

Supporting Information for Ytterbium(III) Triflate/TMSCl; Efficient Catalyst for Imino Ene Reaction

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General Experimental.

All reactions were carried out under argon atmosphere. Tetrahydrofuran was distilled from sodium/ benzophenone prior to use. Dichloromethane was distilled from P2O5 and redistilled from CaH2 prior to use. Analytical TLC was performed on Merck DC-Platten Kieselgel 60 GF254 plates. Silica gel column chromatography was carried out with Merck Kieselgel (60 mesh). ¹H-NMR spectra were recorded on JNM-GSX 400 α (400 MHz). ¹³C-NMR spectra were recorded on JNM-GSX 400 α (100 MHz). IR spectra were obtained with JASCO FT/ IR-230. Mass spectra (MS) were recorded on JEOL HX-100A (LR-FAB, HR-FAB) mass spectrometer. The imines **1a**,¹ **1b**,² **1c**,¹ **1d**,³ and **1e**,⁴ were prepared by known methods.

1 Jennings, W. B; Lovely, C. J. *Tetrahedron* **1991**, *47*, 5561-5568.

2 Kupfer, R.; Meier, S.; Wüthwein, E. U. *Synthesis* **1984**, 688-690.

3 Texier-Boullet, F. *Synthesis* **1985**, 679-681.

4 Bigelow, L. A.; Eatough, H. *Org. Synth.* **1941**, I, 80-81.

General Procedure of Imino Ene Reaction

Dichloromethane (4 mL) solution of imine (0.5 mmol) was added to the tetrahydrofuran (1 mL) solution of ytterbium(III) trifluoromethanesulfonate ($\text{Yb}(\text{OTf})_3$). Then silyl reagent and α -methylstyrene (1.0 mmol) were added successively. Then the reaction mixture was stirred at room temperature. The reaction was monitored by TLC analysis. After usual work-up, obtained crude residue was purified by column chromatography.

General Procedure of Three-Component Coupling

Benzaldehyde (1.0 mmol) was added to the dichloromethane (8 mL) and tetrahydrofuran (2 mL) solution of ytterbium(III) trifluoromethanesulfonate ($\text{Yb}(\text{OTf})_3$) and p-toluenesulfonamide (1.0 mmol). Then silyl reagent and *a*-methylstyrene (2.0 mmol) were added successively. Then the reaction mixture was stirred at room temperature. The reaction was monitored by TLC analysis. After usual work-up, obtained crude residue was purified by column chromatography.

Chemical yield of Figure 1

with TMSCl : Sc(OTf)₃ : 88%, Y(OTf)₃ : 67%, La(OTf)₃ : 17%, Ce(OTf)₃ : 21%, Pr(OTf)₃ : 34%, Nd(OTf)₃ : 25%, Sm(OTf)₃ : 26%, Eu(OTf)₃ : 42%, Gd(OTf)₃ : 30%, Tb(OTf)₃ : 34%, Dy(OTf)₃ : 37%, Ho(OTf)₃ : 47%, Er(OTf)₃ : 53%, Tm(OTf)₃ : 58%, Yb(OTf)₃ : 93%, Lu(OTf)₃ : 63%.

Spectral Data

2,4-Diphenyl-4-tosylamino-1-butene (**2a**)

pale yellow oil; IR (neat) 3278, 3031, 2925, 1599, 1495, 1443, 1323, 1158, 1093, 963, 904, 698, 663 cm^{-1} ; ¹H-NMR (CDCl_3) δ 2.33 (3H, s), 2.89 (2H, m), 4.23 (1H, dd, J = 7.2, 12.9 Hz), 4.94 (1H, d, J = 1.0 Hz), 5.17 (1H, brs), 5.22 (1H, d, J = 1.2 Hz), 7.02-7.05 (4H, m), 7.13-7.26 (8H, m), 7.44 (2H, d, J = 8.0 Hz); ¹³C-NMR (CDCl_3) δ 21.37, 44.05, 56.36, 111.34, 126.07, 126.60, 127.04, 127.35, 127.65, 128.25, 128.37, 129.18, 136.93, 139.26, 140.65, 142.86, 143.70; LRMS (FAB) m/z 376 [5, $\text{M}^+ \text{- H}$], 260 [100]; HRMS ($\text{C}_{23}\text{H}_{22}\text{NO}_2\text{S}$) found 376.1371; calcd 376.1371.

2,4-Diphenyl-4-methoxycarbonylamino-1-butene (**2b**)

colorless oil; IR (neat) 3328, 3030, 1718, 1701, 1543, 1509, 1458, 1363, 1252, 1193, 904, 778, 699 cm^{-1} ; ¹H-NMR (CDCl_3) δ 2.95 (2H, d, J = 6.8 Hz), 3.57 (3H, s), 4.72 (1H, brs), 4.94 (1H, brs), 5.00 (1H, d, J = 1.2 Hz), 5.27 (1H, d, J = 1.2 Hz), 7.18-7.37 (10H, m); ¹³C-NMR (CDCl_3) δ 43.23, 51.89, 54.20, 115.59, 126.30, 127.31, 127.69, 128.44, 128.50, 140.49, 142.35, 144.86, 156.24; LRMS (FAB) m/z 282 [7, $\text{M}^+ \text{+ H}$], 164 [100]; HRMS ($\text{C}_{18}\text{H}_{20}\text{NO}_2$) found 282.1504; calcd 282.1494.

2,4-Diphenyl-4-diphenylphosphinoylamino-1-butene (2c**)**

white powder; IR (KBr) 3446, 3135, 2918, 1437, 1187, 1125, 1090, 900, 778, 749, 694 cm⁻¹; ¹H-NMR (CDCl₃) δ 2.99 (1H, dd, *J* = 8.2, 14.0 Hz), 3.24 (1H, dd, *J* = 6.3, 13.8 Hz), 3.35 (1H, brs), 4.27 (1H, dd, *J* = 6.5, 8.4 Hz), 4.87 (1H, d, *J* = 0.9 Hz), 5.21 (1H, d, *J* = 1.2 Hz), 7.06 (2H, d, *J* = 6.4 Hz), 7.16-7.75 (18H, m); ¹³C-NMR (CDCl₃) δ 45.51, 54.75, 115.96, 126.22, 126.61, 127.19, 127.51, 128.30, 128.35, 128.40, 128.47, 131.78, 131.86, 131.95, 132.22, 132.31, 140.00, 142.89, 144.37; LRMS (FAB) *m/z* 424 [20, M⁺+H], 306 [100]; HRMS (C₂₈H₂₇NOP) found 424.1817; calcd 424.1830.

2,4-Diphenyl-4-methyl-1,2,3,4-tetrahydroquinoline

(3**, major diastereomer)**

yellow solid; IR (KBr) 3383, 2966, 1602, 1495, 1474, 1345, 1311, 1256, 1113, 1028, 752, 702 cm⁻¹; ¹H-NMR (CDCl₃) δ 1.82 (3H, s), 1.95 (1H, dd, *J* = 2.7, 13.4 Hz), 2.26 (1H, dd, *J* = 12.7, 12.7 Hz), 4.60 (1H, dd, *J* = 2.7, 12.0 Hz), 6.58 (1H, d, *J* = 8.5 Hz), 7.00 (1H, ddd, *J* = 1.6, 7.5, 7.6 Hz), 7.14-7.37 (10H, m), 7.44 (2H, d, *J* = 7.3 Hz); ¹³C-NMR (CDCl₃) δ 29.73, 42.28, 48.30, 54.01, 114.50, 117.63, 125.82, 126.66, 126.86, 127.20, 127.32, 127.48, 127.63, 127.96, 128.58, 143.94, 144.73, 150.19; LRMS (FAB) *m/z* 299 [100, M⁺]; HRMS (C₂₂H₂₁N) found 299.1659; calcd 299.1674.

