFID_Sequence_Index\RVemula\RV-7-643 race,8-705,real sample.seq



DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		45.411	13802.93	337.63	100.00	100.00			BB	0.0138	0.0138
			13802.93	337.63	100.00	100.00				0.0138	0.0138

GC Chromatogram of (*R*)-1

Software Version	: 6.2.0.0.0:B27	Date	: 8/17/07 1:13:15 PM
Reprocess Number	: gc_server: 12544		
Operator	: chp04rv	Sample Name	: RV/7637
Sample Number	: 003	Study	:
AutoSampler	: BUILT-IN	Rack/Vial	: 0/12
Instrument Name	: AutoSystemFID	Channel	: A
Instrument Serial #	: None	A/D mV Range	: 1000
Delay Time	: 0.00 min	End Time	: 53.00 min
Sampling Rate	: 6.2500 pts/s		
Sample Volume	: 1.000000	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 8/17/07 12:20:08 PM	Cycle	: 2

Raw Data File : \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.raw
Result File : \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.rst
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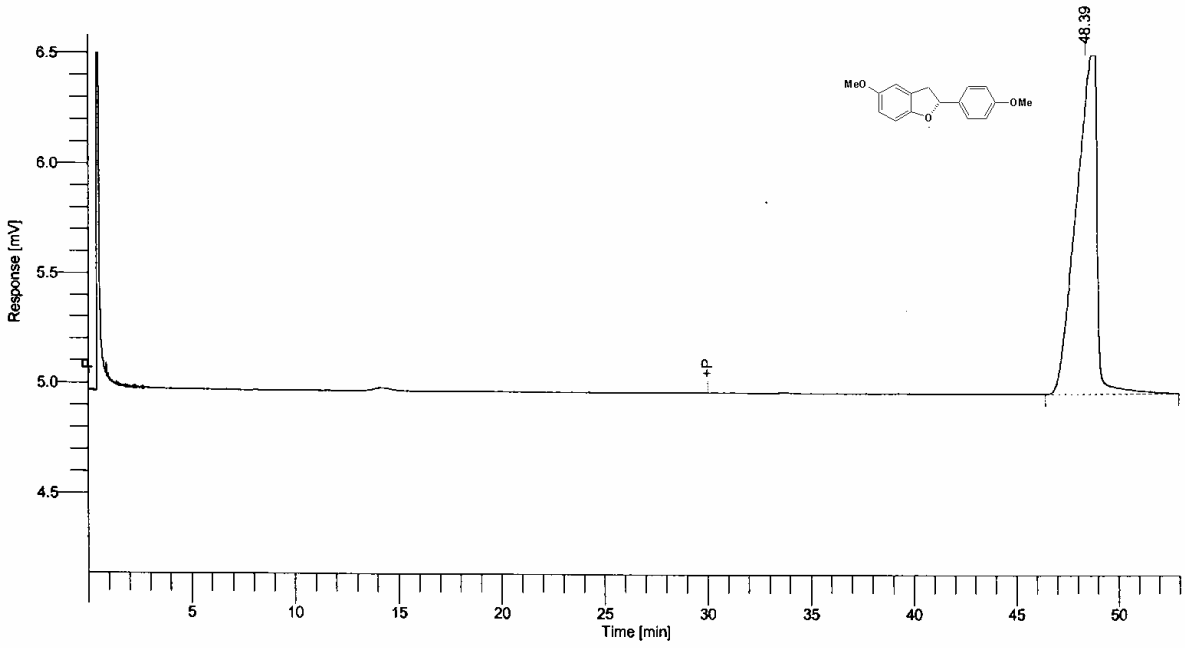
Proc Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.rst
Calib Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.rst
Report Format File: \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H.rpt
Sequence File : \\gc_server\TCData\AutoSystemFID_Sequence_Index\RVemula\RV-7-643.seq



DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		48.386	111854.09	1305.89	100.00	100.00			BB	0.1119	0.1119
			111854.09	1305.89	100.00	100.00				0.1119	0.1119

GC Chromatogram of racemic corsifuran 1

Software Version	: 6.2.0.0.0:B27	Date	: 8/17/07 11:12:33 AM
Reprocess Number	: gc_server: 12542		
Operator	: chp04rv	Sample Name	: RV/7/643
Sample Number	: 001	Study	:
AutoSampler	: BUILT-IN	Rack/Vial	: 0/10
Instrument Name	: AutoSystemFID	Channel	: A
Instrument Serial #	: None	A/D mV Range	: 1000
Delay Time	: 0.00 min	End Time	: 50.00 min
Sampling Rate	: 6.2500 pts/s		
Sample Volume	: 1.000000	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 8/17/07 10:00:52 AM	Cycle	: 1

