

Supplementary Data

P=C-N-Heterocycles: Synthesis of Biaryl-Type 1,3-Benzazaphospholes with ortho-Substituted Phenyl or 2-Heteroaryl Groups

Bhaskar Reddy Aluri, Basit Niaz, Markus K. Kindermann, Peter G. Jones, and Joachim Heinicke*

DOI: 10.1039/b000000x

¹³C NMR Spectra of new 2-anilidophosphonates 2b-k and 2-(hetero)aryl-1,3-benzazaphospholes 3a-d,f-h.

¹³C NMR Spectrum of compound **2b**.

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandentkoppelt)
B.Reddy: BR79-3P
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = CDCl₃
Messtemperatur: 298K (25 oC)

Current Data Parameters
NAME BH_BR79-3P
EXPNO 13
PROCNO 1
USER hein

F2 - Acquisition Parameters
Date_ 20051011
Time 12.02

INSTRUM spect
PROBHD 5 mm Multima
PULPROG zgpg
TD 65536
NS 4096
DS 0

SWH 21739.131 Hz
FIDRES 0.331713 Hz
AQ 1.5073780 sec
RG 32768

DW 23.000 usec
DE 32.86 usec
TE 298.0 K

D12 0.0002000 sec
DL6 30.00 dB

D1 2.0000000 sec
CPDPRG waltz16

P31 110.00 usec
D11 0.03000000 sec

DL5 22.00 dB
P1 2.50 usec

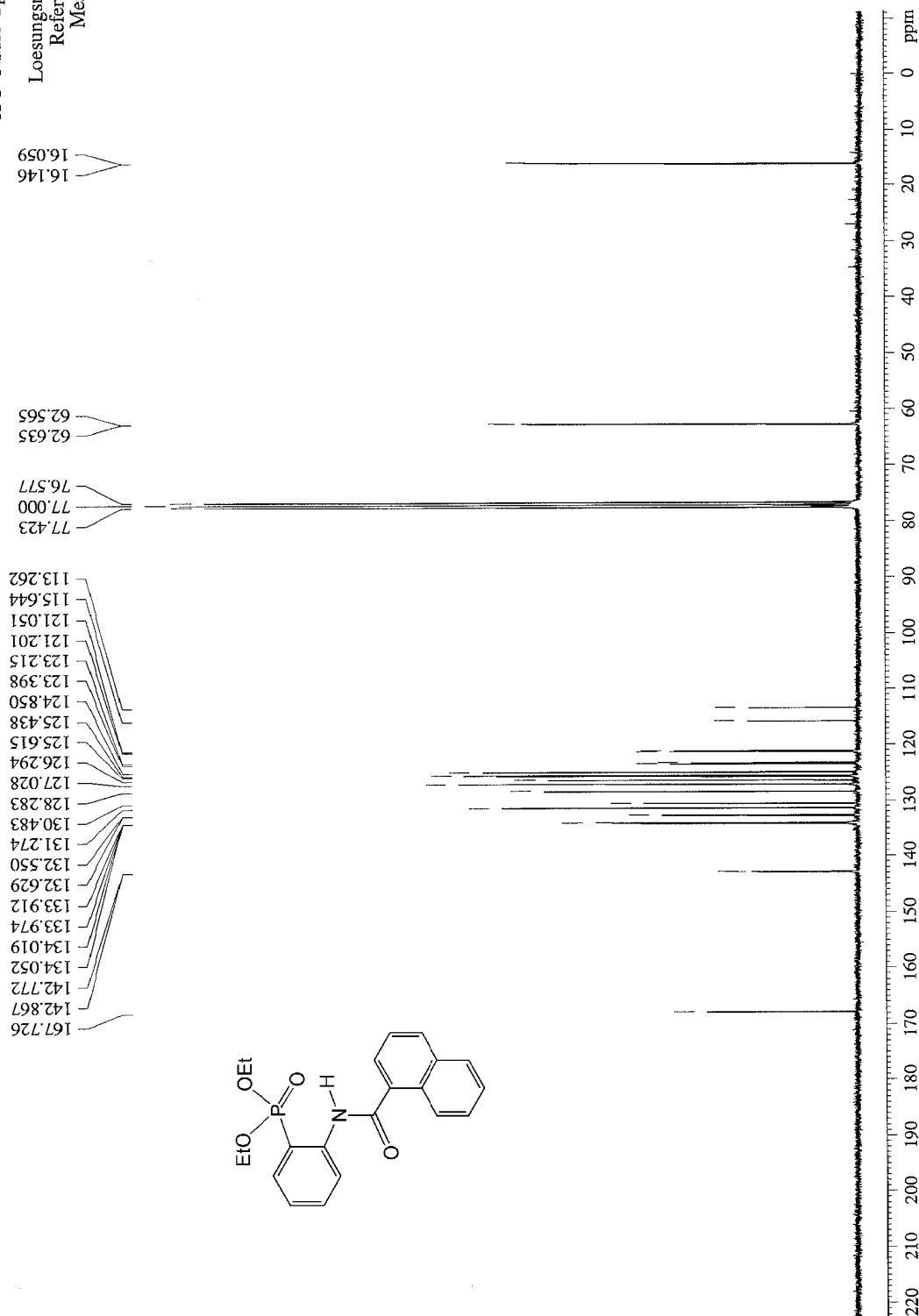
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 32768