2,4-Diphenyl-4-methyl-1,2,3,4-tetrahydroquinoline

(3**, minor diastereomer)**

yellow solid; IR (KBr) 3385, 2958, 1602, 1482, 1444, 1338, 1312, 1259, 1119, 1030, 770, 751, 702 cm⁻¹; ¹H-NMR (CDCl₃) δ 1.75 (3H, s), 2.14 (1H, dd, *J* = 11.7, 13.1 Hz), 2.26 (1H, dd, *J* = 3.1, 13.1 Hz), 4.01 (1H, dd, *J* = 2.9, 11.7 Hz), 6.60 (1H, dd, *J* = 1.2, 8.5 Hz), 6.75 (1H, ddd, *J* = 1.2, 7.6, 7.6 Hz), 7.09-7.13 (3H, m), 7.16-7.32 (9H, m); ¹³C-NMR (CDCl₃) δ 29.62, 41.57, 47.93, 53.12, 114.19, 117.25, 125.77, 126.40, 126.63, 127.08, 127.53, 127.55, 128.16, 128.24, 128.53, 144.27, 144.56, 150.41; LRMS (FAB) *m/z* 299 [100, M⁺]; HRMS (C₂₂H₂₁N) found 299.1672; calcd 299.1674.

2-Phenyl-4-tosylamino-1-pentene (4**)**

IR (neat) 3278, 2973, 2929, 1599, 1495, 1426, 1323, 1161, 1092, 991, 906, 815, 758, 706, 664 cm⁻¹; ¹H NMR (CDCl₃) δ 0.98 (3H, d, *J* = 6.6 Hz), 2.31 (3H, s), 2.40 (1H, dd, *J* = 7.7, 14.1 Hz), 2.65 (1H, dd, *J* = 16.4, 13.4 Hz), 3.18 (1H, ddq, *J* = 6.6, 6.7, 7.0 Hz), 4.66 (1H, brs), 4.95 (1H, s), 5.20 (1H, s), 7.08-7.17 (7H, m), 7.56-7.58 (2H, m); ¹³C NMR (CDCl₃) δ 21.21, 21.43, 43.44, 48.08, 115.73, 125.98, 127.03, 127.61, 128.33, 129.48, 137.47, 139.39, 143.06, 144.33; LRMS (FAB) *m/z* 315 [40, M⁺], 198 [100]; HRMS (C₁₈H₂₁NO₂S) found 315.1301; calcd 315.1293.

2-Phenyl-4-tosylamino-5-methyl-1-hexene (5**)**

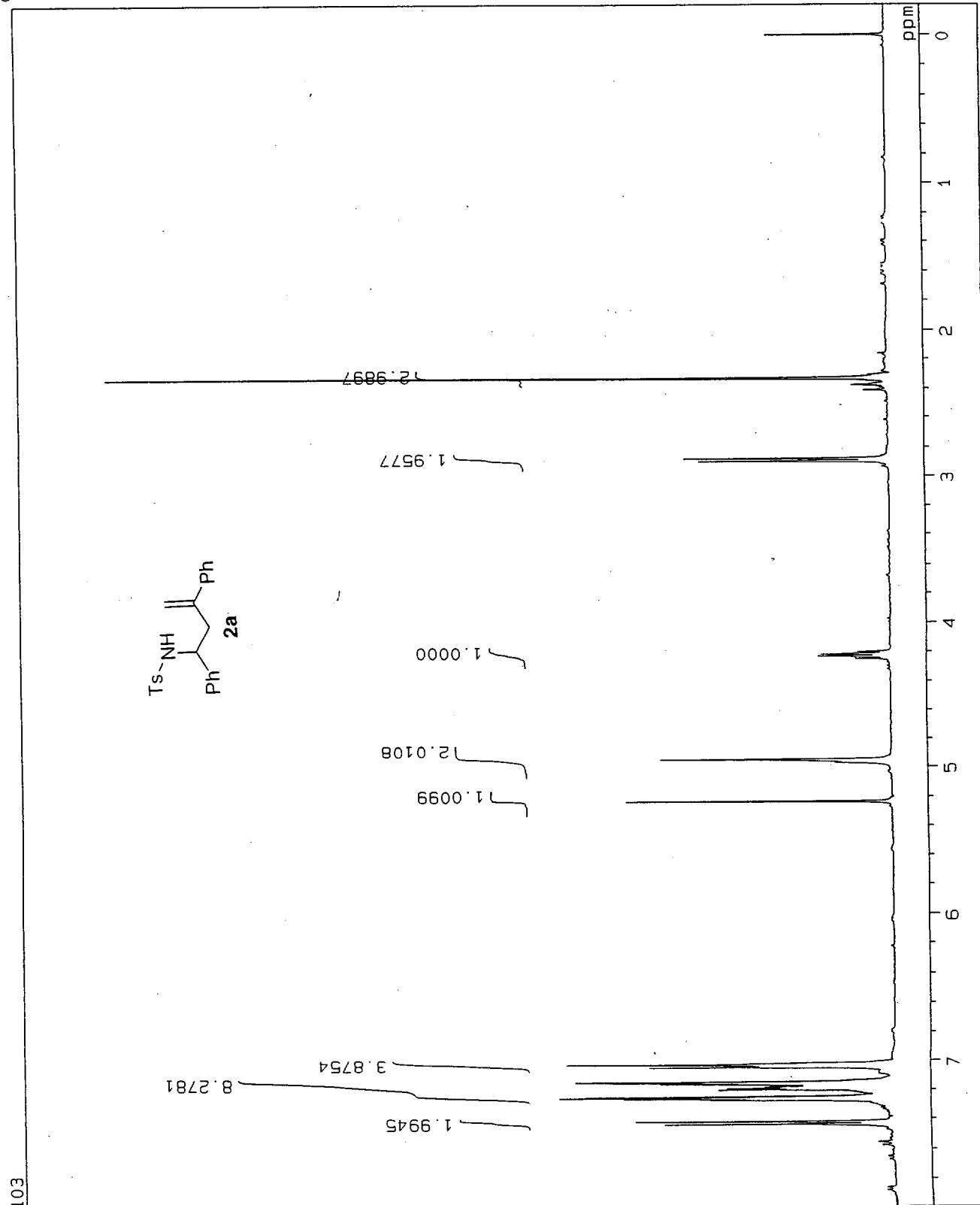
colorless solid, mp. 104-105 °C (acetone/*n*-hexane); IR (KBr) 3290, 2952, 1629, 1602, 1464, 1420, 1319, 1162, 1095, 1056, 956, 906, 812, 783, 715, 666 cm⁻¹; ¹H NMR (CDCl₃) δ 0.72 (3H, d, *J* = 6.8 Hz), 0.82 (3H, d, *J* = 6.8 Hz), 1.86 (1H, m), 2.40, (3H, s), 2.54 (1H, ddd, *J* = 1.0, 7.1, 14.4 Hz), 2.60 (1H, ddd, *J* = 1.0, 7.9, 14.4 Hz), 3.10 (1H, dddd, *J* = 3.4, 7.4, 7.4, 7.4 Hz), 4.57 (1H, d, *J* = 7.6 Hz), 5.01 (1H, d, *J* = 1.2 Hz), 5.22 (1H, d, *J* = 1.5 Hz), 7.11-7.27 (7H, m), 7.64 (2H, d, *J* = 8.3 Hz); ¹³C NMR (CDCl₃) δ 16.29, 18.30, 21.47, 29.53, 37.77, 56.76, 115.59, 126.07, 127.21, 127.61, 128.29, 129.41, 137.65, 139.44, 143.01, 144.64; LRMS (FAB) *m/z* 343 [35, M⁺], 226 [100]; HRMS (C₂₀H₂₅NO₂S) found 343.1606; calcd 343.1606.

