

Electrophilic dimerization of naphthols in the presence of aluminum halides

Konstantin Yu. Koltunov, Aleksey N. Chernov, Ludmila A. Ostashevskaya and Pavel A. Gribov

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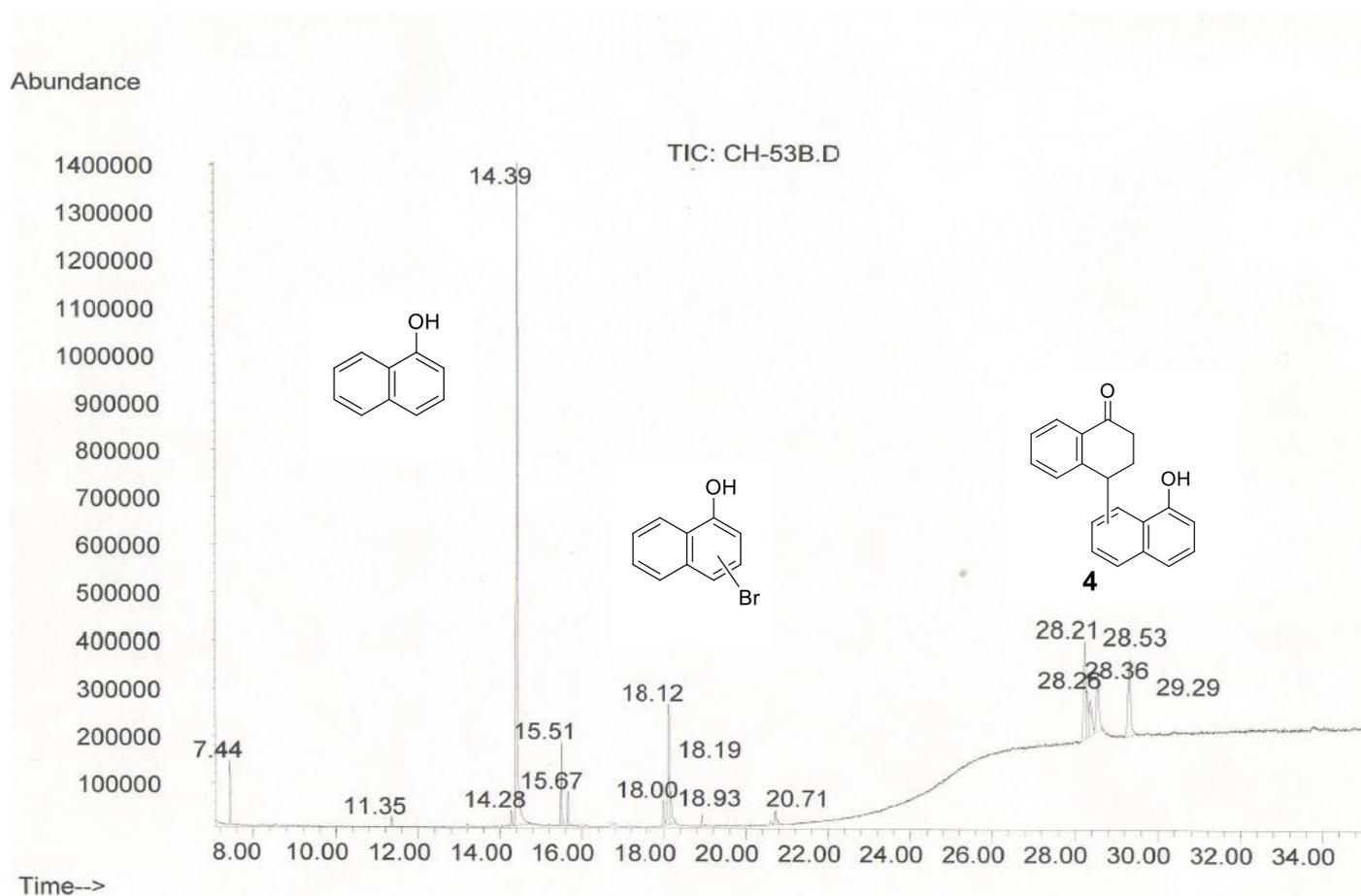
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1. General information

The ^1H and ^{13}C NMR spectra were recorded on a Bruker AVANCE III 500 spectrometer at 500 and 125 MHz, and on a Bruker DPX-250 spectrometer at 250.13 and 62.9 MHz, respectively. The chemical shifts were measured relative to the solvent signals (CDCl_3 δ 7.26 ppm and δ_{C} 77.16 ppm; d_6 -acetone δ 2.06 ppm and δ_{C} 206 ppm). GC-MS data were acquired on an Agilent 6890N/5973N EI/PCI instrument. Chromatographing was performed using a HP-5MS column; the temperature was programmed from 50 to 280°C, 10 K/min. The temperature of the vaporizer was 280°C.

Anhydrous aluminum bromide and aluminum chloride (purity 98-99%), and 1- and 2-naphthols (purity 99%) were purchased from chemical suppliers and used as received. However, in order to provide reproducibility of dimerization of 2-naphthol, aluminum chloride was sublimed before using.