Raw Data File : \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_001.raw
Result File : \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_001-20070817-111232.rst
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Proc Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H.mth from
\\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_001-20070817-111232.rst
Calib Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H.mth from
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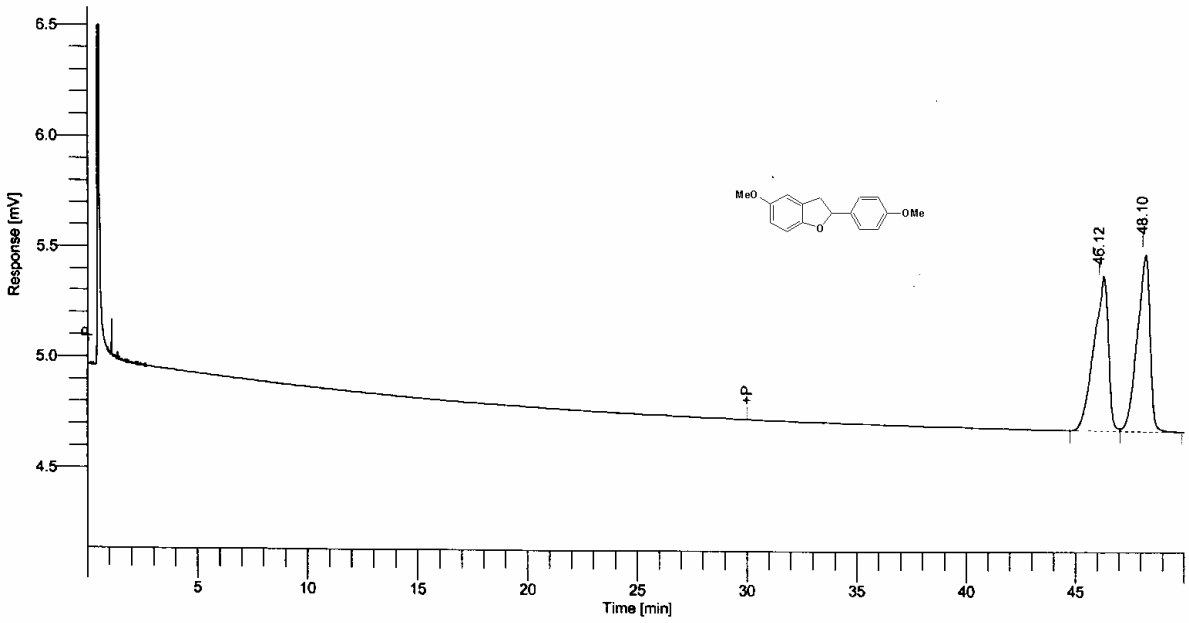
Report Format File: \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H.rpt
Sequence File : \\gc_server\TCData\AutoSystemFID_Sequence_Index\RVemula\RV-7-643-20070817-094637.idx



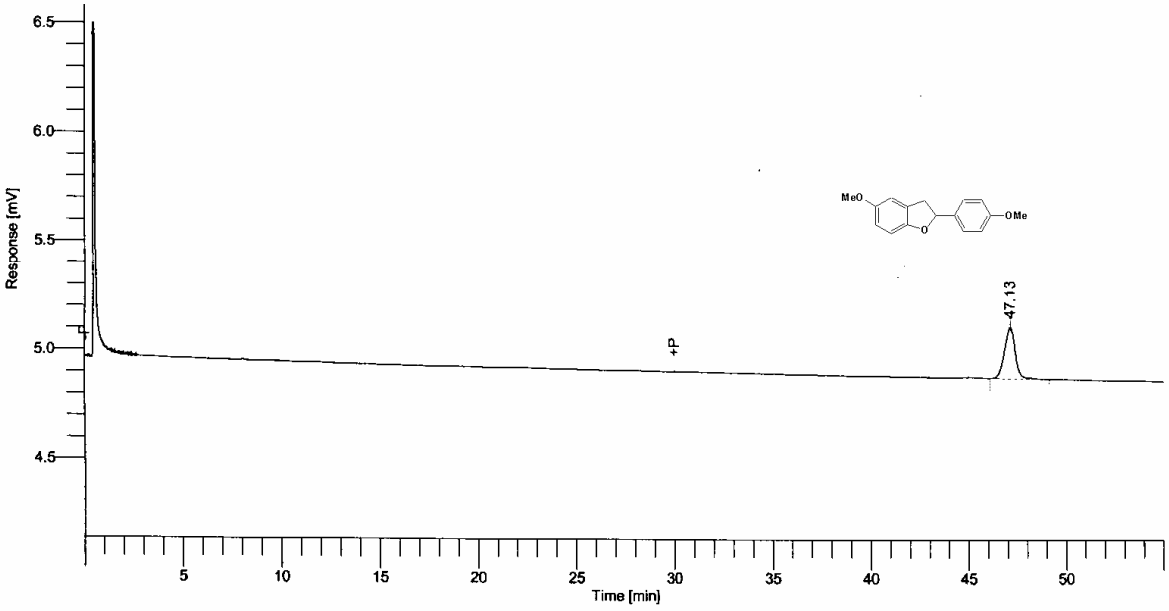
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		46.117	32352.73	544.61	49.53	49.53			BV	0.0324	0.0324
2		48.103	32966.69	684.56	50.47	50.47			VB	0.0330	0.0330
			65319.42	1229.17	100.00	100.00				0.0653	0.0653