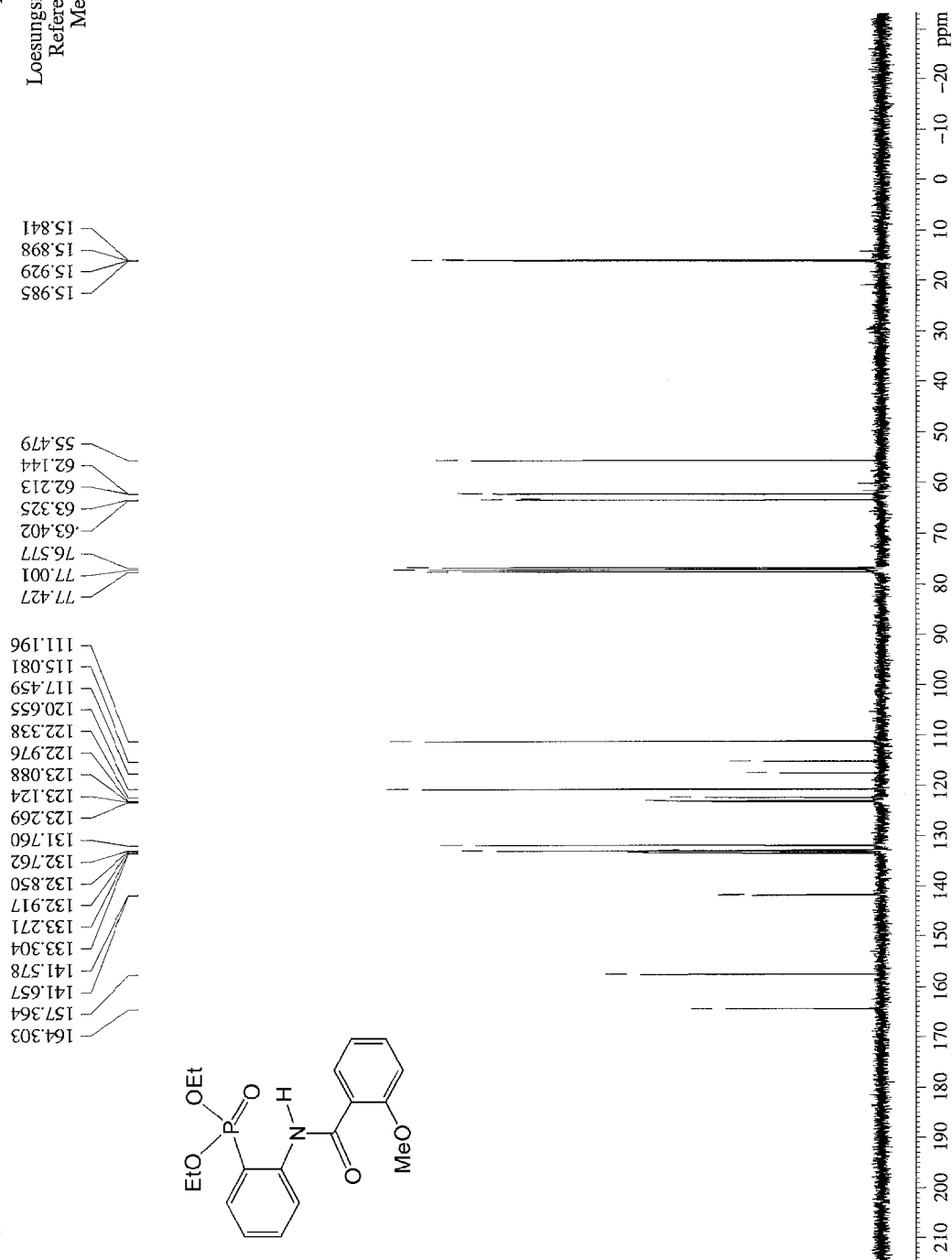
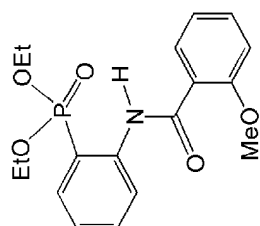
SF 75.4677345 MHz
WDW EM