JEOL
19-AUG-1999 19: 10: 20.56

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PW1 : 4.90 usec

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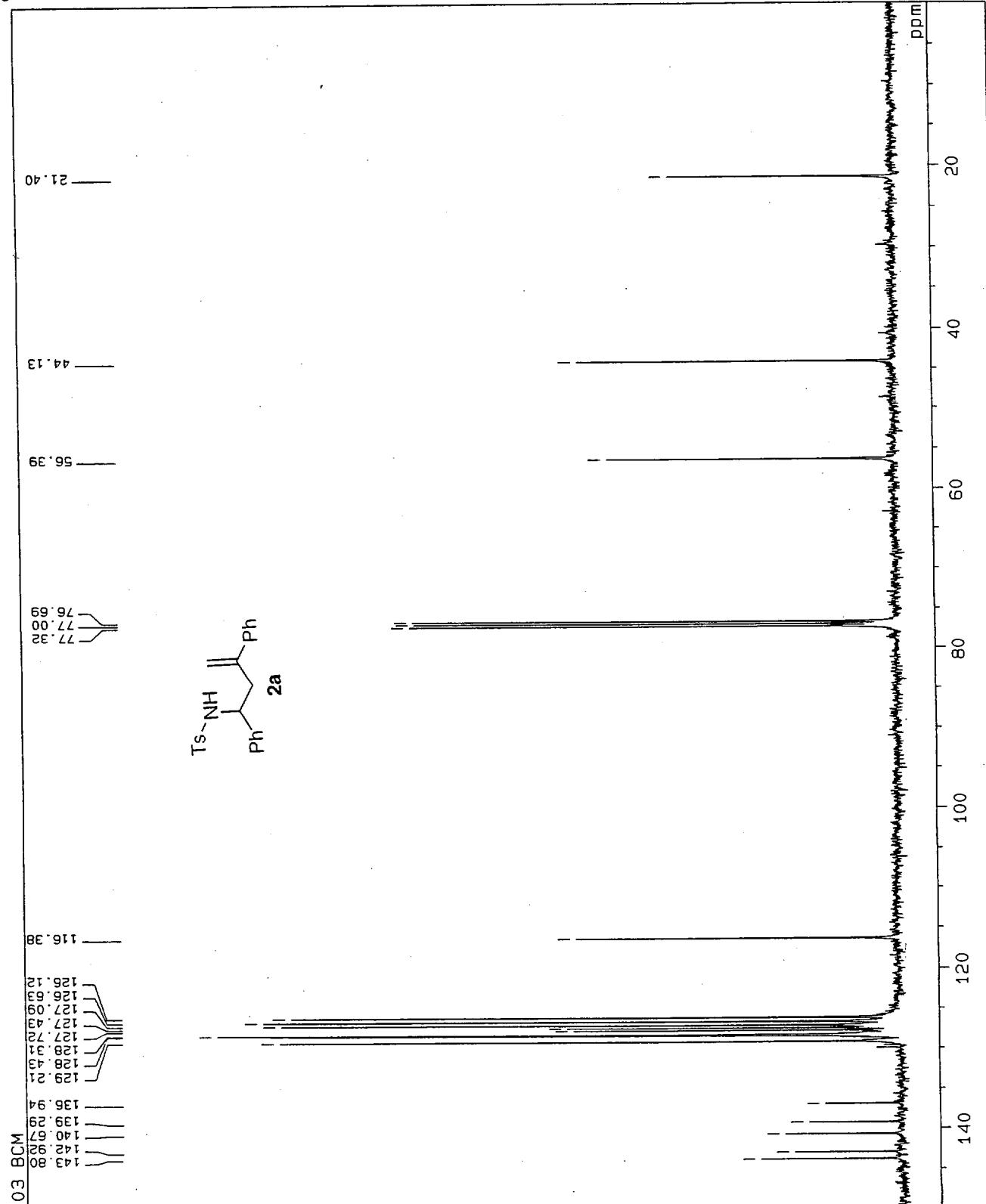
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XS : 428.25 Hz
operator



20-AUG-1999 12: 14: 17.11
QSOOL

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 PW1 : 4.55 usec

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 operator



JECOL

20-AUG-1999 19: 24: 57.55

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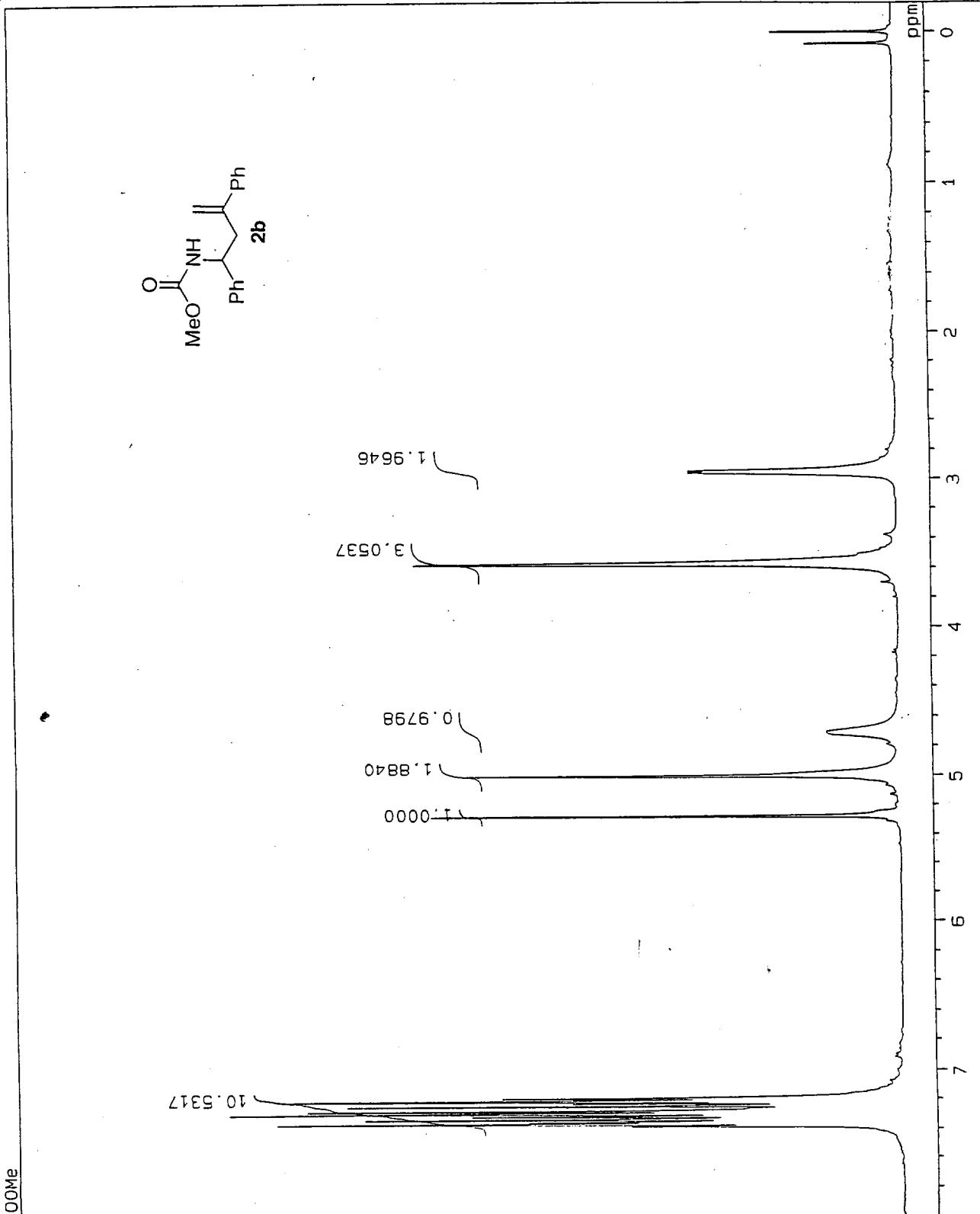
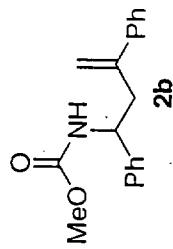
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 OBSET : 134500.00 Hz

IRNUC : 1H
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 IRATN : 511
 IRAPW : 50.0 usec
 IRBP1 : 25
 IRBP2 : 6
 ITRANS : 0

ADBT : 16
 CTEMP : 28.4 °C
 CSPED : 13 Hz
 SLVNT : CDCL₃

RESOL : 0.31 Hz
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 XS : 426.03 Hz
 operator