2. GC-MS data of reaction mixture obtained from the reaction of 1-naphthol with a 3.5-fold molar excess of AlBr_3 in CH_2Br_2 at 25°C for 24 h



Library Search Report

Data Path : D:\MSD-Data\kafedra\Koltunov\
Data File : CH-53B.D
Acq On : 21 Oct 2008 11:19
Operator : Karpova E.V.
Sample : Ch-53(b)
Misc : after 7 min (CHCl3+DMSO+acetone)
ALS Vial : 1 Sample Multiplier: 1

Search Libraries: C:\Database\NIST02.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: Chemstation Integrator - EVENTS_T_15_5.E

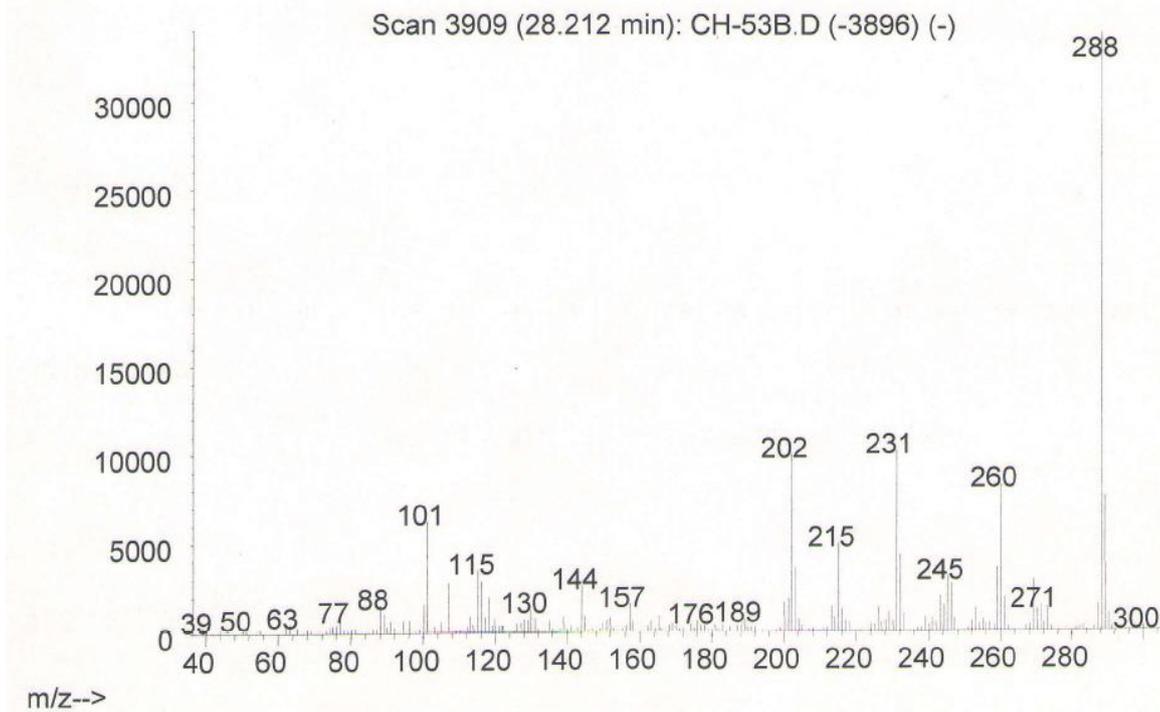
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2	11.35	0.58	Ethane, 1,1,2,2-tetrabromo-	138205	000079-27-6	98
3	14.28	0.81	Naphthalene, 1,4,5-trimethyl-	36209	002131-41-1	72
4	14.39	41.67	1-Naphthalenol	19576	000090-15-3	95
5	15.51	4.02	Naphthalene, 1,6,7-trimethyl-	36214	002245-38-7	90
6	15.67	1.78	2-Naphthalenol, 1-bromo- 1-Naphthalenol, 4-bromo- 2-Naphthalenol, 6-bromo-	70366 70364 70365	000573-97-7 000571-57-3 015231-91-1	95 94 70
7	18.00	1.63	1-Naphthalenol, 4-bromo- 2-Naphthalenol, 6-bromo- 2-Naphthalenol, 1-bromo-	70364 70365 70366	000571-57-3 015231-91-1 000573-97-7	94 86 86
8	18.12	8.77	1-Naphthalenol, 4-bromo- Naphthalene, 7-bromo-2-hydroxy- 2-Naphthalenol, 6-bromo-	70364 70369 70365	000571-57-3 116230-30-9 015231-91-1	96 89 89
9	18.18	2.52	1-Naphthalenol, 4-bromo- 2-Naphthalenol, 1-bromo- 2-Naphthalenol, 6-bromo-	70364 70366 70365	000571-57-3 000573-97-7 015231-91-1	94 93 70
10	18.94	0.59	2-Naphthalenol, 1,6-dibromo-	117245	016239-18-2	89
11	20.71	0.96	?			
12	28.21	9.17	?			
13	28.27	4.38	?			
14	28.36	4.55	?			

15 28.53 6.59 ?

16 29.29 9.06 ?

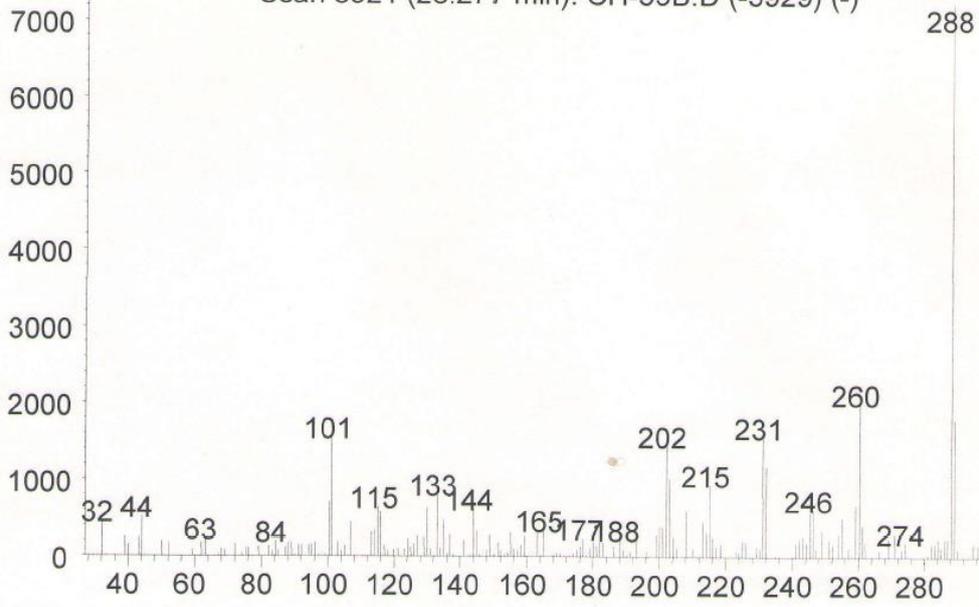
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Abundance



