Electronic Supplementary Material (ESI) for ChemComm.

Cu(II)-catalyzed Asymmetric Boron Conjugate Addition to α,β-Unsaturated Imines in Water

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Supporting Information

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Experimental

1. General

Nuclear magnetic resonance (NMR) spectra were recorded on a JEOL ECX-600 or ECX-500 or ECX-400 spectrometer, operating at 600 or 500 MHz or 400 MHz for ^{1}H and 150 or 125 or 100 MHz for ^{13}C NMR in $CDCl_3$ unless otherwise noted. Trimethylsilane (TMS) served as the internal standard (δ= 0) for ^{1}H NMR and $CDCl_3$ was used as the internal standard (δ = 77.0) for ^{13}C NMR. Infrared (IR) spectra were obtained using a JASCO FT/IR-4200 spectrometer. Data are represented as frequency of absorption (cm^{-1}). High-performance liquid chromatography was carried out using following apparatuses; SHIMADZU LC-10ATvp (liquid chromatograph), SHIMADZU SPD-10A (UV detector) and SHIMADZU C-R8A (Chromatopac) using Daicel chiralpak® or chiralcel® columns. Preparative thin-layer chromatography (PTLC) was carried out using Wakogel B-5F from Wako Pure Chemical Industries, Ltd. High Resolution Mass Spectra (HRMS) were recorded using a Brucker Daltonics BioTOF II (ESI) spectrometer. Optical Rotations were measured on a JASCO P1010 polarimeter using a 2 mL cell with 1 dm path length. Data are reported as follows: $[\alpha]_D^T$ (*c* in g/100 mL, solvent). Deionized water from a MILLIPORE MilliQ machine (Gradient A 10) was used as solvent without further treatment. All reagents used as additives were either distilled or recrystallized before use.

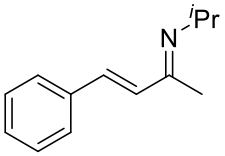
2. Synthesis of α,β-Unsaturated Imines

[General Method]

Method A: The corresponding amine (10 mmol), corresponding ketone (10 mmol), montmorillonite K10 (1 g) and molecular sieves 5A (1 g) were stirred in CH_3CN (10 mL) for 16 h at room temperature. The reaction mixture was filtered through celite®, and the product was isolated by distillation.

Method B: A mixture of ketone (10 mmol) and benzylamine (10 mmol) in 20 mL of hexane (freshly distilled from calcium hydride) was refluxed for 15 h over molecular sieves 5A (1 g). After filtration, the crude oil was crystallized under refrigeration, and recrystallization from THF/^{n}hexane = 1/4.

(2*E*, 3*E*)-*N*-isopropyl-4-phenylbut-3-en-2-imine



The title compound was prepared according to Method A.

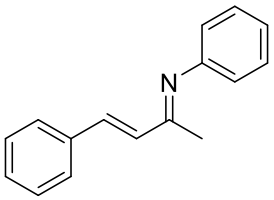
Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$); δ = 1.00 (d, *J* = 5.6 Hz, 6H), 2.05 (s, 3H), 2.49-2.57 (m, 1H), 6.83 (d, *J* = 16.6 Hz, 1H), 7.32-7.59 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$); δ = 11.6, 23.4, 67.3, 119.9, 127.8, 128.8, 128.9, 129.8, 136.2, 165.0.

HRMS (ESI) calcd for $C_{13}H_{18}N$ $[M+H]^+$ 188.1439, found 188.1426.

(2*E*, 3*E*)-N,4-diphenylbut-3-en-2-imine11



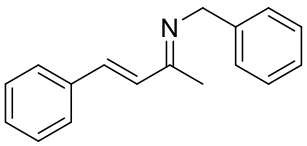
The title compound was prepared according to Method B.

White solid; mp 47-53 ^{o}C.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.28 (s, 3H), 6.69 (d, *J* = 15.9 Hz, 1H), 7.18-7.53 (m, 11H).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 13.7, 115.8, 116.6, 119.5, 120.1, 122.3, 128.1, 130.9, 136.3, 145.4, 149.1, 161.2.

(2*E*, 3*E*)-*N*-benzyl-4-phenylbut-3-en-2-imine [1]



The title compound was prepared according to Method B.

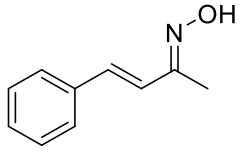
Pale yellow solid; mp 61-66 ^{o}C.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.19 (s, 3H), 4.67 (s, 1H), 4.74 (s, 1H), 6.99 (d, 1H, *J*

= 16.4 Hz), 7.03 (d, 2H, *J* = 8.5 Hz), 7.26-7.53 (m, 10H).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 12.2, 50.1, 124.5, 125.4, 126.0, 126.3, 126.9, 128.5, 133.4, 137.1, 139.9, 162.8.

(2*E*, 3*E)*-4-phenylbut-3-en-2-one oxime[11]



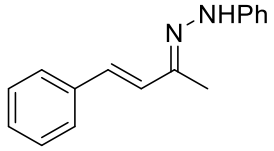
To a solution of (*E*)-4-phenylbut-3-en-2-one (1.46 g, 10 mmol) and pyridine (2.0 mL, 25 mmol) in EtOH (20 mL) was added NH_2OH•HCl (1.04 g, 15 mmol) in one portion and the reaction mixture was stirred at 60 °C for 12 h. The reaction was quenched with water and extracted twice with AcOEt. The combined organic layers was washed with 1N aqueous HCl and brine, and dried over $MgSO_4$. Volatile materials were removed under reduced pressure.

White solid; 122-124 ^{o}C.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.15 (s, 3H), 6.87-6.93 (m, 2H), 7.28-7.35 (m, 3H), 7.46 (d, 2H, *J* = 7.9 Hz).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 9.7, 125.7, 126.9, 128.4, 128.7, 133.4, 136.3, 156.8.

(*E*)-1-phenyl-2-((*E*)-4-phenylbut-3-en-2-ylidene)hydrazine[2]



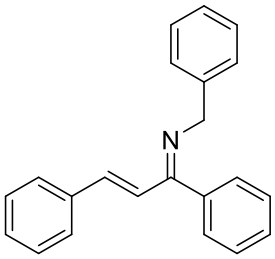
Benzalacetone (1.46 g, 10 mmol) and phenylhydrazine (1.16 g, 11 mmol) were dissolved in MeOH and AcOH (1mL) was added. The reaction solution was stirred at room temperature. After the completion of the reaction, the solid was collected by filtration and washed with cooled MeOH. Then the solid was dissolved in dichloromethane, and the organic layer was washed with saturated aqueous $NaHCO_3$, brine. After dried over Na_2SO_4, the mixture was filtered and evaporated to give the solid, which was further purified by recrystallization from AcOEt/MeOH.

Yellow solid; mp 132-135 ^{o}C.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 1.96 (s, 3H), 5.40 (br s, 1H), 6.64 (d, 1H, *J* = 8.2 Hz), 6.89 (d, 1H, *J* = 8.0 Hz), 7.24-7.37 (m, 8H), 7.47-7.50 (d, 2H, *J* = 8.8 Hz).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 14.5, 113.5, 120.5, 126.5, 127.6, 128.4, 129.3, 129.4, 130.1, 138.7, 143.0, 147.1.

(1*Z*, 2*E*)-*N*-benzyl-1,3-diphenylprop-2-en-1-imine [1]



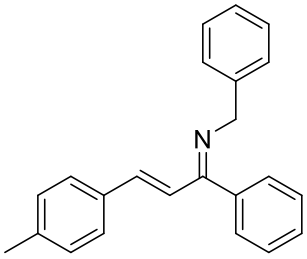
The title compound was prepared according to Method B, then recrystallized from ^{n}pentane/THF = 4/1.

White solid.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 4.48 (s, 2H), 6.48 (d, 1H, *J* = 16.4 Hz), 7.20-7.24 (m, 3H), 7.24-7.29 (m, 4H), 7.30-7.40 (m, 6H), 7.46-7.51 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 57.6, 126.6, 127.3, 127.6, 127.9, 128.5, 128.6, 128.7, 128.9, 132.4, 135.8, 136.0, 139.9, 140.2, 170.4.

(1*Z*, 2*E*)-*N*-benzyl-1-phenyl-3-(*p*-tolyl)prop-2-en-1-imine



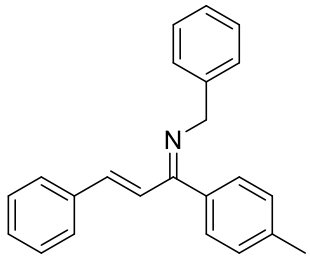
The title compound was prepared according to Method B, then recrystallized from ^{n}pentane/THF = 4/1.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.28 (s, 3H), 4.62 (s, 1H), 4.78 (s, 1H), 6.29 (d, 1H, *J* =16.4 Hz), 6.57 (d, 1H, *J* = 16.4 Hz), 7.04-7.32 (m, 14H).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 17.6, 22.5, 55.7, 57.5, 126.4, 126.8, 127.3, 127.6, 128.1, 128.7, 128.8, 132.2, 135.9, 136.0, 140.1, 140.6, 169.9.

HRMS (ESI) calcd for $C_{23}H_{21}N$ $[M+H]^+$ 312.1752, found 312.1744.