COOME

DEOL

18-OCT-1978 08: 09: 16.67

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BC1

BC1</div

JEOIL 97.111 - 1999 07: 59: 12.18

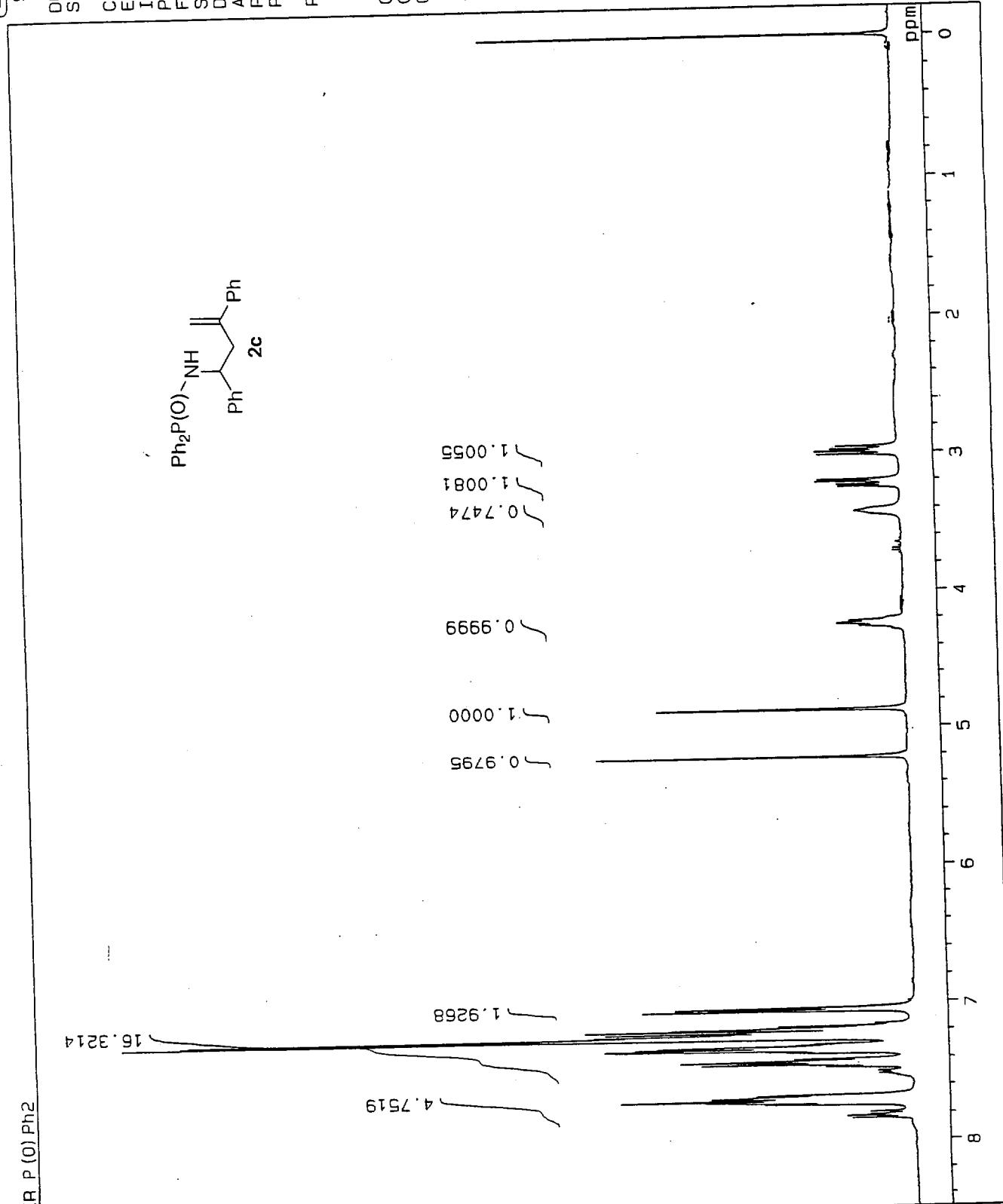
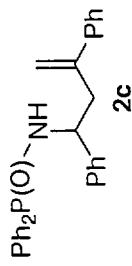
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RGAIN : 16

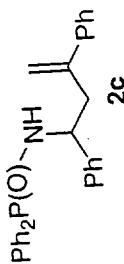
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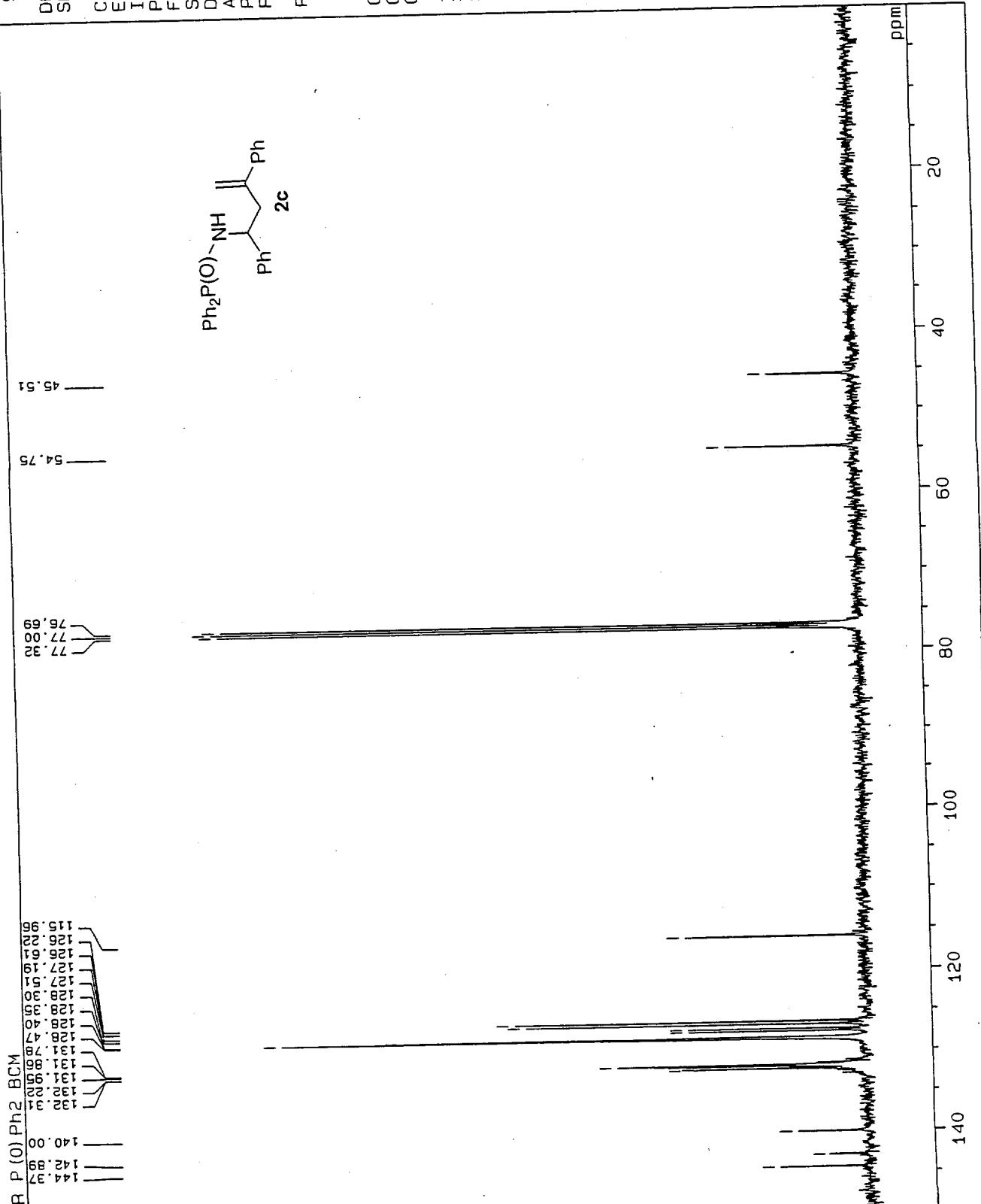


JEOL
9-JUL-1999 08: 40: 17.91

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 ACQTM : 1.2091 sec
 PD : 0.3000 sec
 AGAIN : 23
 PW1 : 4.55 usec



OBNUC : ^{13}C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz
 IRNUC : ^1H
 IRFRQ : 339.65 MHz
 IRSET : 134300.00 Hz
 IRATN : 51.1
 IRAPW : 50.0 usec
 IRBP1 : 25
 IRBP2 : 6
 IRANS : 0
 ADBIT : 16
 CTEMP : 24.7 °C
 CSPED : 1.1 Hz
 SLVNT : CDCL₃



DJEDOL
2-AUG-1999 08: 20: 19.43

1H SINGL NON MY-AZAB

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TRMOD : NON
POINT : 16384
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SCANS : 16
DUMMY : 4
ACQTM : 2.0495 sec
PD : 3.9504 sec
RGAIN : 14