SSB 0
LB 0.70 Hz

GB 0
PC 0.80



¹³C NMR Spectrum of compound 2c.



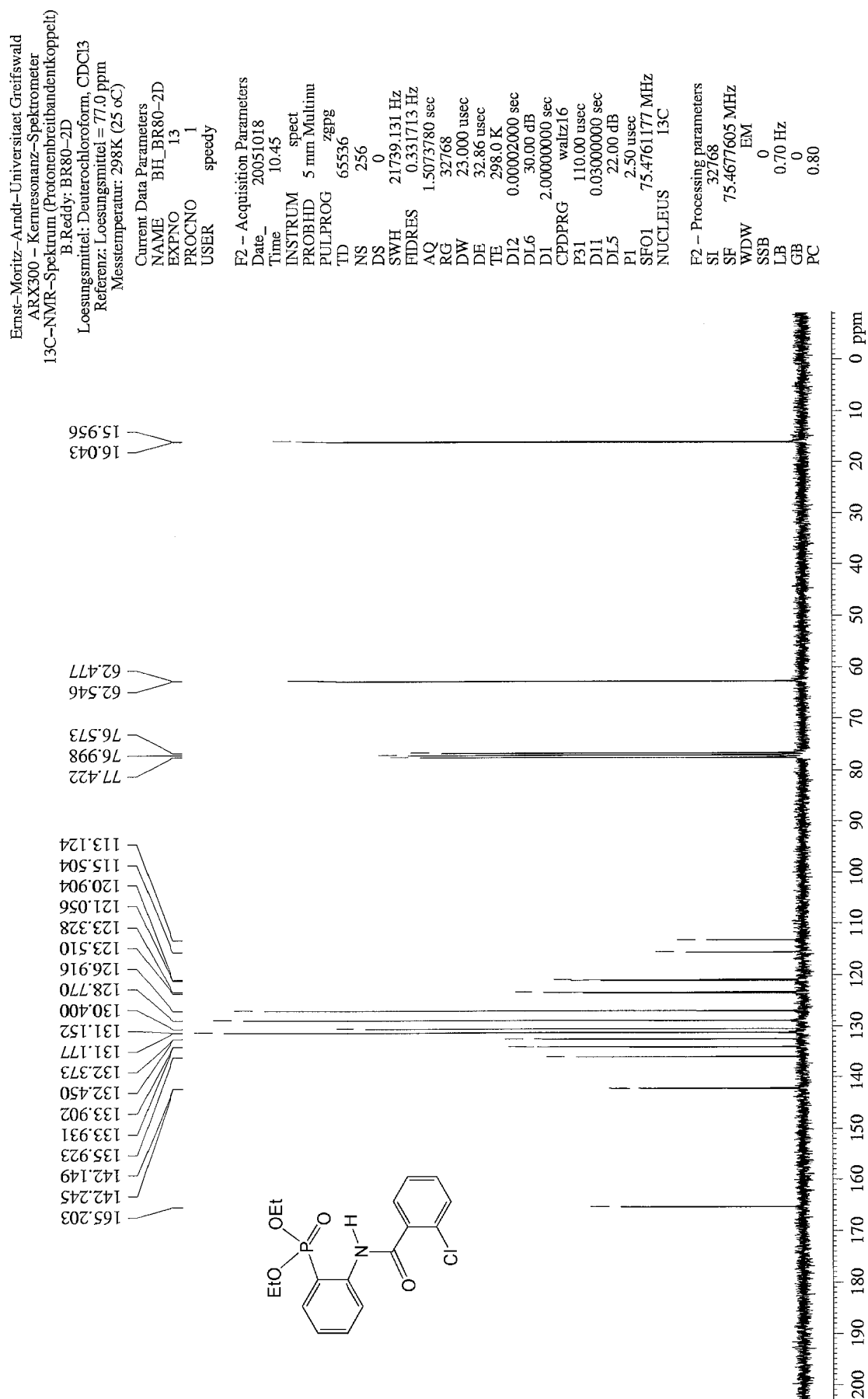
Ernst-Moritz-Arndt-Universitaet Greifswald
ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)
B.Reddy: BR163P
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Loesungsmittel = 77.0 ppm
Messtemperatur: 298K (25 oC)