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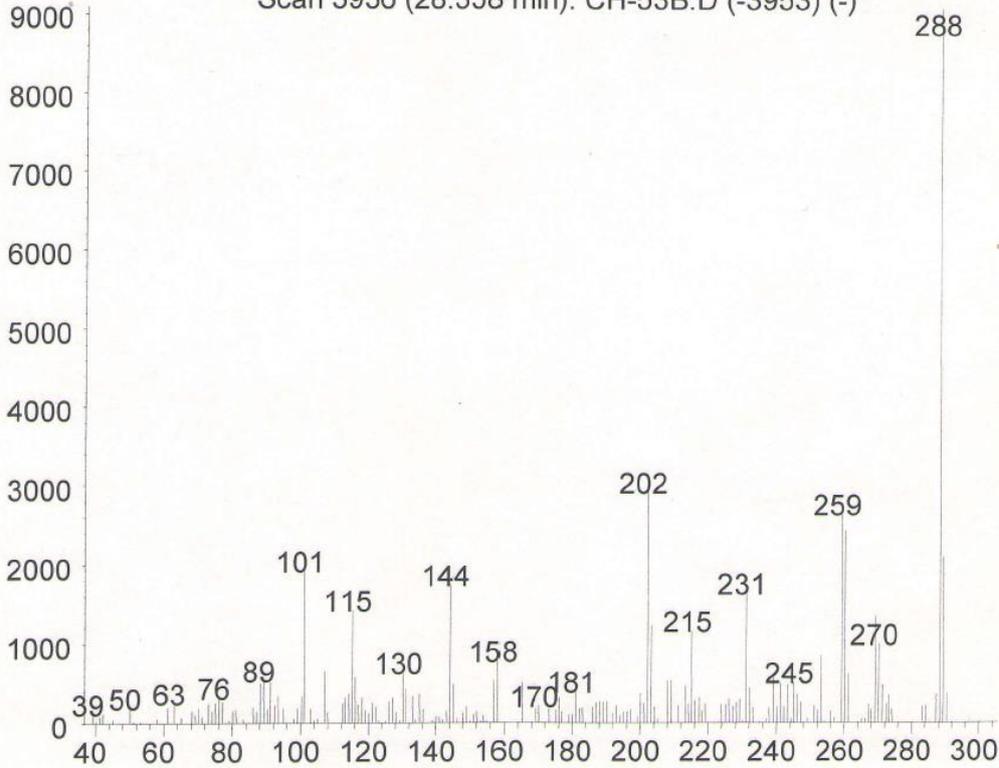
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m/z-->

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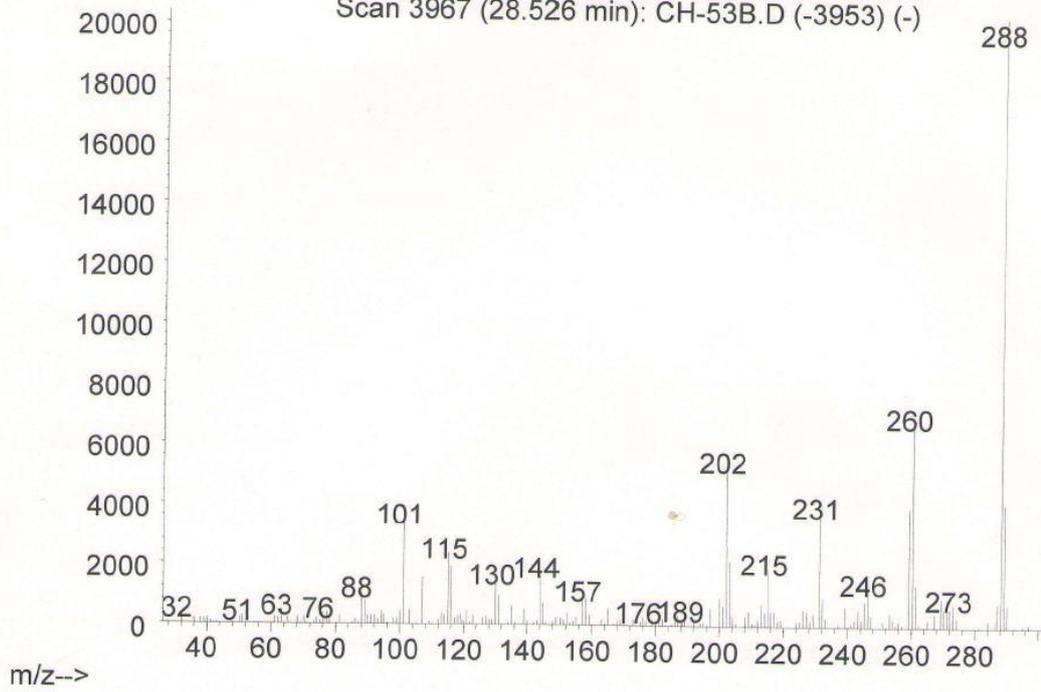
Scan 3936 (28.358 min): CH-53B.D (-3953) (-)