(1*Z*, 2*E*)-N-benzyl-3-phenyl-1-(*p*-tolyl)prop-2-en-1-imine



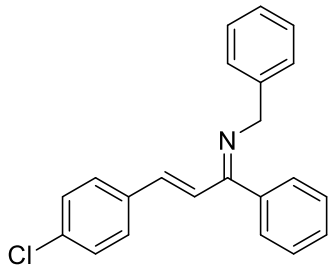
The title compound was prepared according to Method B, then recrystallized from ^{n}hexane/1,4-dioxane = 4/1.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.40 (s, 3H), 4.29-4.61 (m, 2H), 6.39 (d, 1H, *J* =16.0 Hz), 6.61 (d, 1H, *J* = 16.0 Hz), 7.07-7.39 (m, 14H).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 18.7, 24.1, 56.1, 59.2, 126.5, 126.8, 127.3, 127.4, 128.1, 128.5, 129.3, 131.9, 136.2, 136.4, 139.9, 140.2, 170.8.

HRMS (ESI) calcd for $C_{23}H_{21}N$ $[M+H]^+$ 312.1752, found 312.1761.

(1*Z*, 2*E*)-*N*-benzyl-3-(4-chlorophenyl)-1-phenylprop-2-en-1-imine



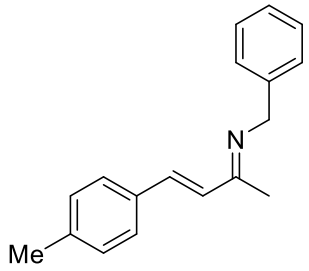
The title compound was prepared according to Method B, then recrystallized from ^{n}pentane/Et_2O = 4/1.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 4.57 (s, 1H), 4.73 (s, 1H), 6.44 (d, 1H, *J* =14.5 Hz), 6.91 (d, 1H, *J* = 14.6 Hz), 7.11-7.58 (m, 12H), 7.94 (d, 2H, *J* = 7.6 Hz).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 60.5, 120.6, 124.5, 127.6, 127.9, 128.4, 128.7, 128.9, 129.1, 129.3, 129.7, 130.2, 132.1, 134.7, 140.3, 167.9.

HRMS (ESI) calcd for $C_{22}H_{18}NCl$ $[M+H]^+$ 332.1206, found 332.1202.

(2*E*, 3*E*)-*N*-benzyl-4-(*p*-tolyl)but-3-en-2-imine



The title compound was prepared according to Method B, then recrystallized from ^{n}pentane/Et_2O = 4/1.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.15 (s, 3H), 2.41 (s, 3H), 4.65 (s, 1H), 4.75 (s, 1H), 6.92 (d, 1H, *J* = 17.2 Hz), 7.21-7.47 (m, 10H).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 12.1, 23.5, 50.0, 120.0, 120.4, 127.5, 127.6, 128.7, 130.3, 130.5, 136.2, 137.8, 139.8, 159.9.

HRMS (ESI) calcd for $C_{18}H_{20}N$ $[M+H]^+$ 250.1596, found 250.1604.

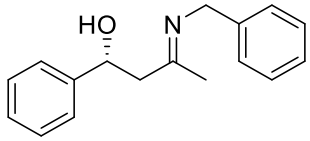
3. ***Typical Experimental Procedure for Chiral Cu(OAc)$_2$-Catalyzed Enantioselective Boron Conjugate Additions to α,β-Unsaturated imines in Water***

[General Method]

An aqueous solution (1 mL) of $Cu(OAc)_2$ (10 mol%) and chiral 2,2'-bipyridine **L1** (12 mol%) was stirred vigorously for 1 h at room temperature. Imine (0.2 mmol) and $B_2(pin)_2$ (0.24 mmol) were then added successively at the same temperature. After stirring for 12 h, dichloromethane (1 mL) was added to the reaction mixture. After washing with saturated aqueous $NaHCO_3$, the mixture was extracted with dichloromethane (20 mL×3). The combined organic layer was dried over anhydrous $MgSO_4$. After concentrated under reduced pressure, THF / H_2O = 3 / 2 (5 mL) and $NaBO_3{\cdot}4H_2O$ (244 mg) were added. After stirring for 3 h, AcOEt (30 mL) was added, and dried over anhydrous $MgSO_4$. After concentrated under reduced pressure, the excess amount of $NaBO_3{\cdot}4H_2O$ (488 mg) was then added and the mixture was stirred at room temperature for 4 h. The aqueous layer was extracted with AcOEt (20 mL) three times, and the combined organic layers were dried over anhydrous Na_2SO_4. After concentrated under reduced pressure, the crude mixture was purified by preparative TLC (^{n}hexane/AcOEt = 4/1) to afford the desired β-hydroxy imines.

4. Analytical Data for β-Hydroxy Imines

(*R*, *E*)-3-(benzylimino)-1-phenylbutan-1-ol[1]



Colorless oil.

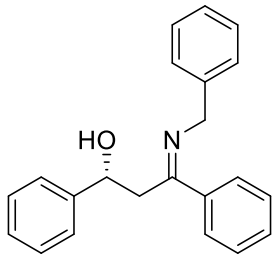
^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.07 (s, 3H), 2.80-2.93 (m, 2H), 3.46 (d, 1H, *J* = 3.3 Hz), 4.66 (s, 2H), 5.11 (dd, 1H, *J* = 5.8 Hz, *J* = 3.5 Hz), 7.28-7.40 (m, 10H).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 27.9, 30.7, 53.4, 70.1, 125.6, 126.5, 126.9, 127.7, 128.1, 128.5, 129.0, 139.9, 169.1.

HPLC; (Dialcel Chiralcel OD-H, ^{n}hexane/ ^{i}PrOH = 90/10, flow rate 1.0 mL/min); t_R = 10.5 min (*S*, minor), t_R = 12.4 min (*R*, major).

$[\alpha]_D^{23}$ = + 35.3 (*c* = 0.63, $CDCl_3$).

(*R*, *Z*)-3-(benzylimino)-1,3-diphenylpropan-1-ol[1]



Colorless oil.

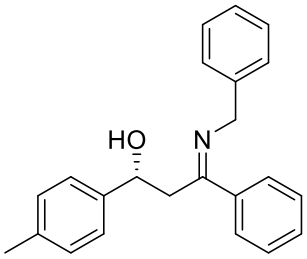
^{1}H NMR (500 MHz, $CDCl_3$); δ = 3.41 (d, 2H, *J* = 5.8 Hz), 4.32 (s, 2H), 5.39 (t, 1H, *J* = 6.5 Hz), 7.28-7.48 (m, 7H), 7.56-7.82 (m, 6H), 7.95 (d, 2H, *J* = 7.0 Hz).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 47.4, 59.2, 70.2, 123.7, 127.5, 128.0, 128.3, 128.4, 128.5, 128.6, 129.1, 129.7, 133.9, 136.6, 143.2, 167.2.

HPLC; (Dialcel Chiralcel OD-H, ^{n}hexane/ ^{i}PrOH = 90/10, flow rate 0.7 mL/min); t_R = 18.1 min (*S*, minor), t_R = 20.9 min (*R*, major).

$[\alpha]_D^{21}$ = + 39.9 (*c* = 0.42, $CDCl_3$).

(*R*, *Z*)-3-(benzylimino)-3-phenyl-1-(*p*-tolyl)propan-1-ol



Colorless oil.

IR (KBr) ν = 1037, 1178, 1648, 2936, 3421 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$); δ = 2.18 (s, 3H), 3.37 (d, 2H, *J* = 8.1 Hz), 4.50 (s, 1H), 4.50 (s, 1H), 5.43 (dd, 1H, *J* = 2.9 Hz, *J* = 4.6 Hz), 7.28-7.54 (m, 12H), 7.95 (d, 2H, *J* = 8.0 Hz).

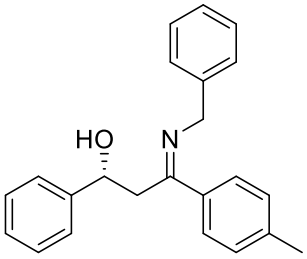
^{13}C NMR (125 MHz, $CDCl_3$); δ = 21.9, 46.8, 59.2, 69.2, 123.7, 126.7, 127.0, 127.1, 128.3, 128.5, 128.9, 129.4, 129.7, 134.4, 140.3, 143.4, 168.3.

HPLC; (Dialcel Chiralcel OD-H, ^{n}hexane/ ^{i}PrOH = 95/5, flow rate 1.0 mL/min); t_R = 17.6 min (*S*, minor), t_R = 21.5 min (*R*, major).

HRMS (ESI) calcd for $C_{23}H_{24}NO$ $[M+H]^+$ 330.1858, found 330.1859.

$[\alpha]_D^{19}$ = + 54.2 (*c* = 0.35, $CDCl_3$).

(*R*, *Z*)-3-(benzylimino)-1-phenyl-3-(*p*-tolyl)propan-1-ol



Yellow oil.

IR (KBr) ν = 1041, 1154, 1646, 2899, 3394 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$); δ = 2.44 (s, 3H), 3.44-3.46 (m, 2H), 4.62 (s, 1H), 4.73 (s, 1H), 5.39-5.41 (m, 1H), 7.27-7.56 (m, 12H), 7.67 (d, 2H, *J* = 7.5 Hz).