Current Data Parameters
NAME BH_BR163P
EXPNO 13
PROCNO 1
USER hein

F2 – Acquisition Parameters
Date_ 20060207
Time 8.00
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 65536
NS 256
DS 0
SWH 21739.131 Hz
FIDRES 0.331713 Hz
AQ 1.5073780 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

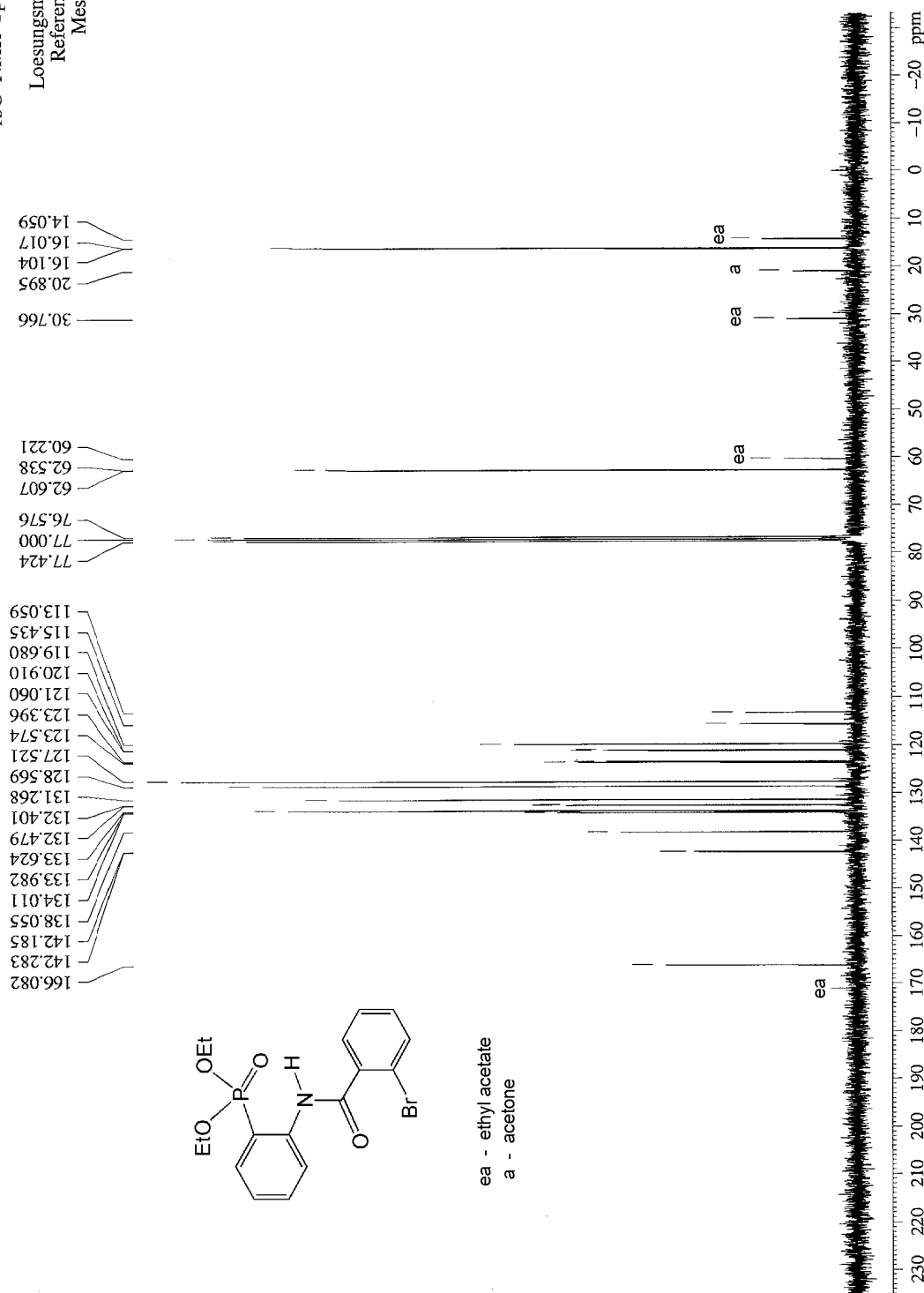
F2 – Processing parameters
SI 32768
SF 75.4677625 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00

¹³C NMR Spectrum of compound 2d.



¹³C NMR Spectrum of compound **2e**.

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
B.Reddy; BR1521D
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = 77.0 ppm
Mess temperatur: 298K (25 oC)



Current Data Parameters
NAME BH_BR1521D
EXPNO 13
PROCNO 1
USER hein

F2 - Acquisition Parameters
Date_ 20060110
Time 8.32
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 65536
NS 256
DS 0
SWH 21739.131 Hz
FIDRES 0.331713 Hz
AQ 1.5073780 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.0002000 sec
DL6 30.00 dB
D1 2.0000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 - Processing parameters
SI 32768
SF 75.4677585 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00

¹³C NMR Spectrum of compound 2f.

Ernst-Moritz-Arndt-Universitaet Greifswald
ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandentkoppelt)
Bhaskar Reddy A:BL34
Loesungsmittel: Deuteriochloroform, CDCl₃
Referenz: Loesungsmittel = 77.0 ppm
Mess temperatur: 298K (25 oC)