GC Chromatogram of natural corsifuran 1

Software Version	: 6.2.0.0.0:B27	Date	: 1/21/08 4:50:05 PM
Reprocess Number	: gc_server: 13069		
Operator	: chp04rv	Sample Name	: RV/8/Real sample
Sample Number	: 003	Study	:
AutoSampler	: BUILT-IN	Rack/Vial	: 0/3
Instrument Name	: AutoSystemFID	Channel	: A
Instrument Serial #	: None	A/D mV Range	: 1000
Delay Time	: 0.00 min	End Time	: 55.00 min
Sampling Rate	: 6.2500 pts/s		
Sample Volume	: 1.000000	Area Reject	: 0.000000
Sample Amount	: 1.0000	Dilution Factor	: 1.00
Data Acquisition Time	: 1/21/08 3:54:59 PM	Cycle	: 3

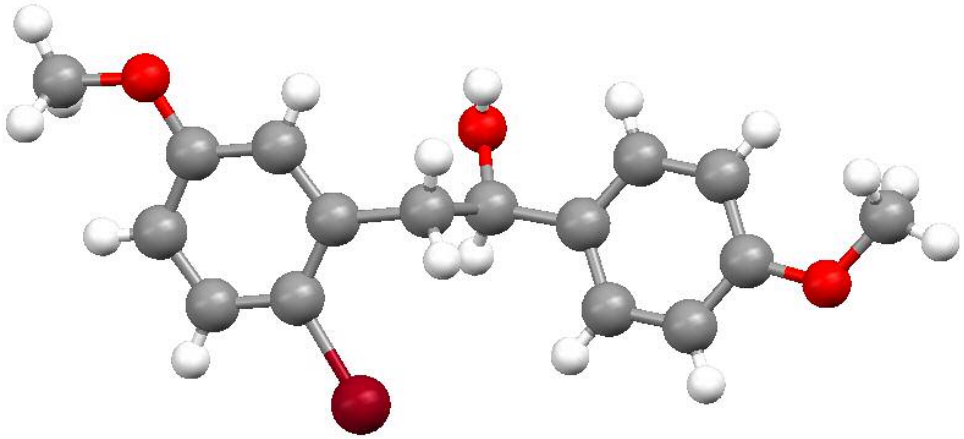
Raw Data File : \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_003.raw
Result File : \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_003.rst
Inst Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_003.raw
Proc Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_003.rst
Calib Method : \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H from \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_003.rst
Report Format File: \\gc_server\TCData\AutoSystemFID_Methods_Reports\Cyclodextrin H.rpt
Sequence File : \\gc_server\TCData\AutoSystemFID_Sequence_Index\RVemula\RV-7-643 race,8-705,real sample.seq



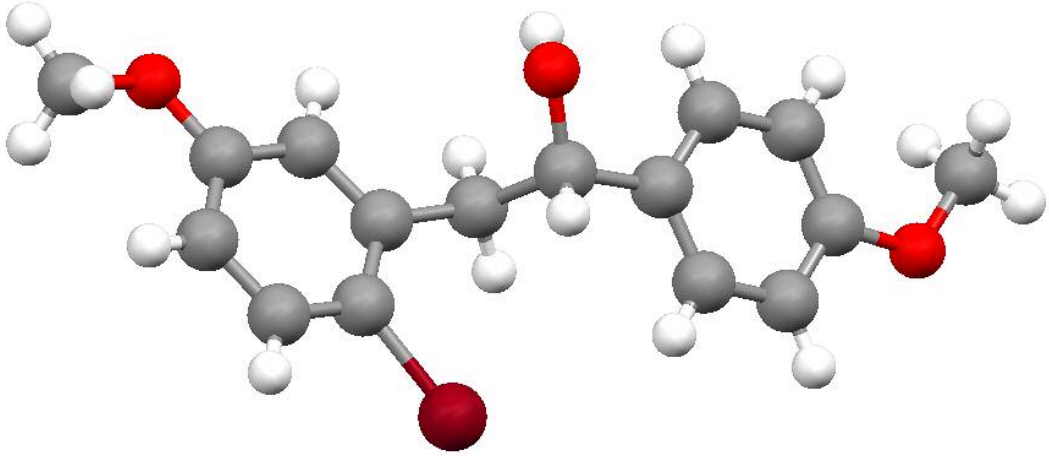
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		47.132	9153.96	234.26	100.00	100.00			BB	0.0092	0.0092
			9153.96	234.26	100.00	100.00				0.0092	0.0092

X-Ray Structure of (*R*)-1



X-Ray Structure of (*S*)-1



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