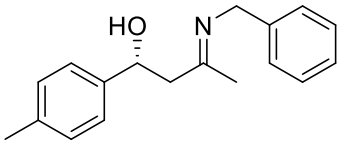
^{13}C NMR (125 MHz, $CDCl_3$); δ = 26.9, 55.9, 61.2, 71.1, 123.8, 125.3, 125.7, 127.6, 128.4, 128.5, 129.1, 129.5, 129.7, 130.5, 133.9, 143.3, 171.7.

HPLC; (Dialcel Chiralcel OD-H, ^{n}hexane/ ^{i}PrOH = 90/10, flow rate 0.7 mL/min); t_R = 14.2 min (*S*, minor), t_R = 16.8 min (*R*, major).

HRMS (ESI) calcd for $C_{23}H_{24}NO$ $[M+H]^+$ 330.1858, found 330.1853.

$[\alpha]_D^{20}$ = + 39.8 (*c* = 0.47, $CDCl_3$).

(*R*, *E*)-3-(benzylimino)-1-(p-tolyl)butan-1-ol



Pale yellow oil.

IR (KBr) ν = 1053, 1160, 1661, 2942, 3401 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.12 (s, 3H), 2.59 (s, 3H), 2.70-2.84 (m, 2H), 3.51 (s, 1H), 4.77 (s, 2H), 5.12 (dd, 1H, *J* = 4.0 Hz, *J* = 9.0 Hz), 7.16-7.33 (m, 9H).

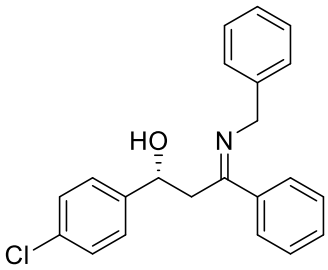
^{13}C NMR (125 MHz, $CDCl_3$); δ = 23.4, 28.6, 31.7, 53.5, 70.0, 123.1, 126.5, 126.8, 127.2, 128.4, 129.0, 138.7, 176.2.

HPLC; (Dialcel Chiralcel OD-H, ^{n}hexane/ ^{i}PrOH = 90/10, flow rate 1.0 mL/min); t_R = 9.8 min (*S*, minor), t_R = 11.8 min (*R*, major).

HRMS (ESI) calcd for $C_{18}H_{22}NO$ $[M+H]^+$ 268.1701, found 268.1697.

$[\alpha]_D^{20}$ = + 21.6 (*c* = 0.23, $CDCl_3$).

(*R*, *Z*)-3-(benzylimino)-1-(4-chlorophenyl)-3-phenylpropan-1-ol



Yellow oil.

IR (KBr) ν = 1041, 1169, 1649, 2875, 3392 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$); δ = 2.80-2.82 (m, 2H), 3.65 (d, 1H, *J* = 7.8 Hz), 4.87 (d, 2H, *J* = 2.9 Hz), 5.37 (t, 1H, *J* = 7.8 Hz), 7.19-7.61 (m, 12H), 7.95 (d, 2H, *J* = 6.9 Hz).

^{13}C NMR (125 MHz, $CDCl_3$); δ = 31.7, 53.5, 70.0, 124.9, 125.7, 126.3, 126.6, 127.7, 127.9, 128.0, 128.5, 130.0, 131.2, 133.9, 137.5, 170.3.

HPLC; (Dialcel Chiralcel OD-H, ^{n}hexane/ ^{i}PrOH = 90/10, flow rate 1.0 mL/min); t_R = 11.4 min (*S*, minor), t_R = 13.7 min (*R*, major).

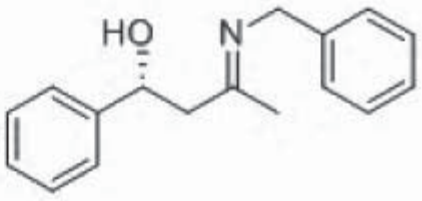
HRMS (ESI) calcd for $C_{22}H_{21}ClNO$ $[M+H]^+$ 350.1312, found 350.1332.

$[\alpha]_D^{22}$ = + 38.6 (*c* = 0.53, $CHCl_3$).

5. *References*

1 C. Sole, E. Fernández, *Chem. Asian J.*, 2009, **4**, 1790-1793.

2 V. G. Desai, P. C. Satardekar, S. Polo, K. Dhumaskar, *Synth. Commun*., 2012, **42**, 836-842.



C-R8A CHROMATOPAC CH=1 Report No.=10 DATA=I:¥CHRM1

** CALCULATION REPORT **

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1	35	10.952	634123	33551			49.7719	
	36	13.067	639934	27216	V		50.228	
		TOTAL	1274056	60767			100	

C-R8A CHROMATOPAC CH=1 Report No.=15 DATA=I:¥CHRM1 C00 14 04 22 15:19:20

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	53	13.154	237395	10182	V		100	
		TOTAL	237395	10182			100	

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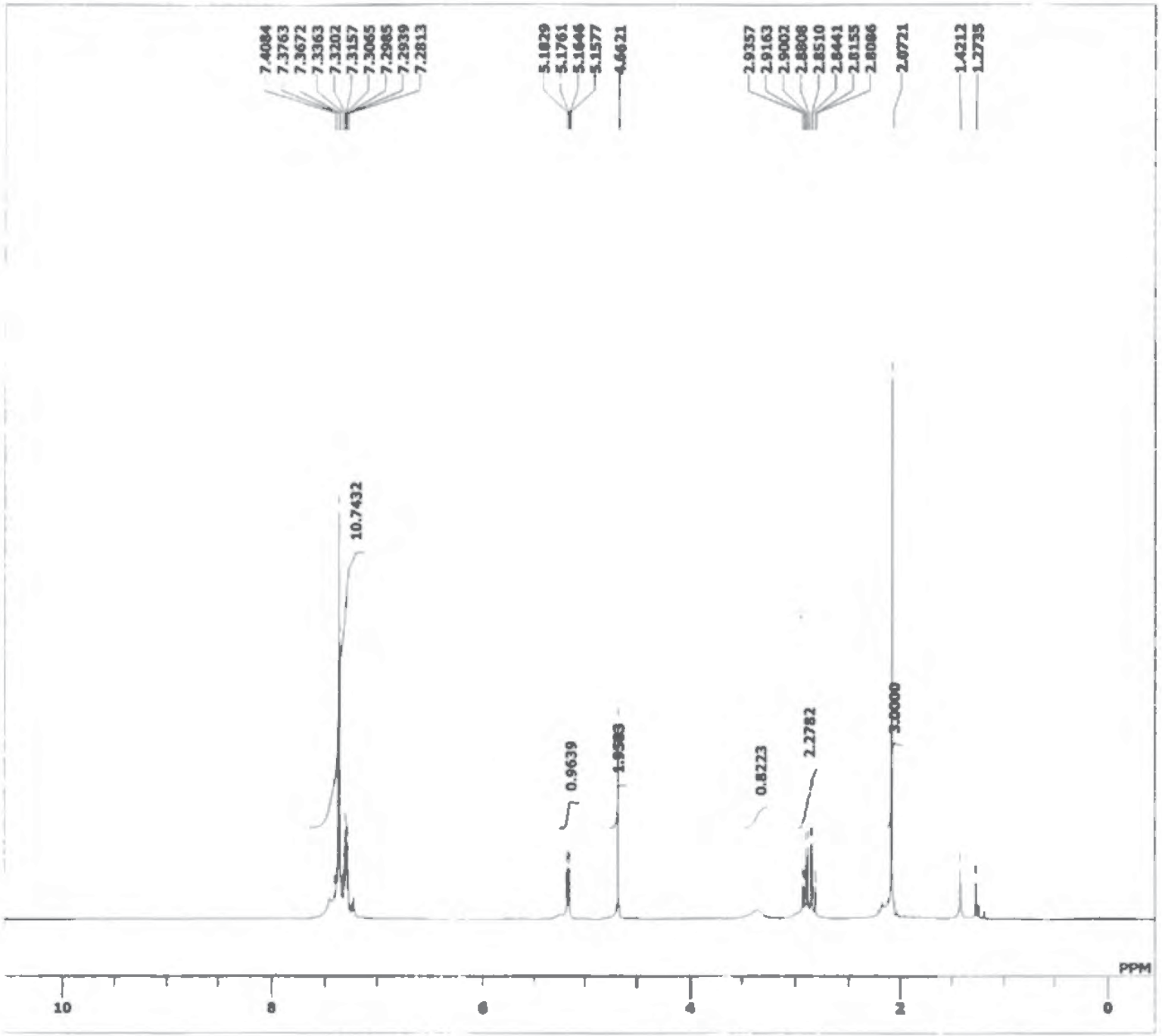