PW1 : 4.90 usec

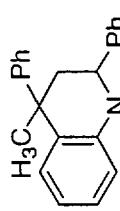
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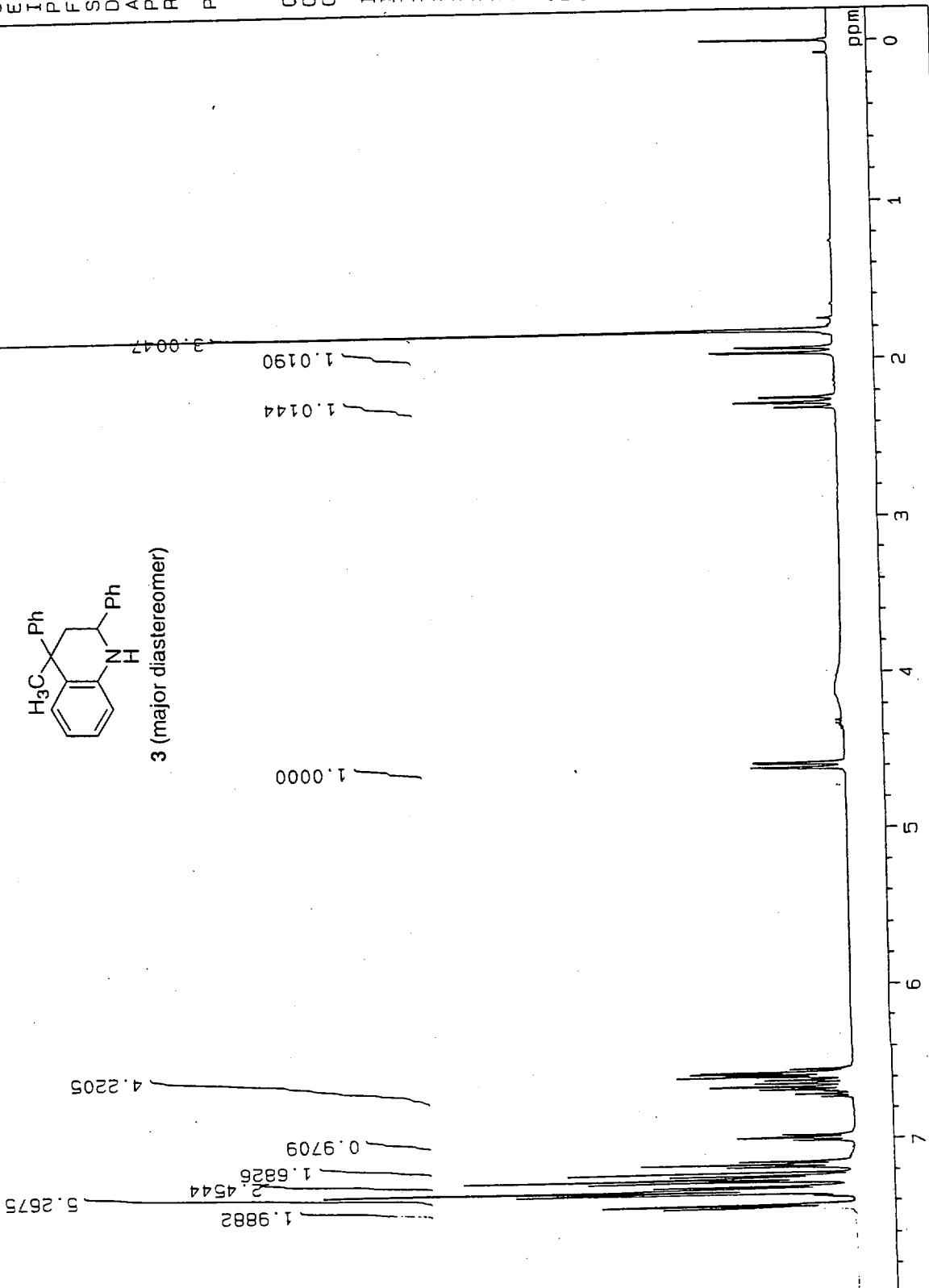
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XE : 3277.65 Hz
XS : 416.66 Hz

operator



3 (major diastereomer)



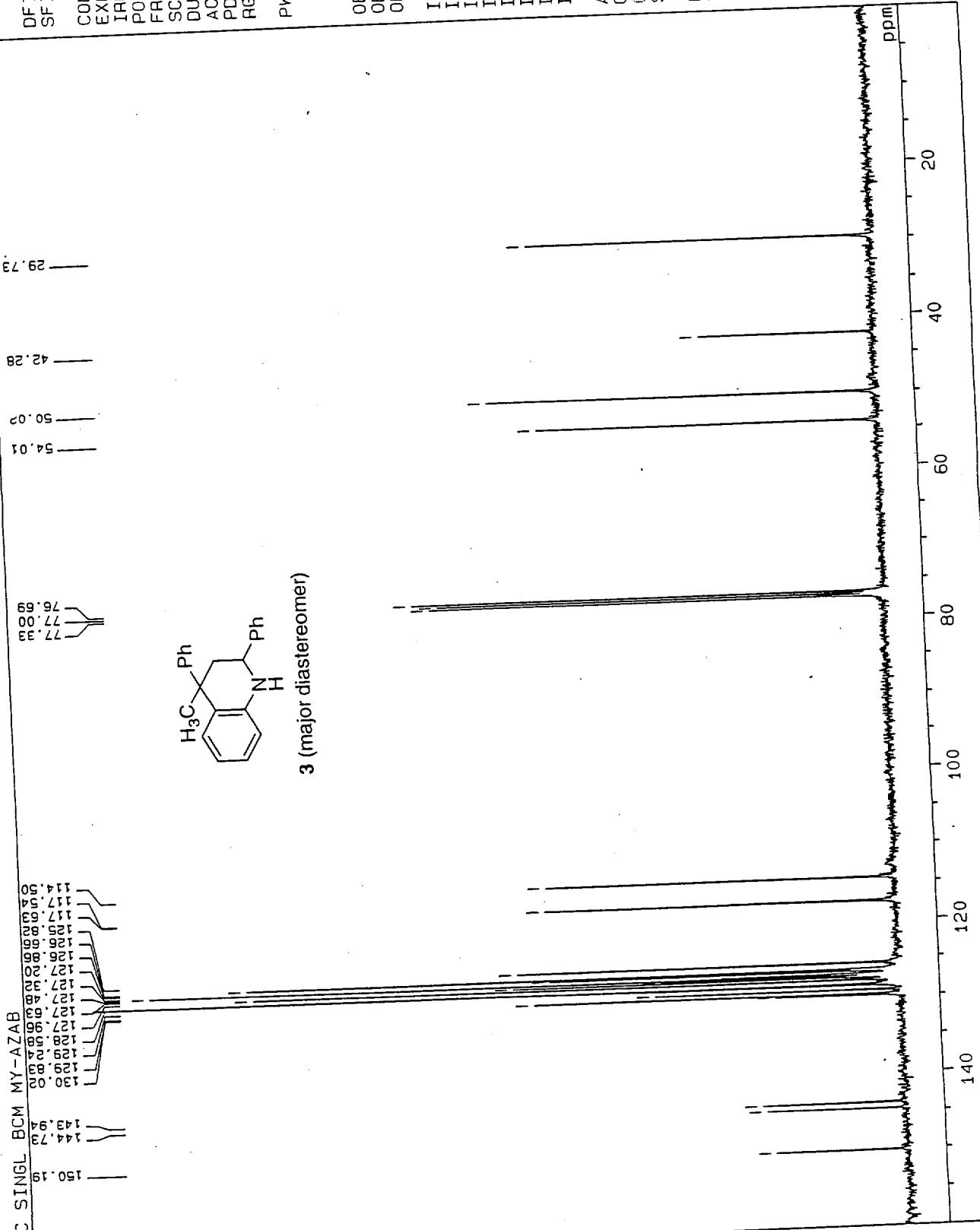
JECOL

2-AUG-1999 0B: 26: 15.15

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 AGAIN : 23
 PW1 : 4.55 usec

OBNUC : 13C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz
 IRNUC : 1H
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 IRBP1 : 25
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 CSPED : 12 Hz
 SLVNT : CDCl₃

RESOL : 1.65 Hz
 BF : 3.00 Hz
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 XE : 16082.52 Hz
 XS : 2063.45 Hz
 operator