Current Data Parameters
NAME BH_BL34
EXPNO 13
PROCNO 1
USER hein

F2 – Acquisition Parameters
Date_ 20060426
Time 8.34

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg

TD 32768
NS 256
DS 0

SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec

RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K

D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec

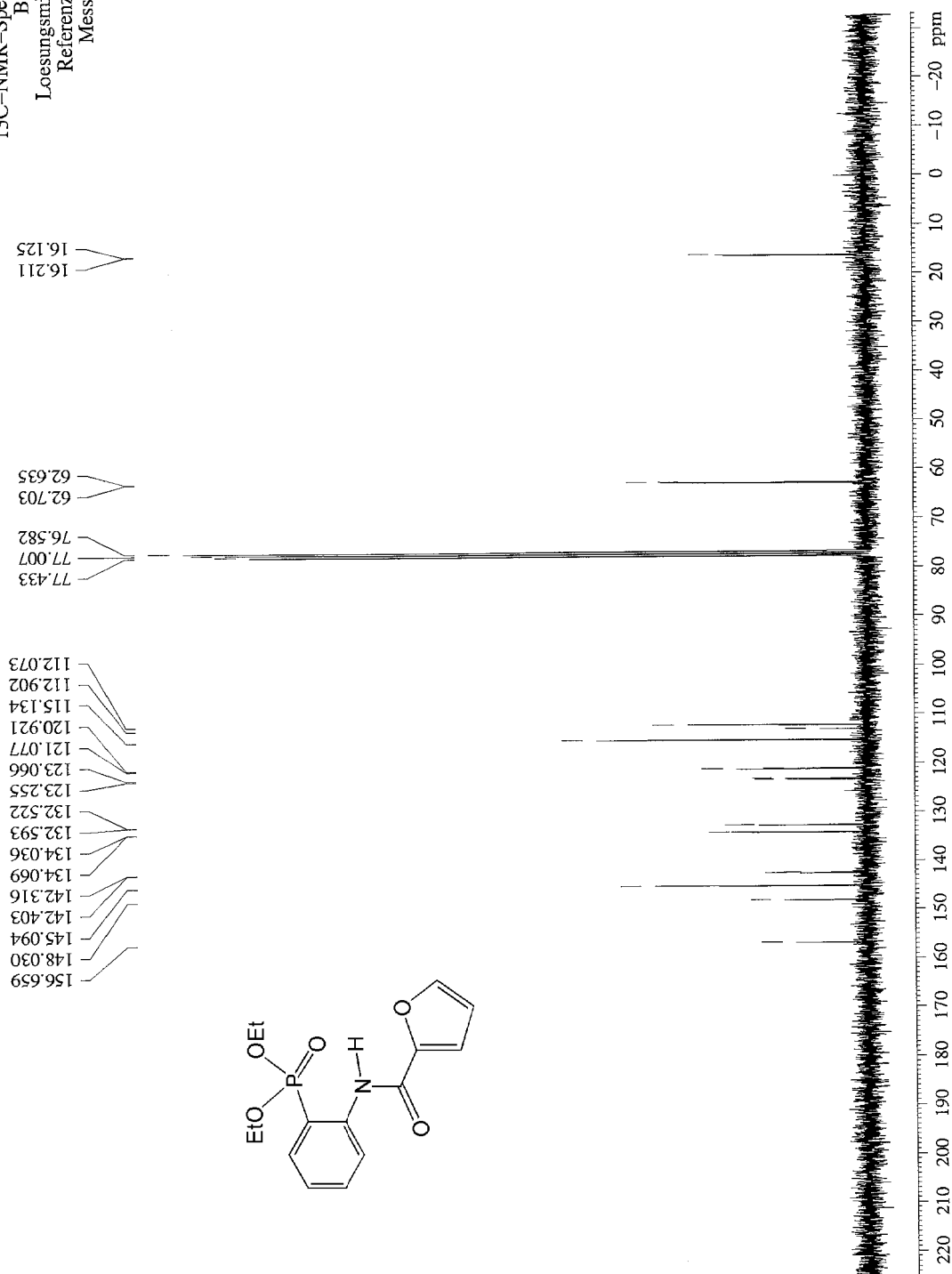
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec

DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz

NUCLEUS ¹³C

F2 – Processing parameters
SI 16384
SF 75.4677512 MHz

WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 0.80



¹³C NMR Spectrum of compound 2g.