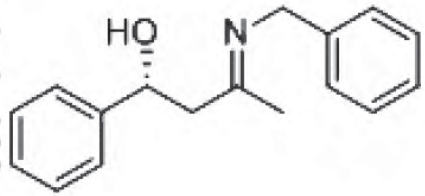

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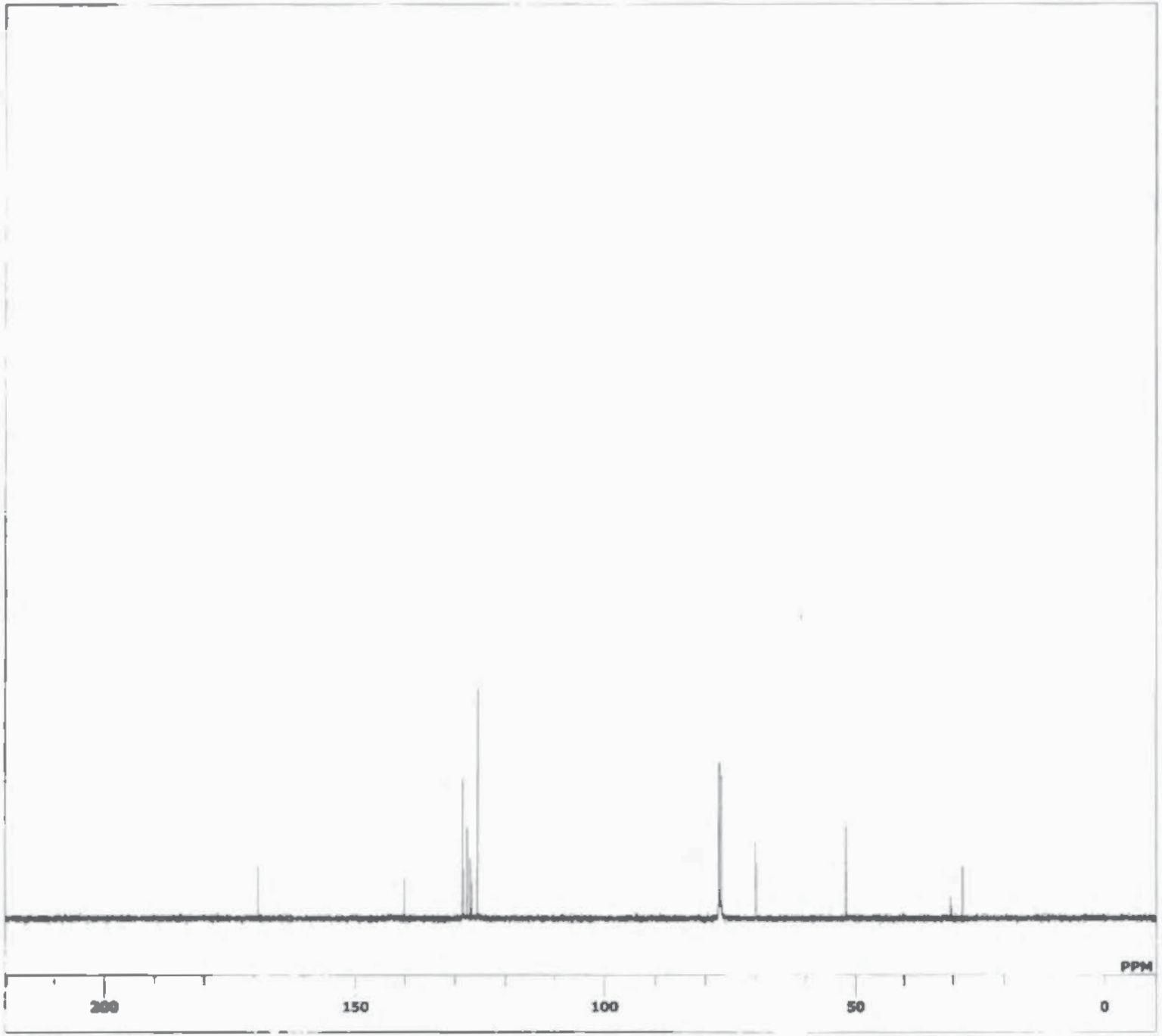

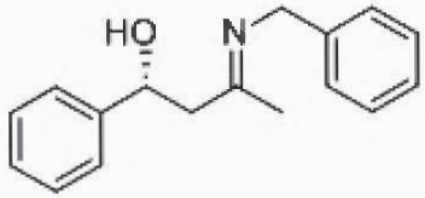

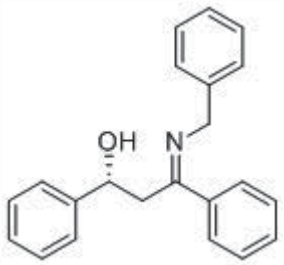

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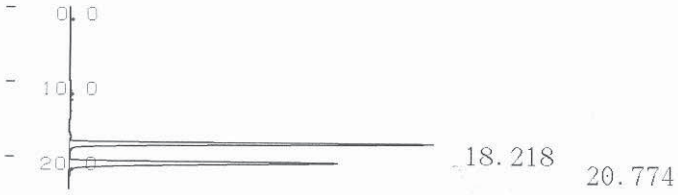

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	9	18.218	1882583	87570	V		49.9445	
	10	20.774	1886770	64573			50.0555	
		TOTAL	3769353	152143			100	

C-R8A CHROMATOPAC CH=1 Report No.=9 DATA=1:@CHRM1.C00 13/02/27 13:30:32

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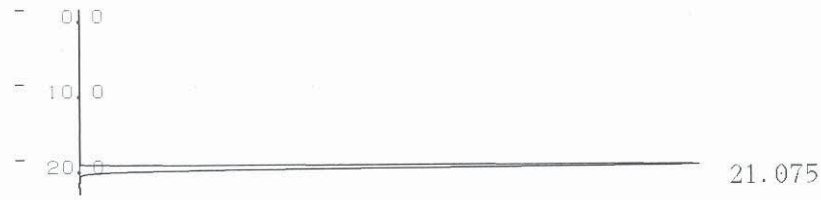
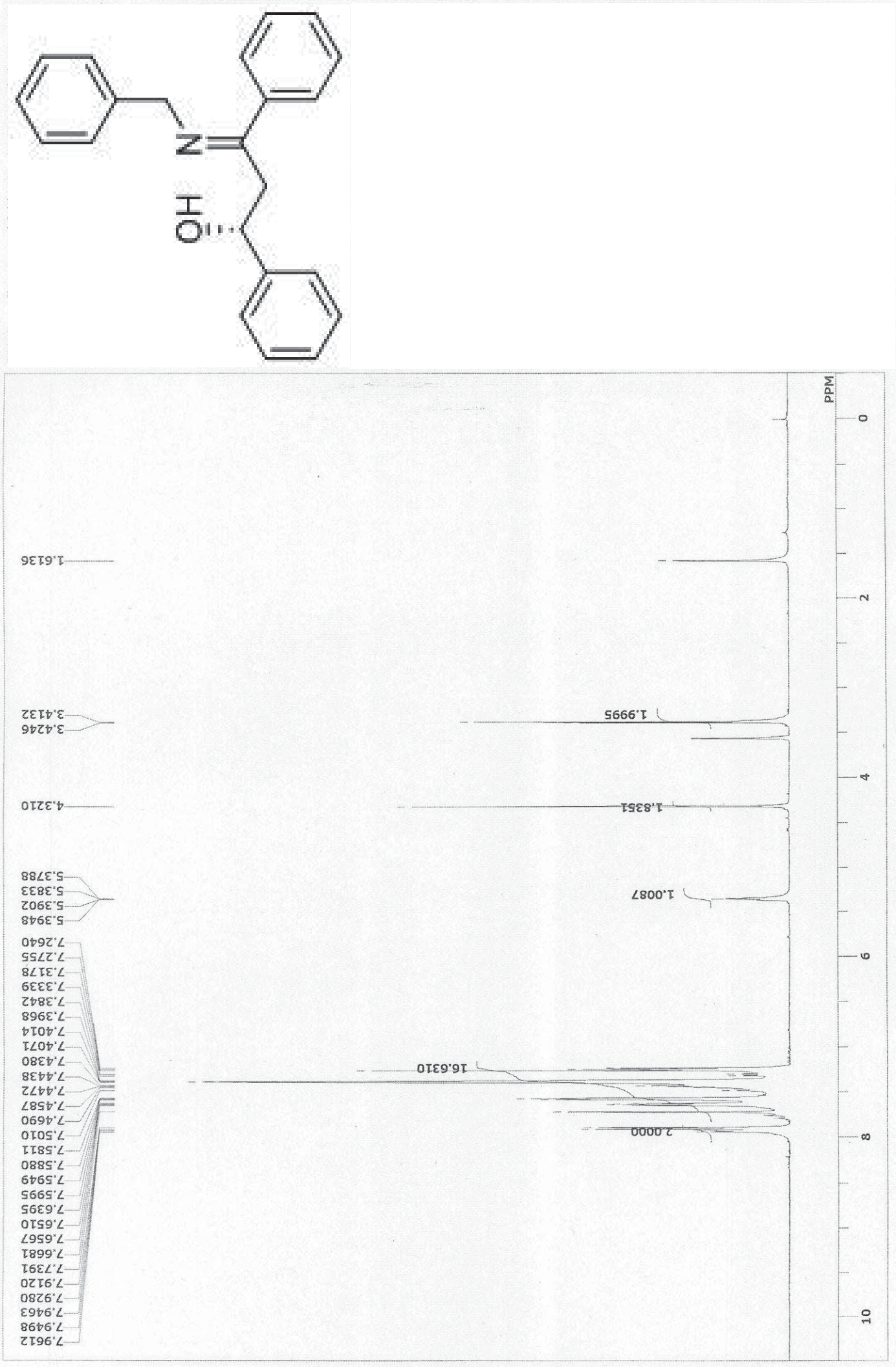


** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	18	21.075	2194315	74638			100	
		TOTAL	2194315	74638			100	

C-R8A CHROMATOPAC CH=1 Report No.=13 DATA=1:@CHRM1.C00 13/02/27 14:15:52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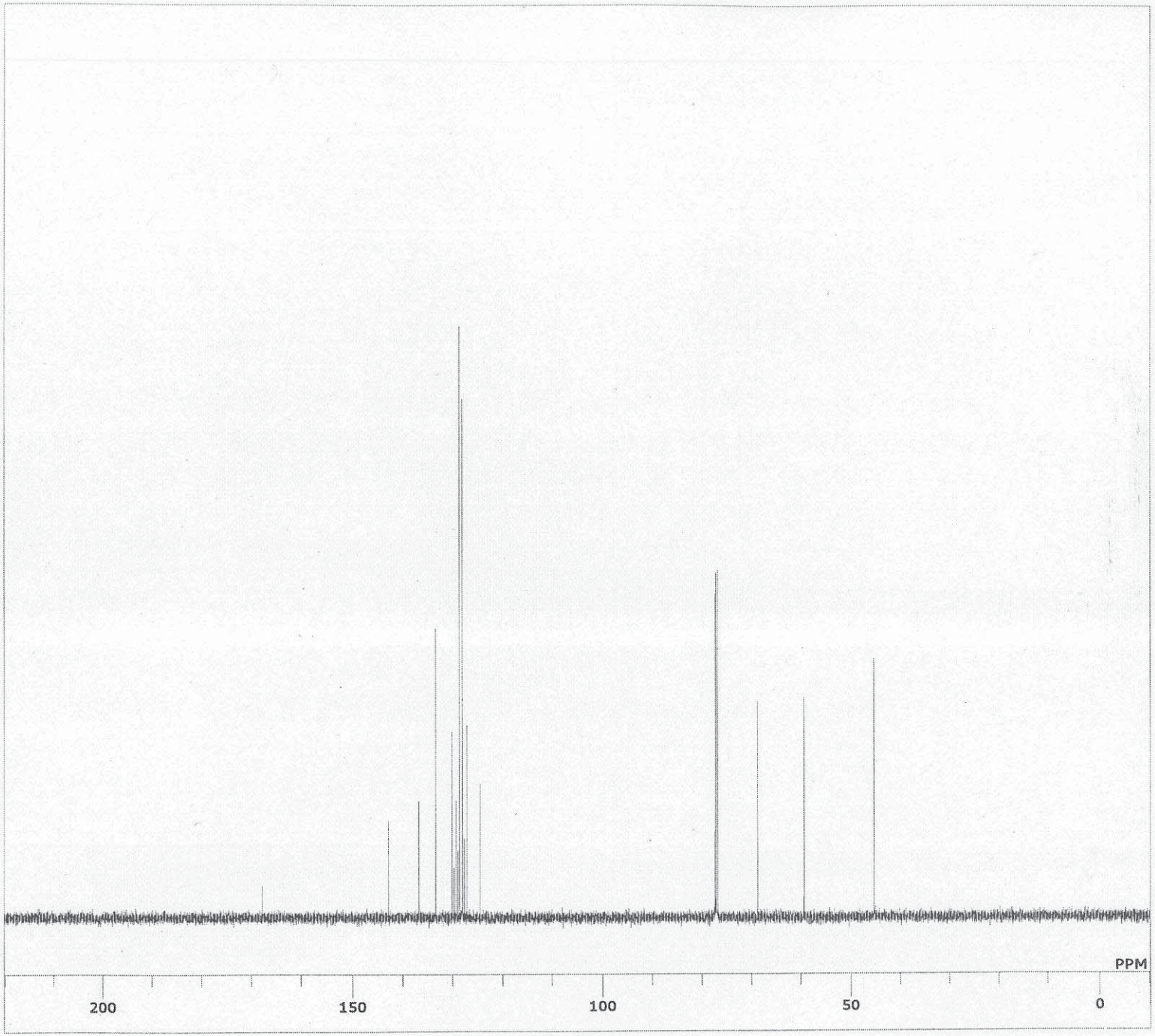

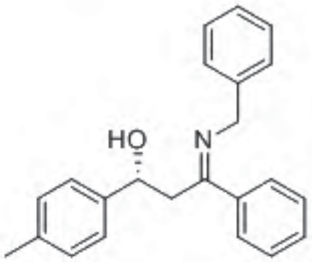

C-R8A CHROMATOPAC CH=1 Report No.-5 DATA=1:@CHRM1.C00

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	26	10.475	3996669	231048			49.7024	
	27	12.369	4044525	189107	V		50.2976	
		TOTAL	8041194	420155			100	

C-R8A CHROMATOPAC CH=1 Report No.-12 DATA=1:@CHRM1.C00 14 03 17 16:54:30

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	41	12.477	714810	31272			100	
		TOTAL	714810	31272			100	

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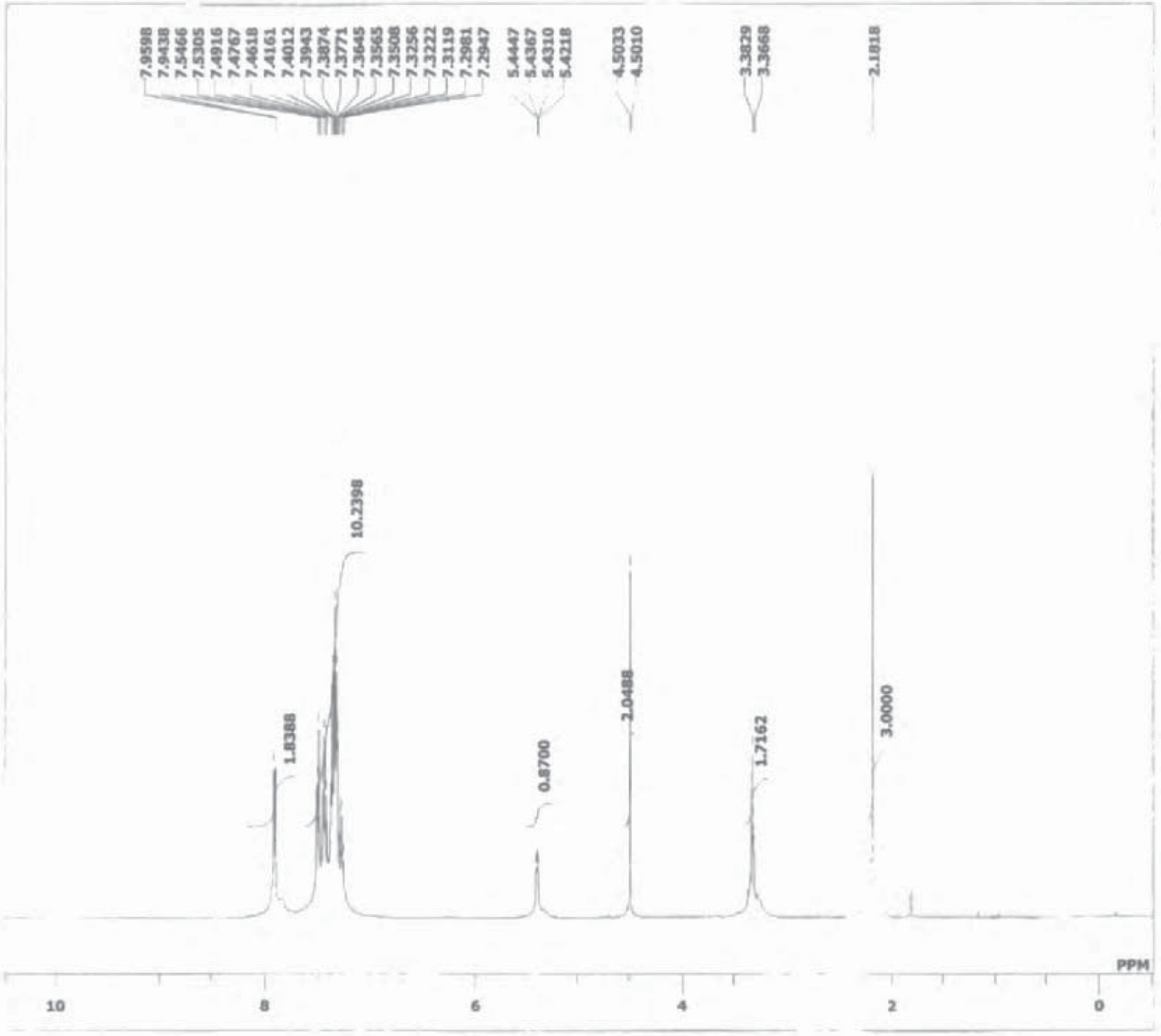