m/z-->

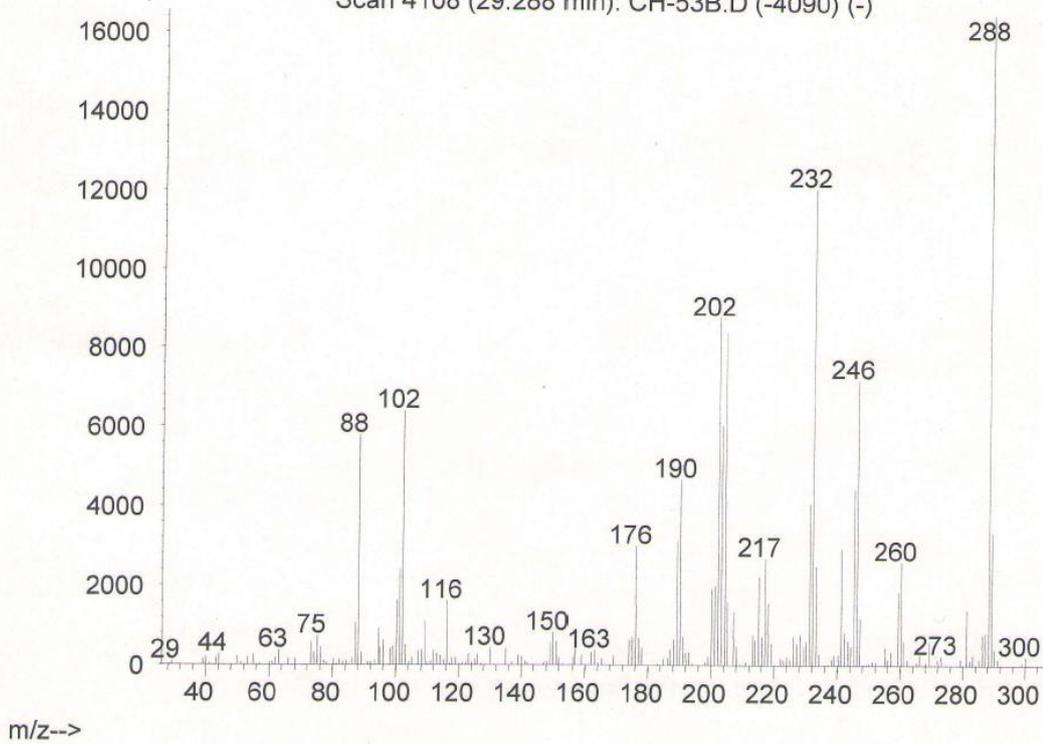
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Scan 3967 (28.526 min): CH-53B.D (-3953) (-)