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandentkoppelt)
Bhaskar Reddy A: BL33
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel 77.0 ppm
Messstemperatur: 298 K (25 °C)

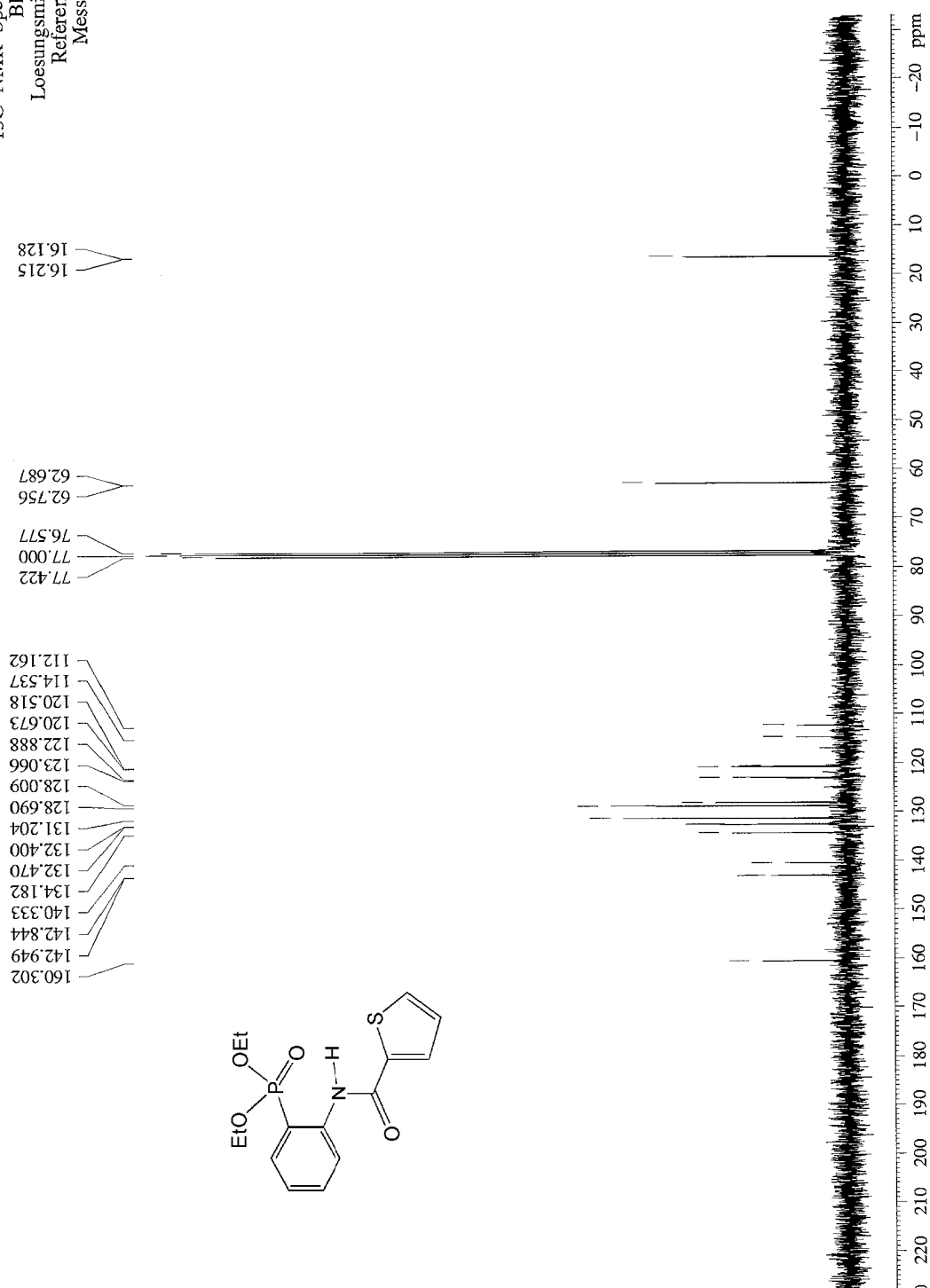
Current Data Parameters
NAME BH_BL33
EXPNO 13
PROCNO 1
USER hein

F2 – Acquisition Parameters
Date_ 20060426
Time 8.12
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 32768
NS 136
DS 0