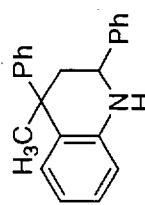
AZA D-A A

JEOL

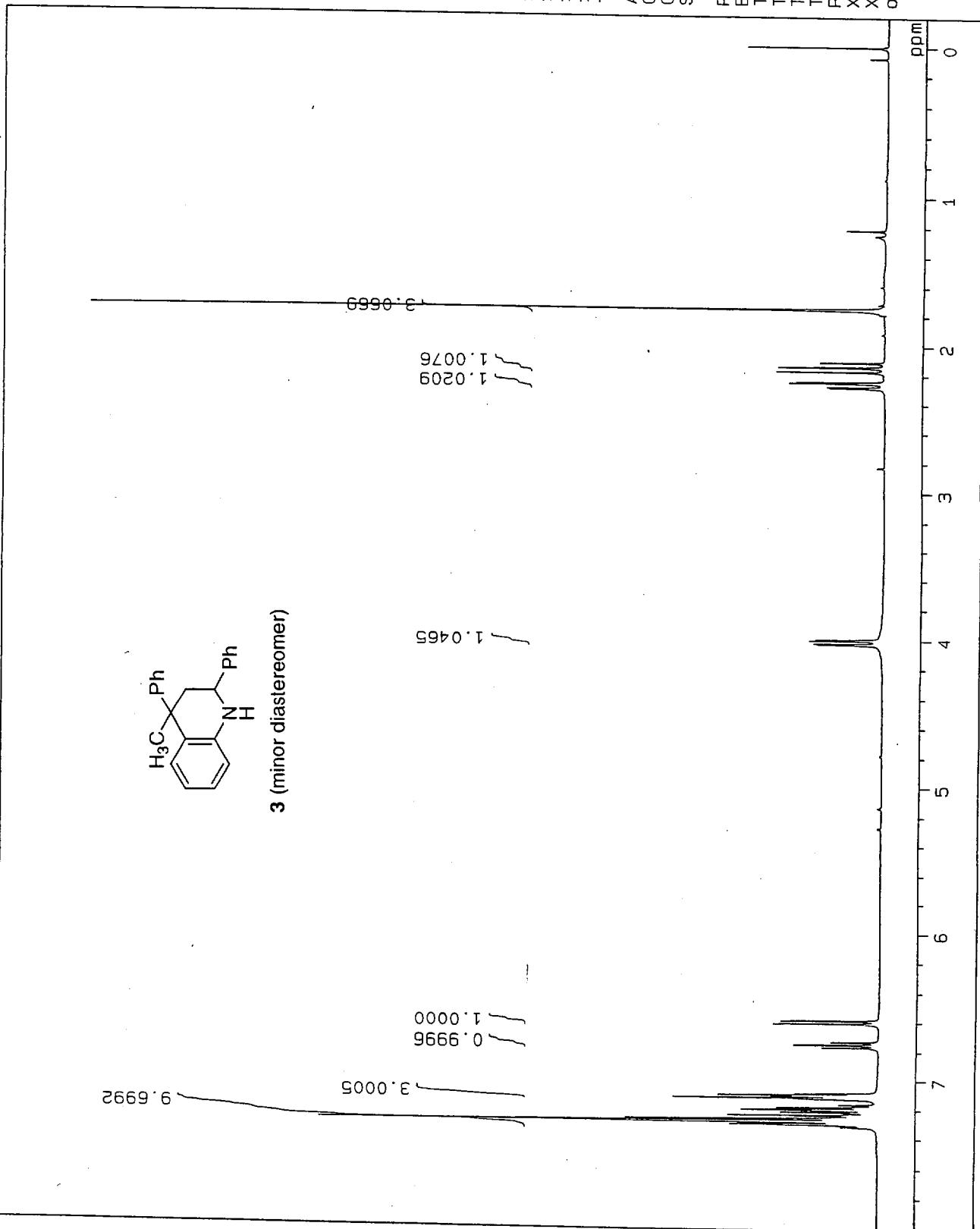
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 SCANS : 16
 DUMMY : 2
 ACQTM : 4.0993 sec
 PD : 3.0000 sec
 RGAIN : 15
 PW1 : 4.90 usec

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 OBFRQ : 399.65 MHz
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 IRFRQ : 399.65 MHz
 IRSET : 134300.00 Hz
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 IRBP1 : 25
 IRBP2 : 6
 IRNNS : 0
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 CSPED : 10 Hz
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 RESOL : 0.24 Hz
 BF : 0.01 Hz
 T1 : 0.00 ppm
 T2 : 0.00 ppm
 T3 : 90.00 ppm
 T4 : 100.00 ppm
 REFVL : 0.00 ppm
 XE : 3277.90 Hz
 XS : 423.86 Hz
 operator



3 (minor diastereomer)



ג'ז

33-AUG-1999 18:36:47.60

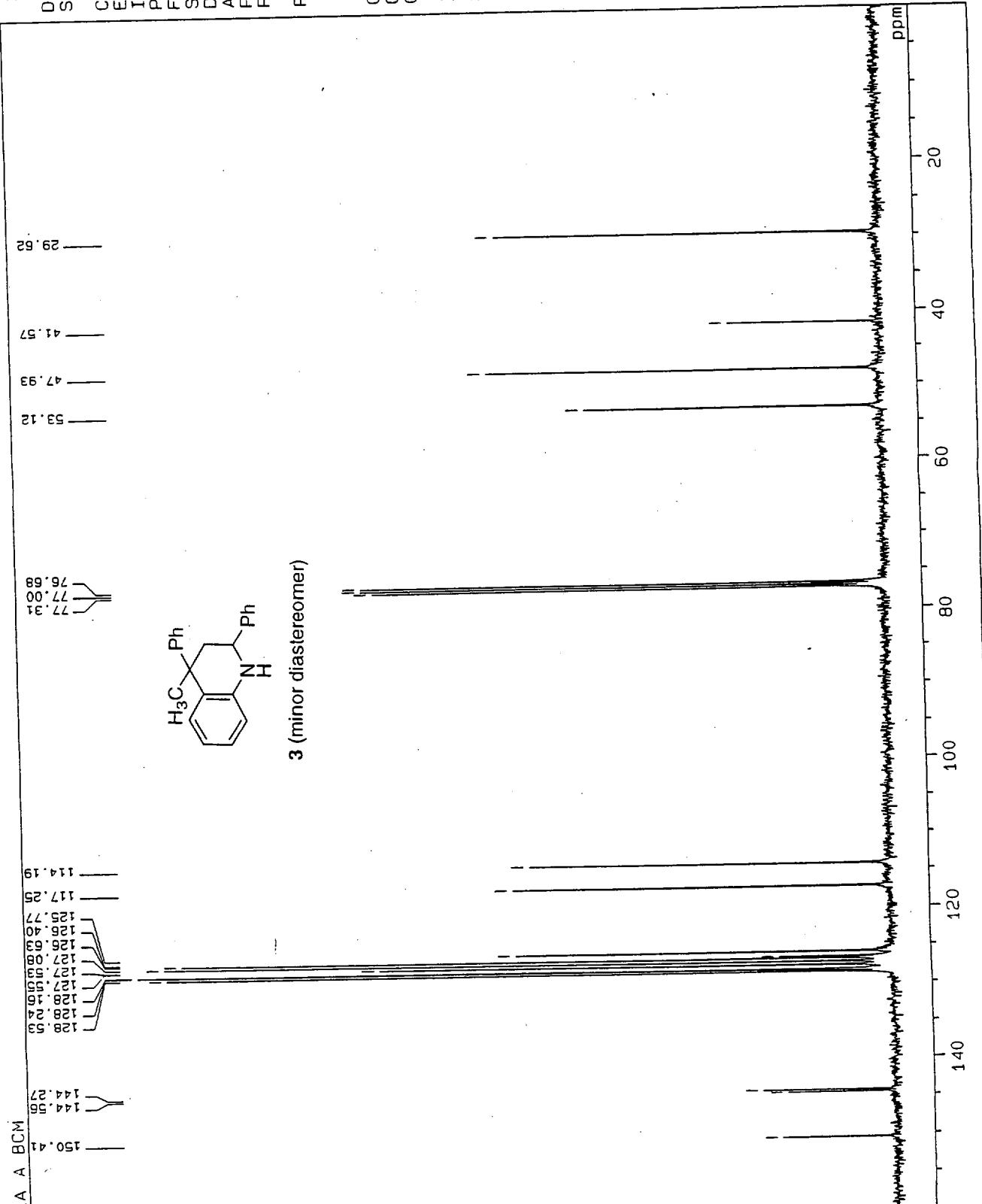
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PD : 0.3000 sec
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IRPPW	:	50.0 usec
IRBP1	:	25
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IRBNS	:	0

ADBIT	16	
CTEMP	28.6	°C
CSPED	1.0	Hz
SLVNT	CDCL3	
PRESOL	0.83	Hz
BF	3.00	Hz
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T2	0.00	%
T3	90.00	%
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XS	2065.52	Hz
operator		