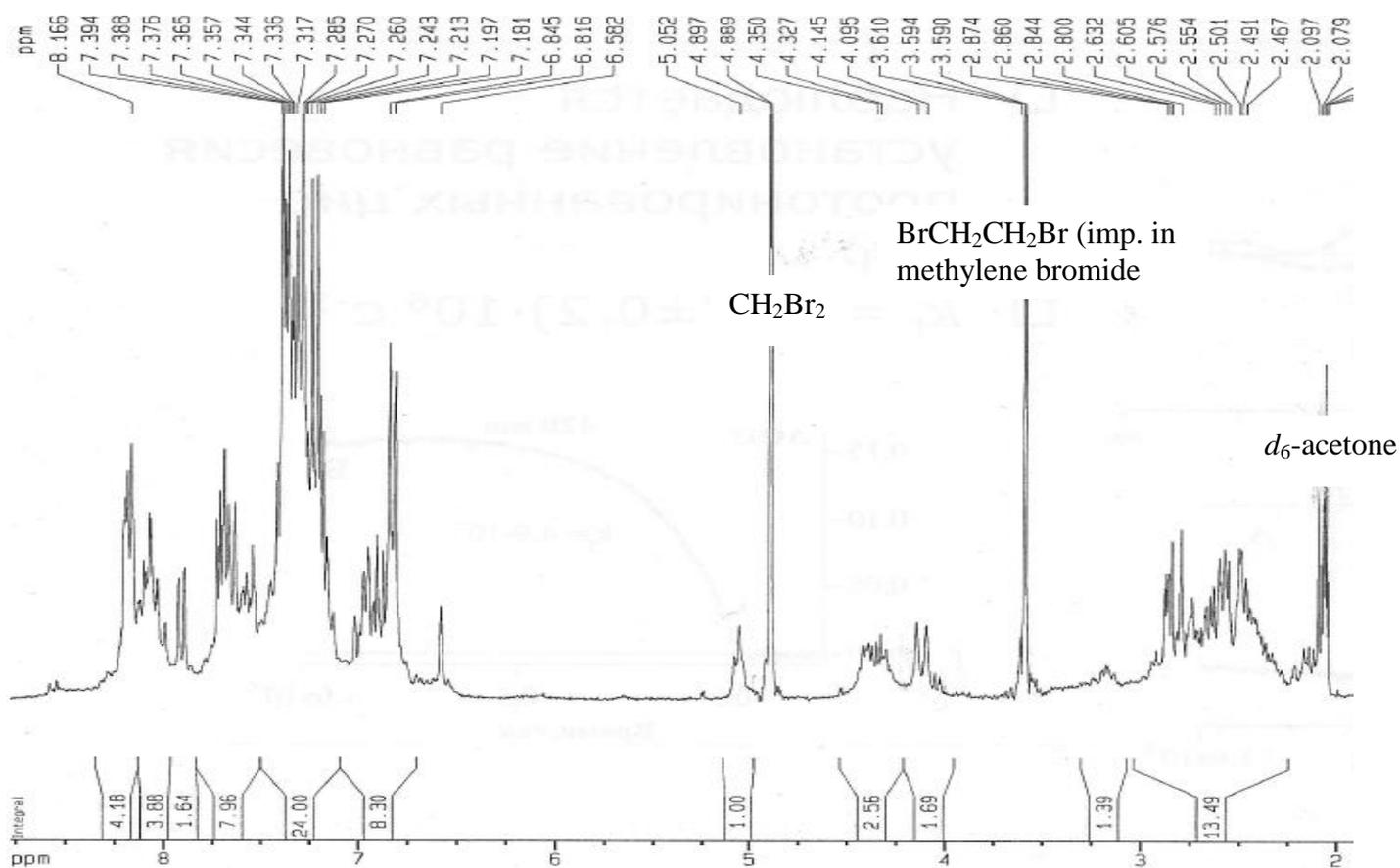


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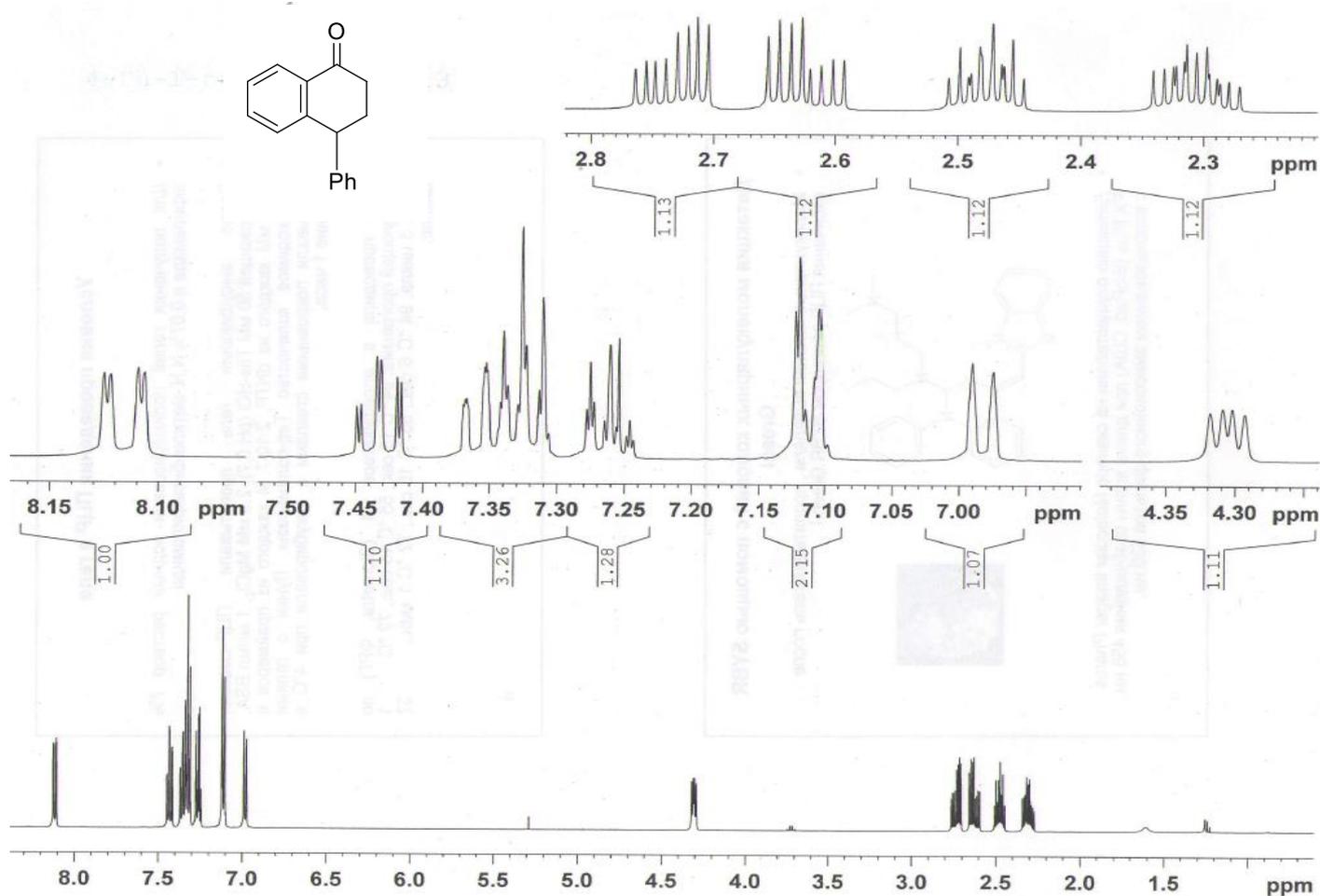
Scan 4108 (29.288 min): CH-53B.D (-4090) (-)



3. 250 MHz Proton NMR spectrum of the reaction mixture obtained from the reaction of 1-naphthol with a 3.5-fold molar excess of AlBr_3 in CH_2Br_2 at 25°C for 150 h (d_6 -acetone solution at room temperature)