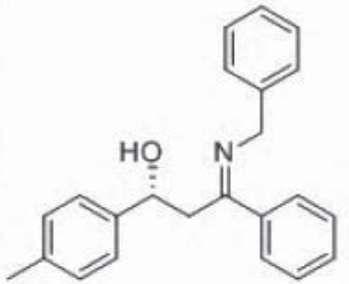

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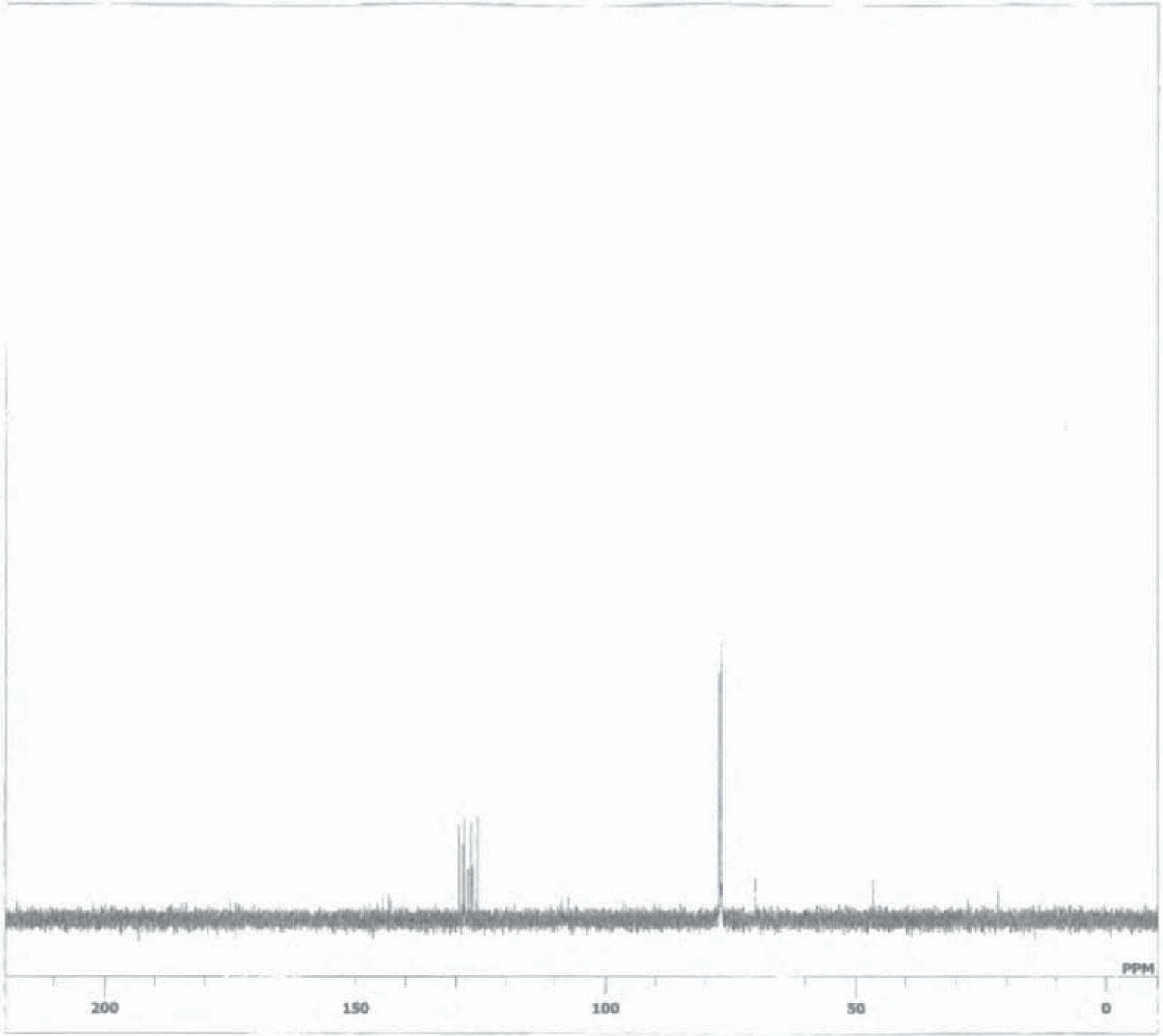

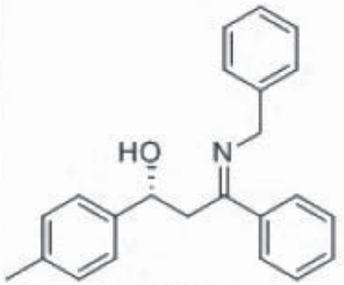

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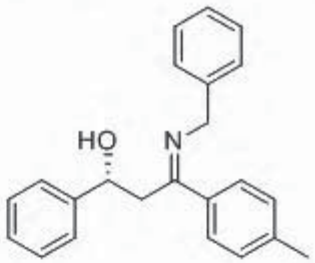

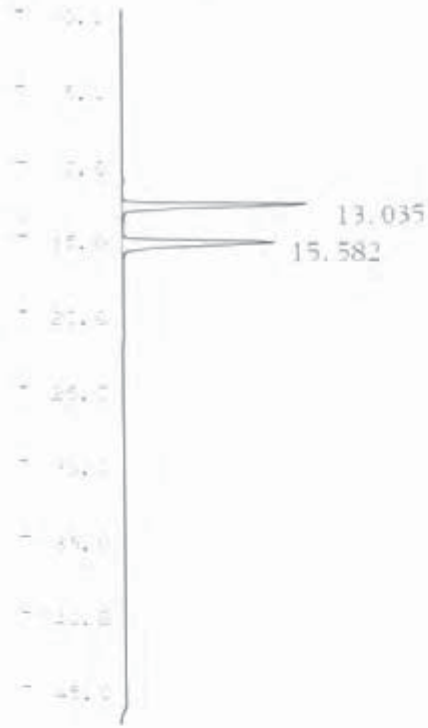

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	50	13.035	957729	44486	S		49.6724	
	52	15.582	970360	36434	SV		50.3276	
		TOTAL	1928088	80920			100	

C-R8A CHROMATOPAC CH-1 Report No.=23 DATA-1:¥CHRM1.C00 14 04 22 17:03:04

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	61	15.438	473751	16187	SV		100	
		TOTAL	473751	16187			100	

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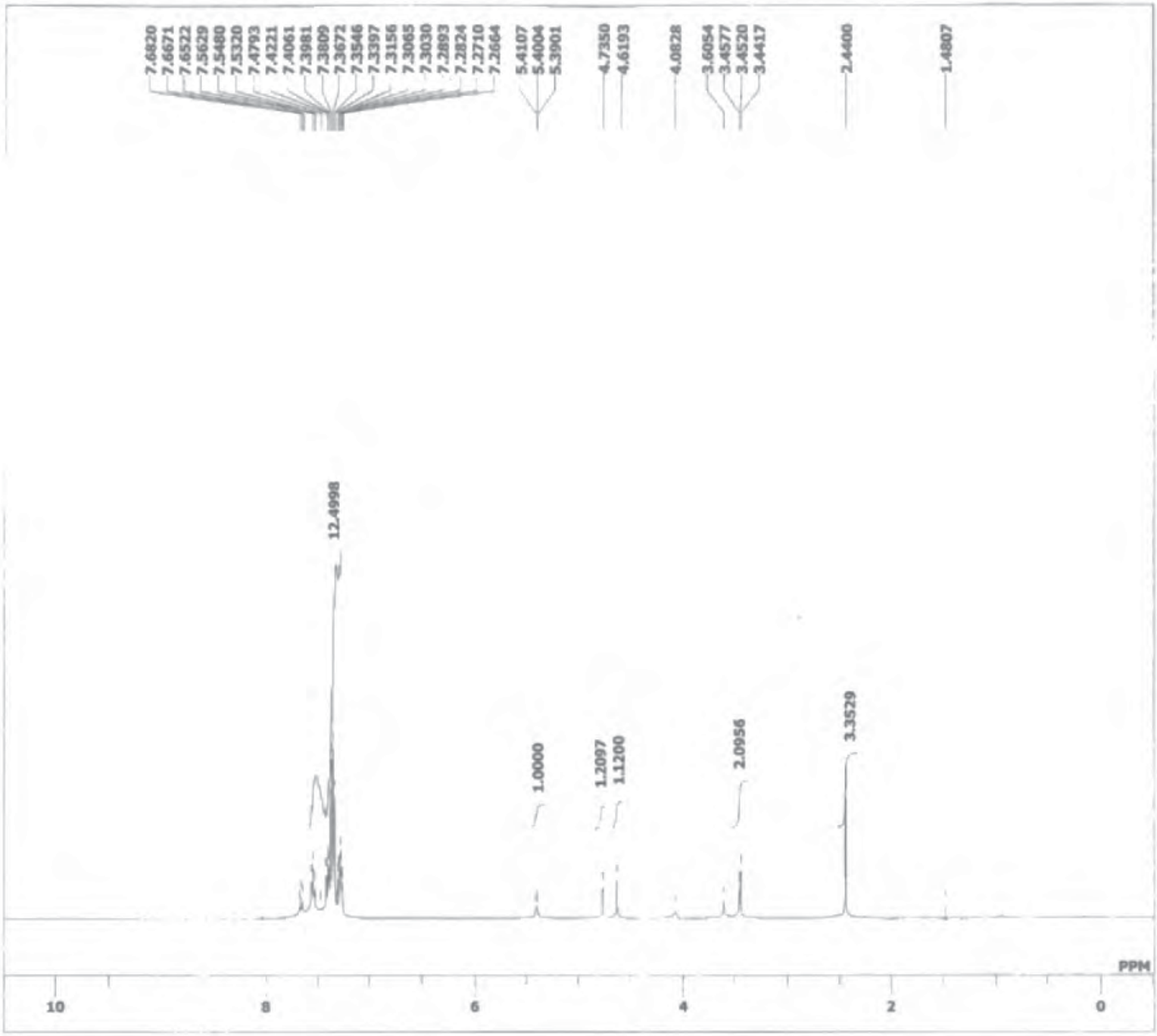