JEOL

11-SEP-1999 11:17:46.99

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TRMOD : NON

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OBFRQ : 399.65 MHz

OBSET : 134500.00 Hz

PW1 : 4.90 usec

POINT : 32768

SAMPO : 16

TIMES : 16

SCANS : 16

DUMMY : 2

FREQU : 7993.61 Hz

FILTR : 4000 Hz

ACQTM : 4.0993 sec

DEADT : 72.55 usec

DELAY : 50.00 usec

PD : 3.0000 sec

ADB1T : 16

RGAIN : 15

IRNUC : 1H

IRFRQ : 399.65 MHz

IRSET : 134300.00 Hz

IRPWR : 50.0 usec

IRATN : 511

IRNS : 0

IRBP1 : 25

IRBP2 : 6

BF : 0.01 Hz

T1 : 0.00 %

T2 : 0.00 %

T3 : 90.00 %

T4 : 100.00 %

XE : 3477.69 Hz

XS : 298.83 Hz

REFVL : 0.00 ppm

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LGAIN : 24

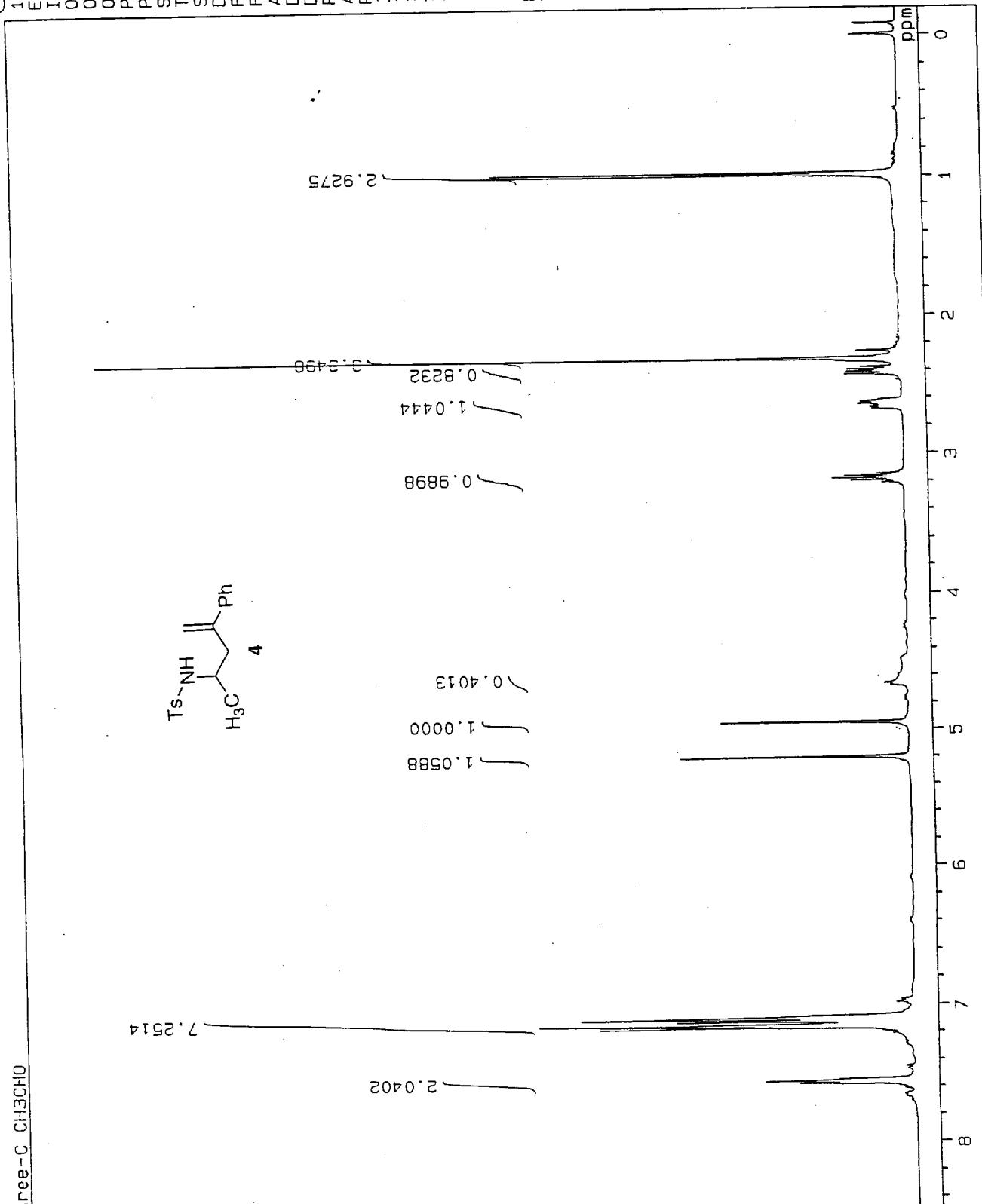
LKPHS : 248.91 deg

LKSIG : 1435

CTEMP : 26.9 C

CSPED : 14 Hz

FIELD : -10867



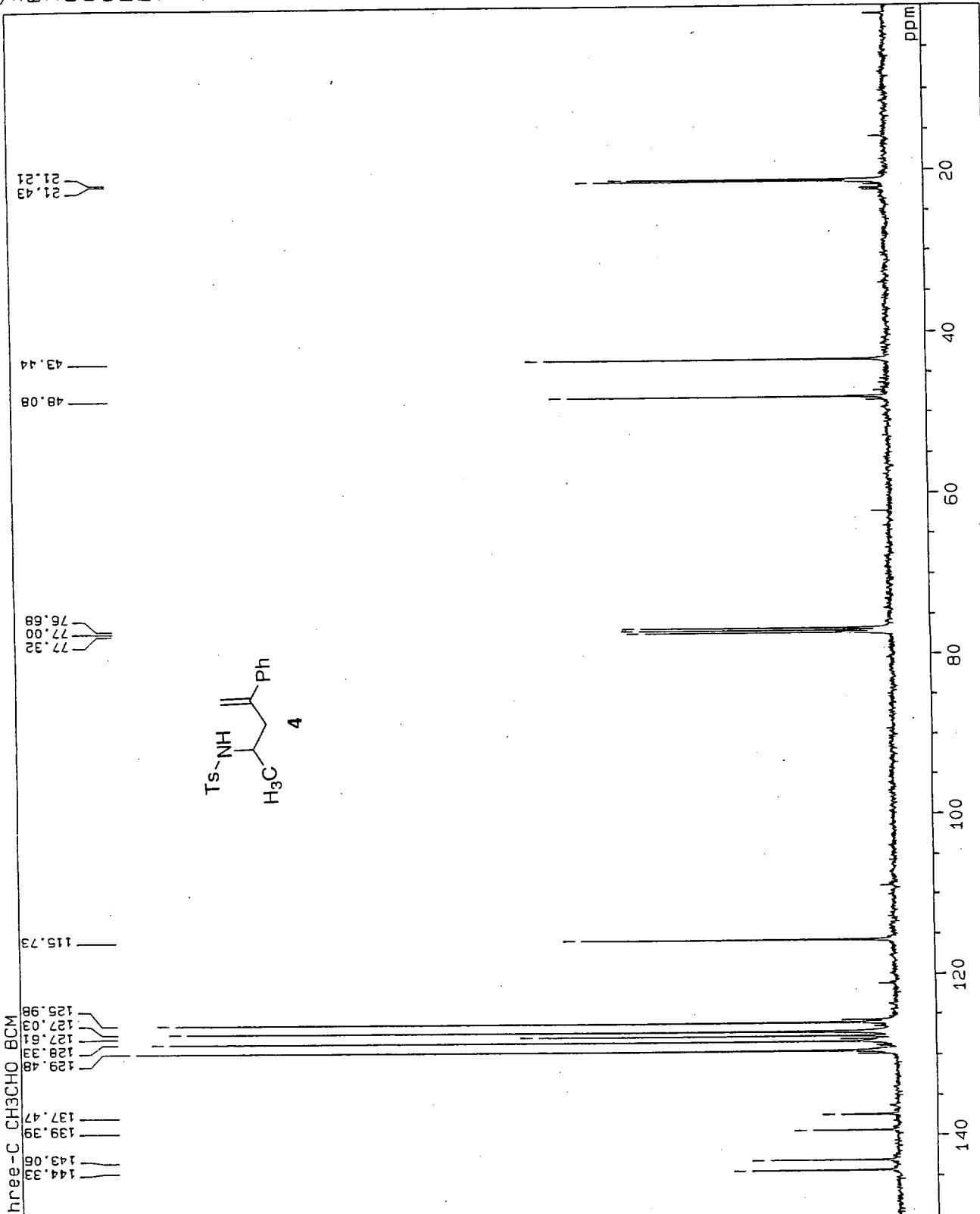
JEOL

11-SEP-1999 11: 58: 11.18

EXMOD : SINGL
 IRMOD : BCM
 OBNUC : 13C
 OBFRQ : 100.40 MHz
 OBSET : 135500.00 Hz
 PW1 : 4.55 usec
 POINT : 32768
 SAMPO : 1280
 SCANS : 1280
 DUMMY : 4