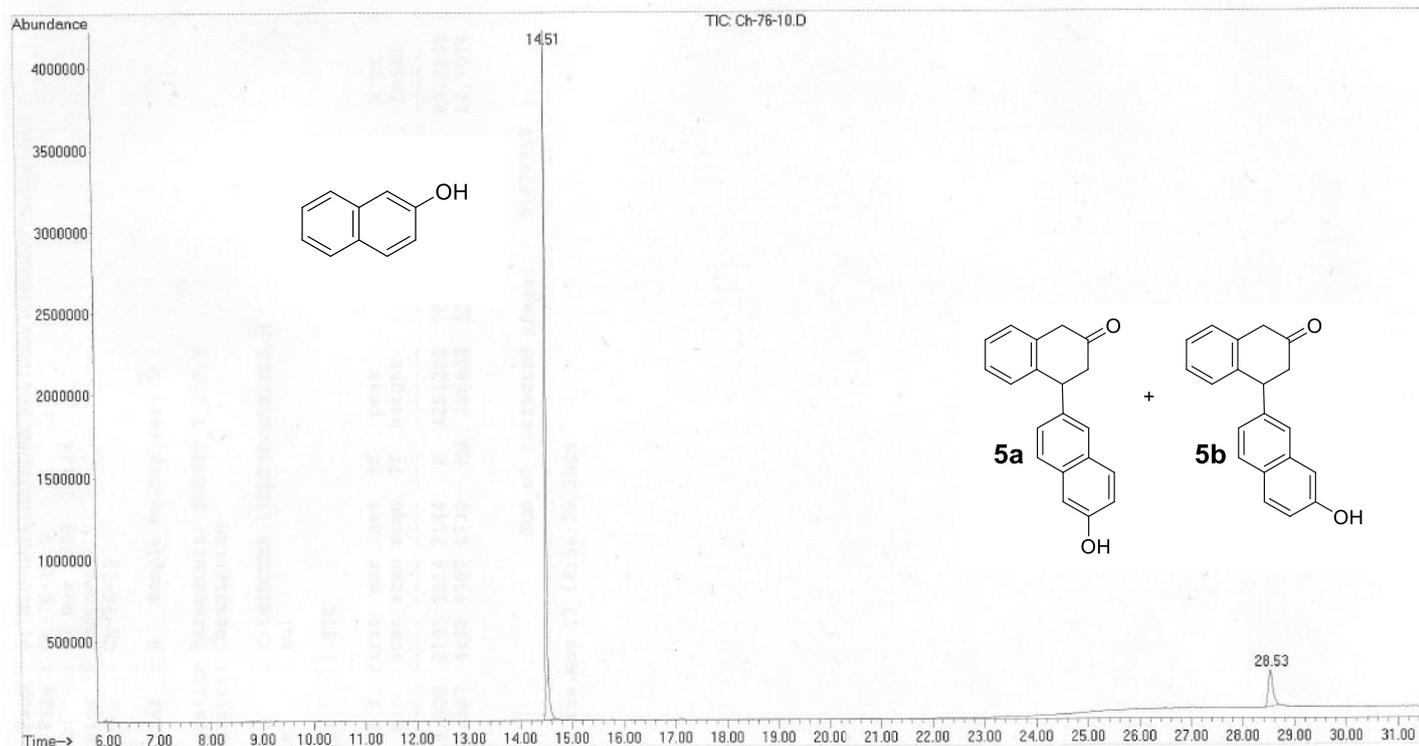


4. 500 MHz Proton NMR spectrum of 4-phenyl-1-tetralone (CDCl_3 solution at room temperature), which is represented for the purpose of comparison



5. GC-MS data of reaction mixture obtained from the reaction of 2-naphthol with a 5-fold molar excess of AlCl₃ at 150°C for 2 h

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 Instrument : Chemist
 Sample Name: Ch-76-10
 Misc Info :
 Vial Number: 8



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 Acq On : 17 Nov 2009 14:24
 Operator : Karpova E.V.
 Sample : Ch-76-10
 Misc :
 ALS Vial : 8 Sample Multiplier: 1

Integration Parameters: EVENTS_T_15T.E
 Integrator: ChemStation

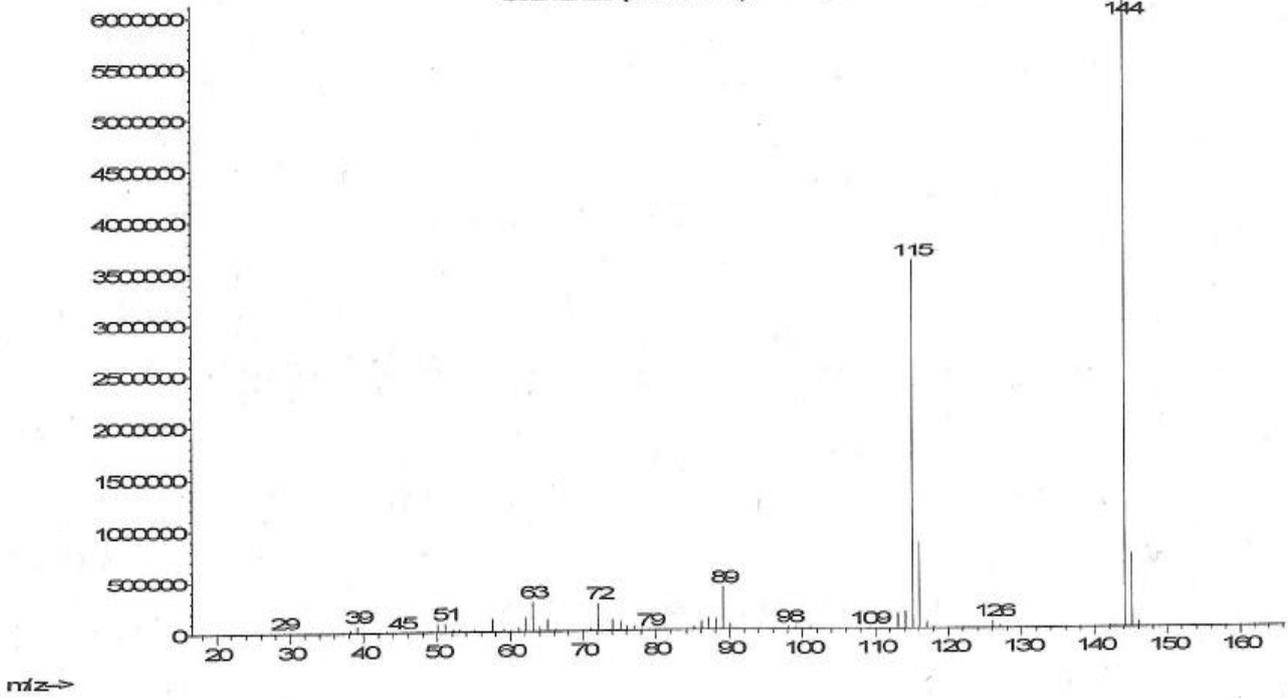
Method : C:\MSDCHEM\1\METHODS\NIOCH.M
 Title : sul

Signal : TIC

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	14.509	2105	2114	2144	M	4251200	78024200	100.00%	85.293%
2	28.527	4688	4707	4739	M3	226633	13454160	17.24%	14.707%