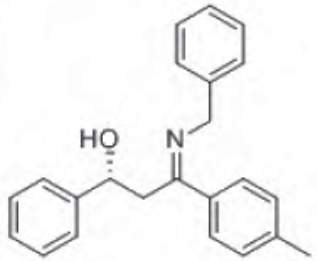

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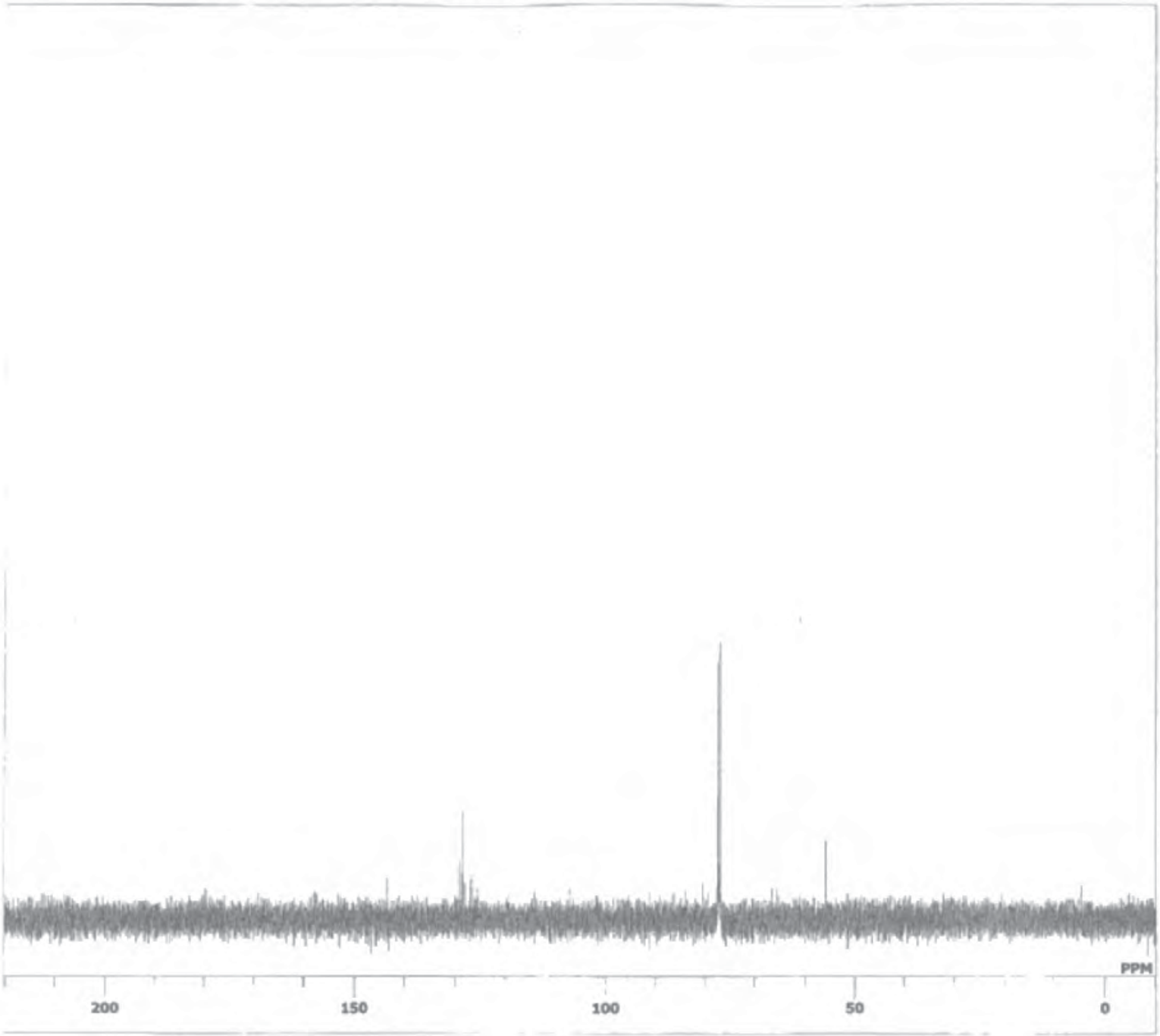

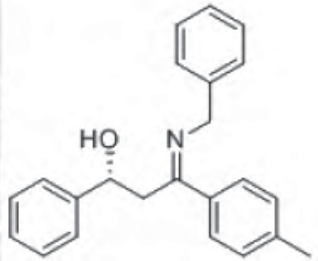

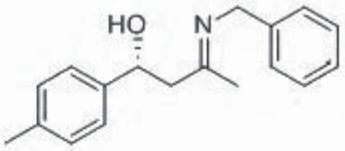

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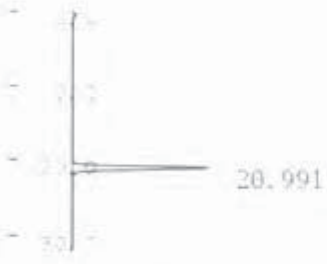
** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	9	17.71	222540	7786			49.9716	
	11	21.05	222793	6667			50.0284	
		TOTAL	445333	14453			100	

C-R8A CHROMATOPAC CH-1 Report No.=15 DATA=1:¥CHRM1.C00 14 04 22 16:11:52

Analysis FILE : 9:¥FIL15.FIL



** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	24	20.991	547691	16297			100	
		TOTAL	547691	16297			100	

C-R8A CHROMATOPAC CH-1 Report No.=21 DATA=1:¥CHRM1.C00 14 04 22 17:06:18

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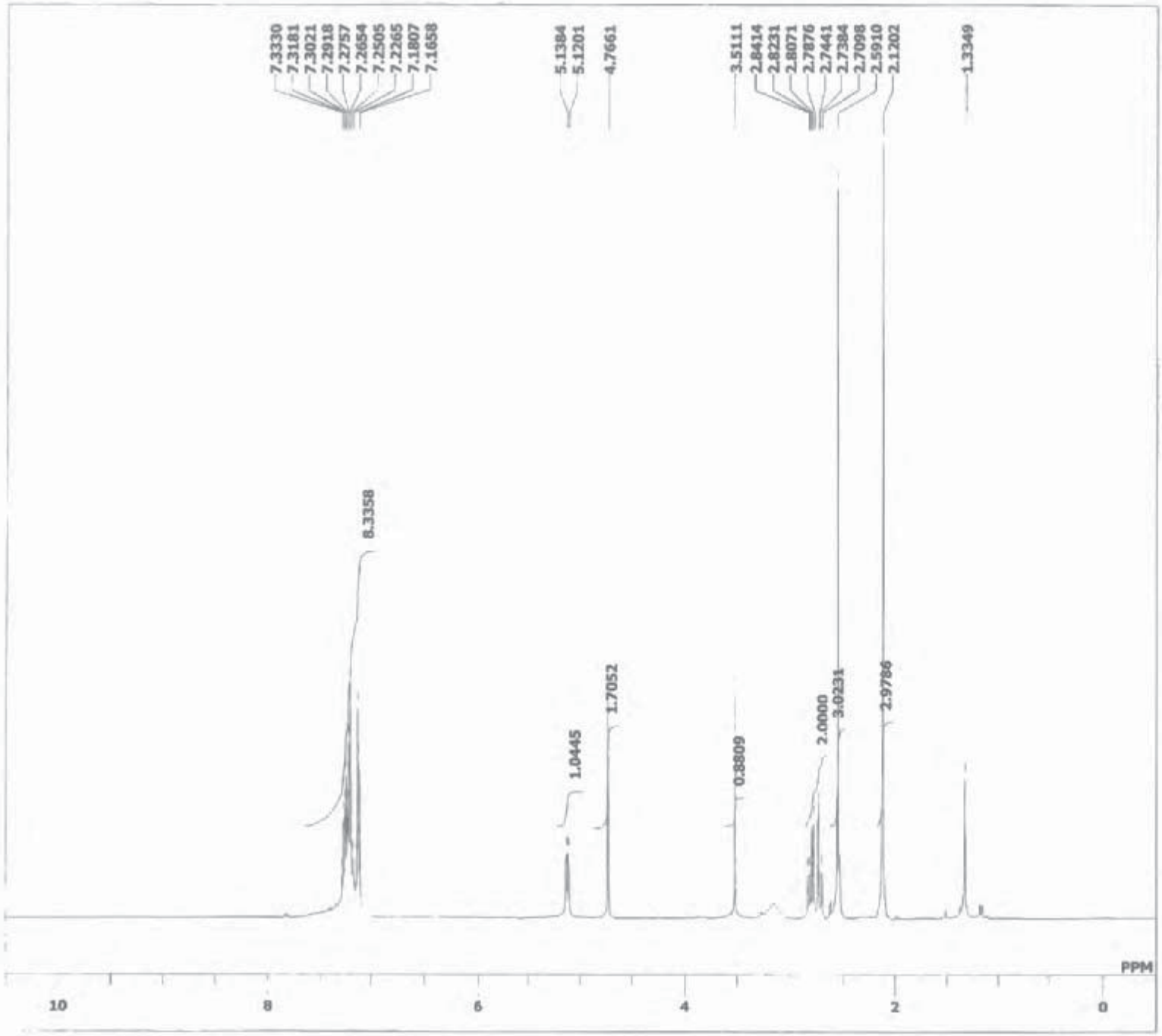