FREQU : 27100.27 Hz
 FILTR : 13550 Hz
 ACQTM : 1.2091 sec
 DEADT : 10.00 usec
 DELAY : 14.76 usec
 PD : 0.3000 sec
 ADBIT : 16
 RGAIN : 23

IRNUC : 1H
 IRFRQ : 339.65 MHz
 IRESET : 134300.00 Hz
 IRRPW : 50.0 usec
 IRATN : 511
 IRRNS : 0
 IRBP1 : 25
 IRBP2 : 6
 BF : 3.00 Hz
 T1 : 0.00 %
 T2 : 0.00 %
 T3 : 90.00 %
 T4 : 100.00 %
 XE : 15078.50 Hz
 XS : 2564.63 Hz
 REFVL : 77.00 ppm
 DF1LE : ALPHA
 MENUF : GS-BCM
 SHMFL : TH5
 LKSET : 61679.1 Hz
 LKLEV : 200
 LGAIN : 24
 LKPHS : 248.91 deg
 LKSIG : 1435
 CTEMP : 27.8 C
 CSPED : 13
 FIELD : -10867



JECOL

13-SEP-1999

09: 11: 23.39

EXMOD : SINGL

TAMOD : NON

OBNUC : 1H

OBFRQ : 399.65 MHz

OBSET : 134500.00 Hz

PW1 : 4.90 usec

POINT : 32768

SAMPO : 2

TIMES : 16

SCANS : 16

DUMMY : 2

FREQU : 7993.61 Hz

FILTR : 4000 Hz

ACQTM : 4.0993 sec

DEADT : 72.55 usec

DELAY : 50.00 usec

PD : 3.0000 sec

ADBIT : 16.

RGAIN : 16

IRNUC : 1H

IRFRQ : 399.65 MHz

IRSET : 134300.00 Hz

IRPWR : 50.0 usec

IRATN : 511

IRANS : 0

IRBP1 : 25

IRBP2 : 6

BF : 0.01 Hz

T1 : 0.00 %

T2 : 0.00 %

T3 : 0.00 %

T4 : 100.00 %

XE : 3477.69 Hz

XS : 331.52 Hz

REFVL : 0.00 ppm

DFILE : ALPHA

MENUF : GS-1H-NON

SHMFL : TH5

LKSET : 61679.1 Hz

LKLEV : 200

LGAIN : 23

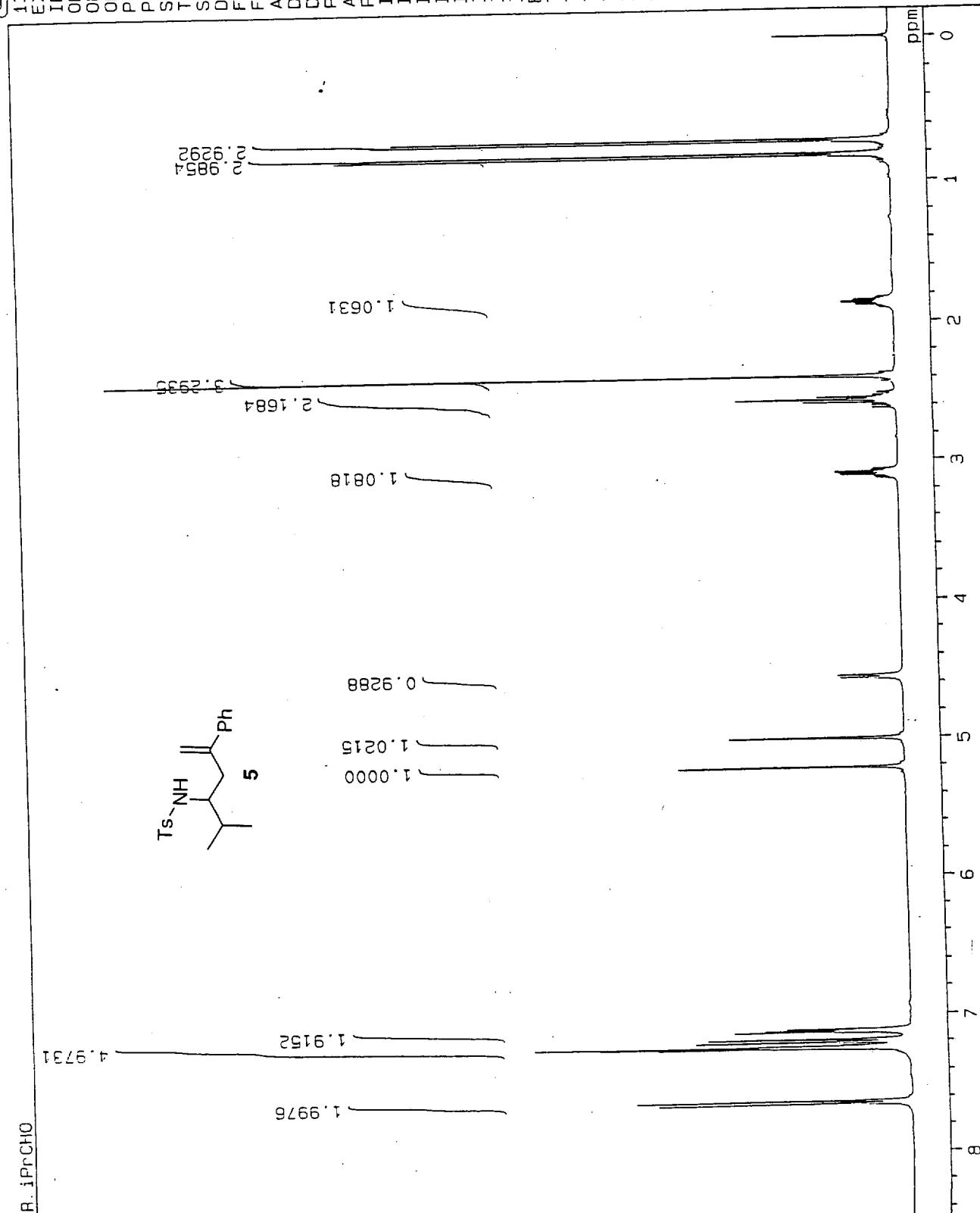
LKPHS : 248.91 deg

LKSIG : 1310

CTEMP : 27.9 C

CSPED : 13

FIELD : -10867



JEOL 13-SEP-1999 09: 49: 36.73
EXWD001.S1N61

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113-SEP-1999 09: 49: 36.73
EXMOD : SINGL
TRMOD : BCM
DBNUC : 13C
DBFRQ 13100.40 MHZ
DBSET 135500.00 Hz
PWI 4.55 usec
POINT 32768
SAMPO 32768
TIMES 1280
SCANS 1280
DUMMY 4
FREQU 27100.27 Hz
FILTR 13550.00 Hz
ACQTM 1.2091 sec
DEADT 10.00 usec
DELAY 14.76 usec
PDW 0.3000 sec
ABIT 16
AGAIN 23
IRNUC 1H
IRSET 339.65 MHz
IRAPW 134300.00 Hz
IRATN 50.0 usec
IRANS 51.1
IRBP1 0
IRBP2 25
IRBF 6
T1 3.00 Hz
T2 0.00 %
T3 0.00 %
T4 90.00 %
XE 100.00 %
XS 15078.50 Hz
XE 2567.94 Hz
XS 77.00 ppm
REFVL DFILE
MENUF GS-BCM
SHMFL TH5
LKSET 61679.1 Hz
LKLEV 200
LGAIN 23
LKPHS 248.91 deg
LKSIG 1310
CTEMP 28.6 C
CSPEED 13 Hz
EFIELD -10867

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