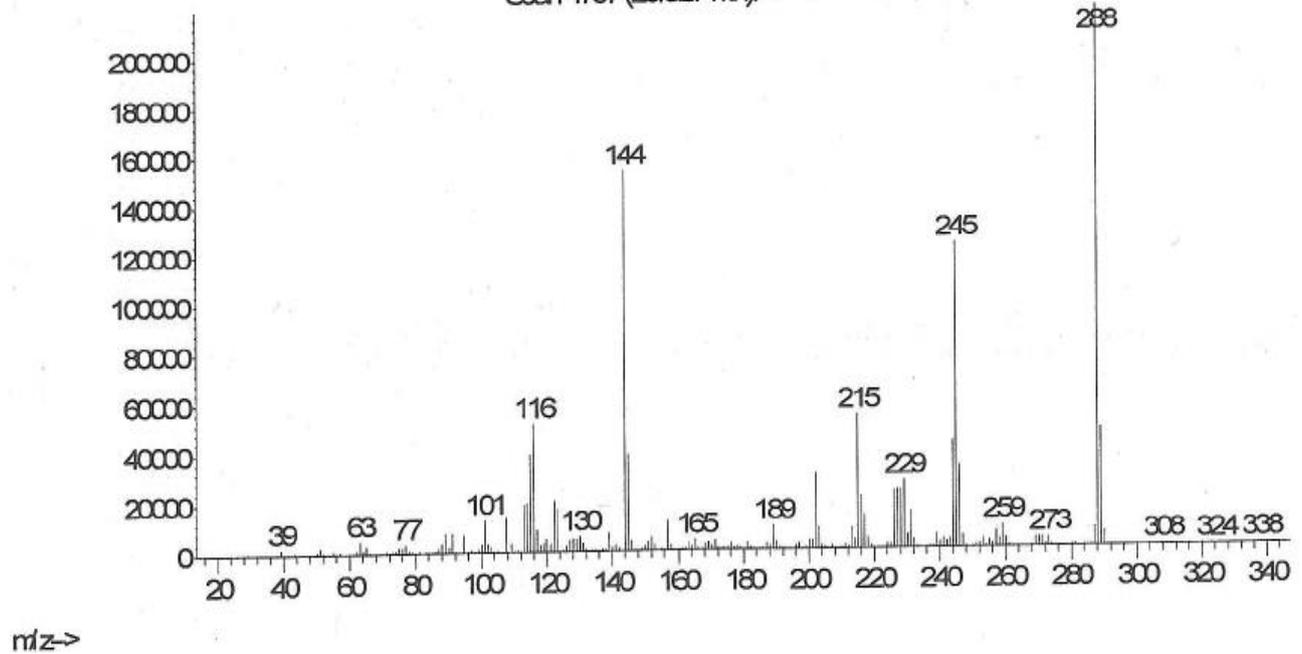
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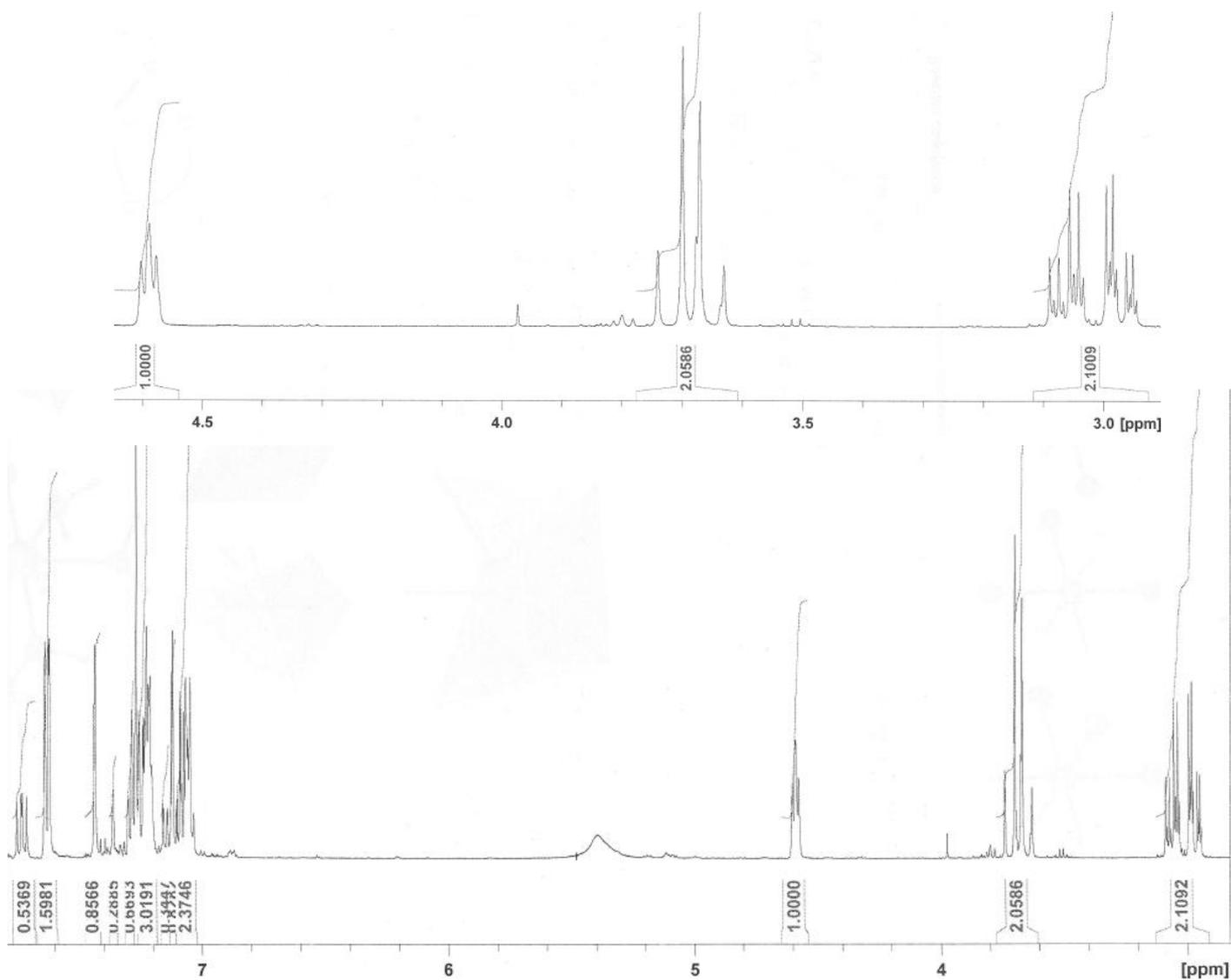
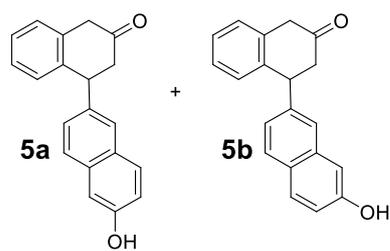