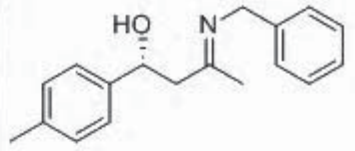

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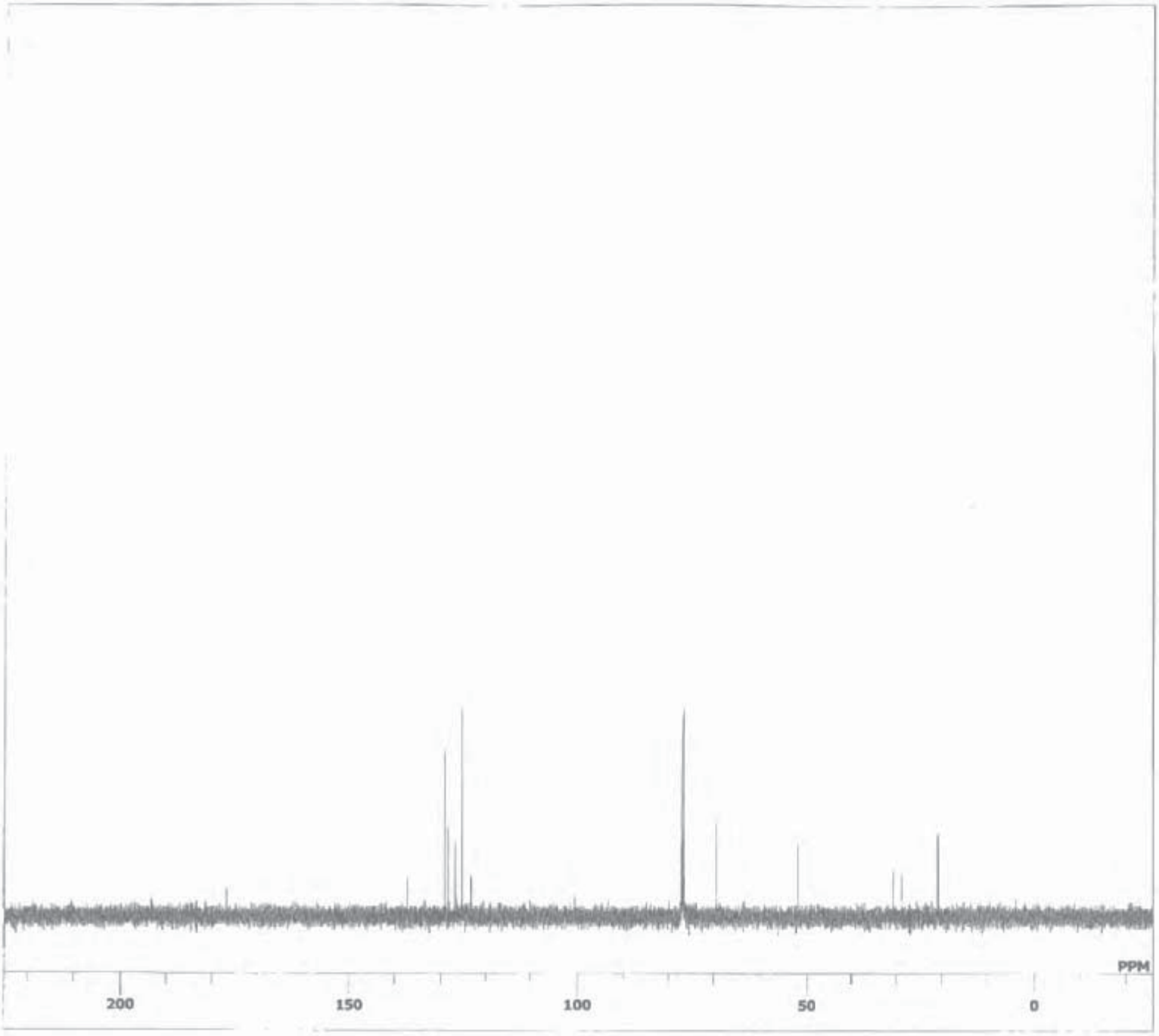

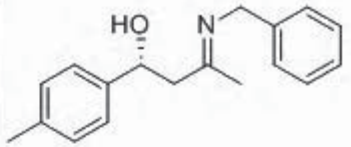

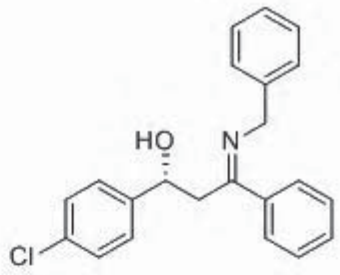

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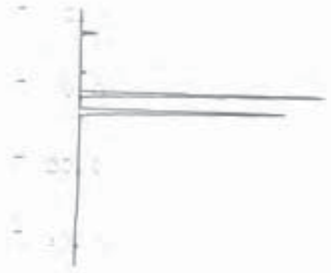

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	5	11.409	303957	14814			50.0102	
	6	13.714	303834	12502			49.9898	
		TOTAL	607791	27317			100	

C-R8A CHROMATOPAC CH=1 Report No.=20 DATA=I:@CHRM1.C00 14 03 26 05:20:10

Analysis FILE : 9:@FIL15.FIL

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	3	11.299	34244	1732			9.6033	
	4	13.529	322344	13073			90.3967	
		TOTAL	356588	14805			100	

C-R8A CHROMATOPAC CH=1 Report No.=13 DATA=I:@CHRM1.C00 14 03 26 07:18:40

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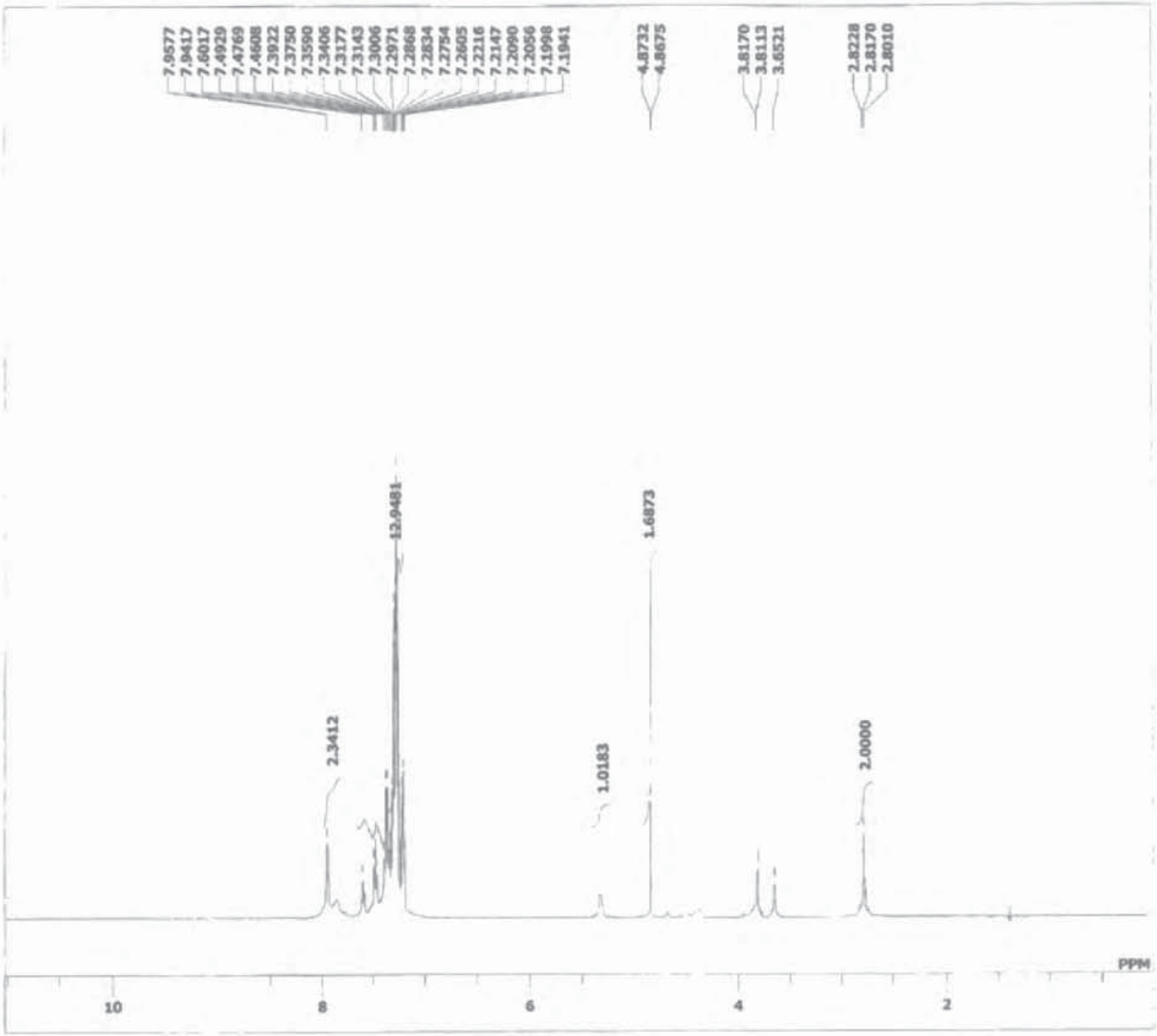

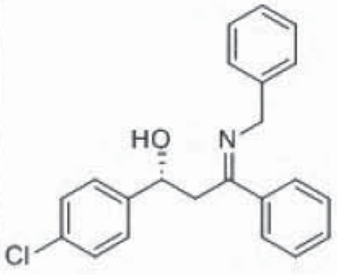

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