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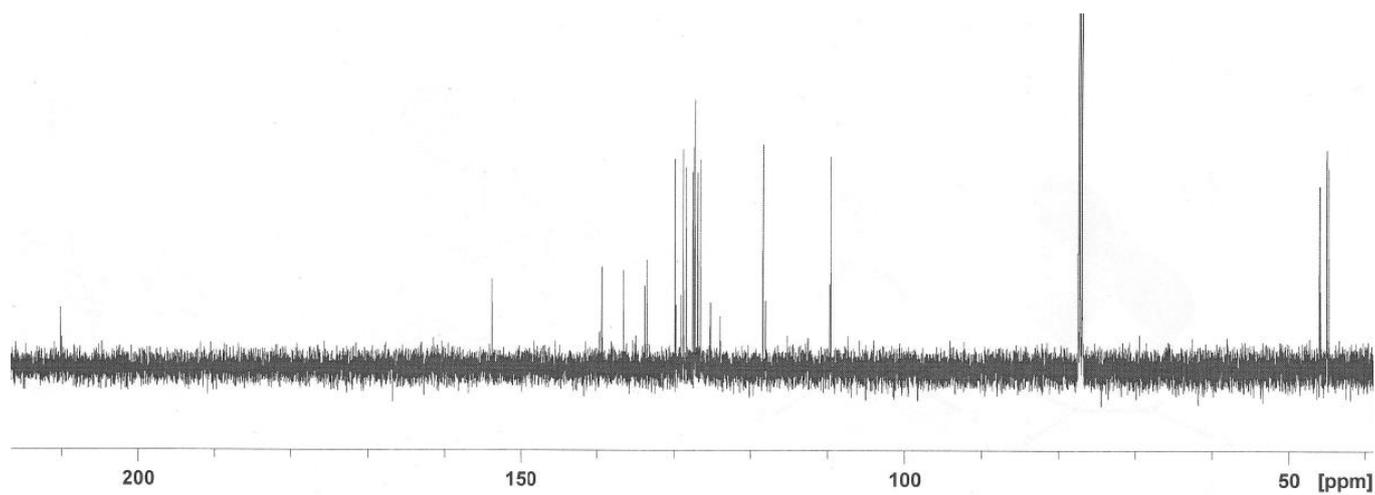
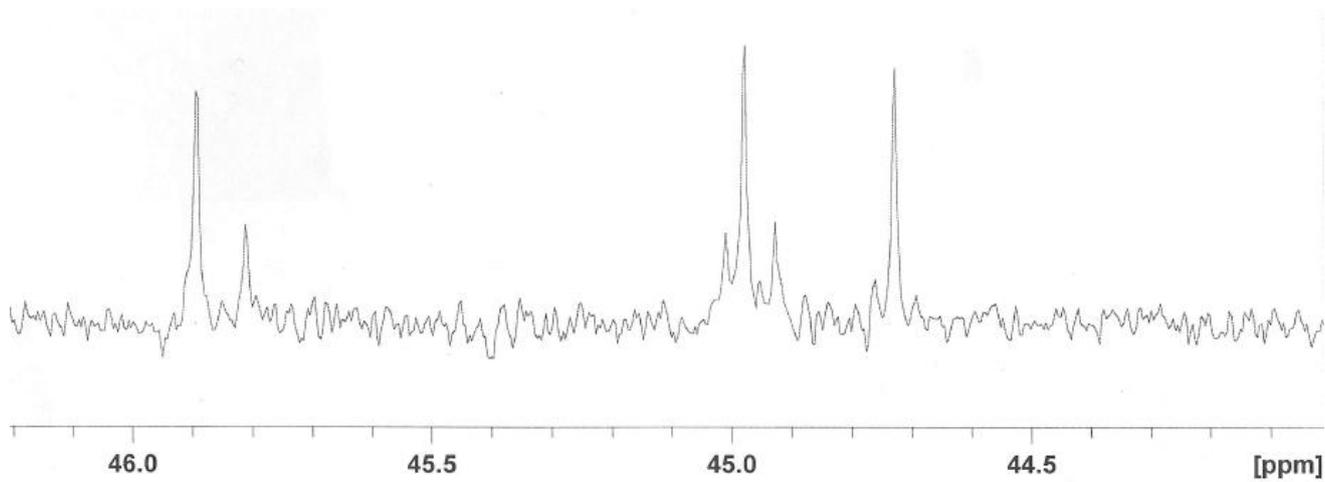
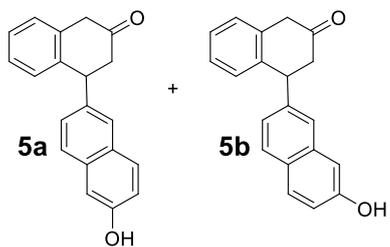
Scan 4707 (28.527 min):



6. 500 MHz Proton NMR spectrum of 5a/5b (~3:1) mixture (CDCl₃ solution at room temperature)



7. 125 MHz ^{13}C NMR spectrum of 5a/5b (~3:1) mixture (CDCl_3 solution at room temperature)



8. 500 MHz Proton NMR spectrum of 4-phenyl-2-tetralone (CDCl₃ solution at room temperature), which is represented for the purpose of comparison