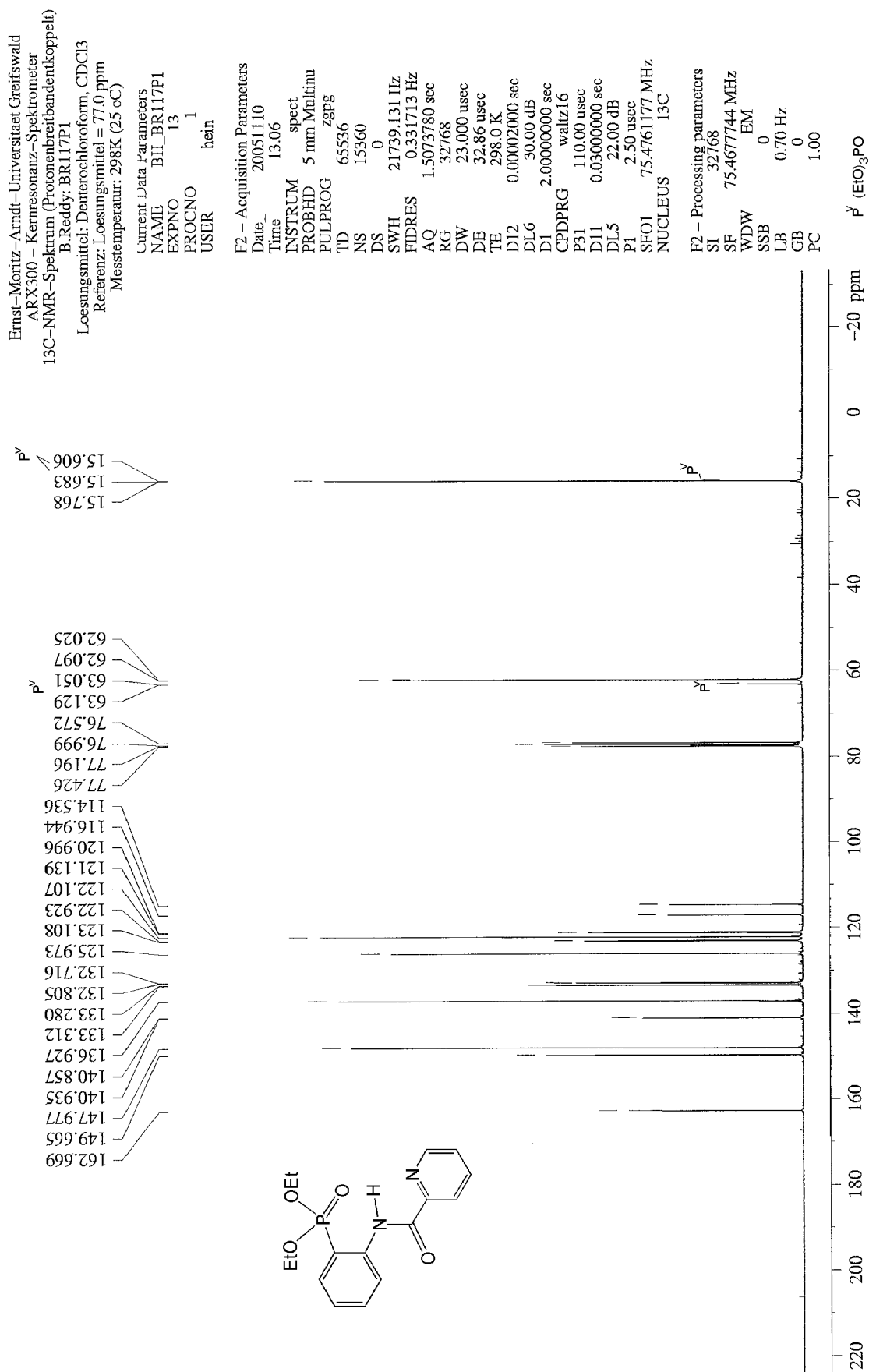
SWH 21739.131 Hz
FIDRES 0.663426 Hz
AQ 0.7537140 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.0002000 sec
DL6 30.00 dB
D1 2.0000000 sec

CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

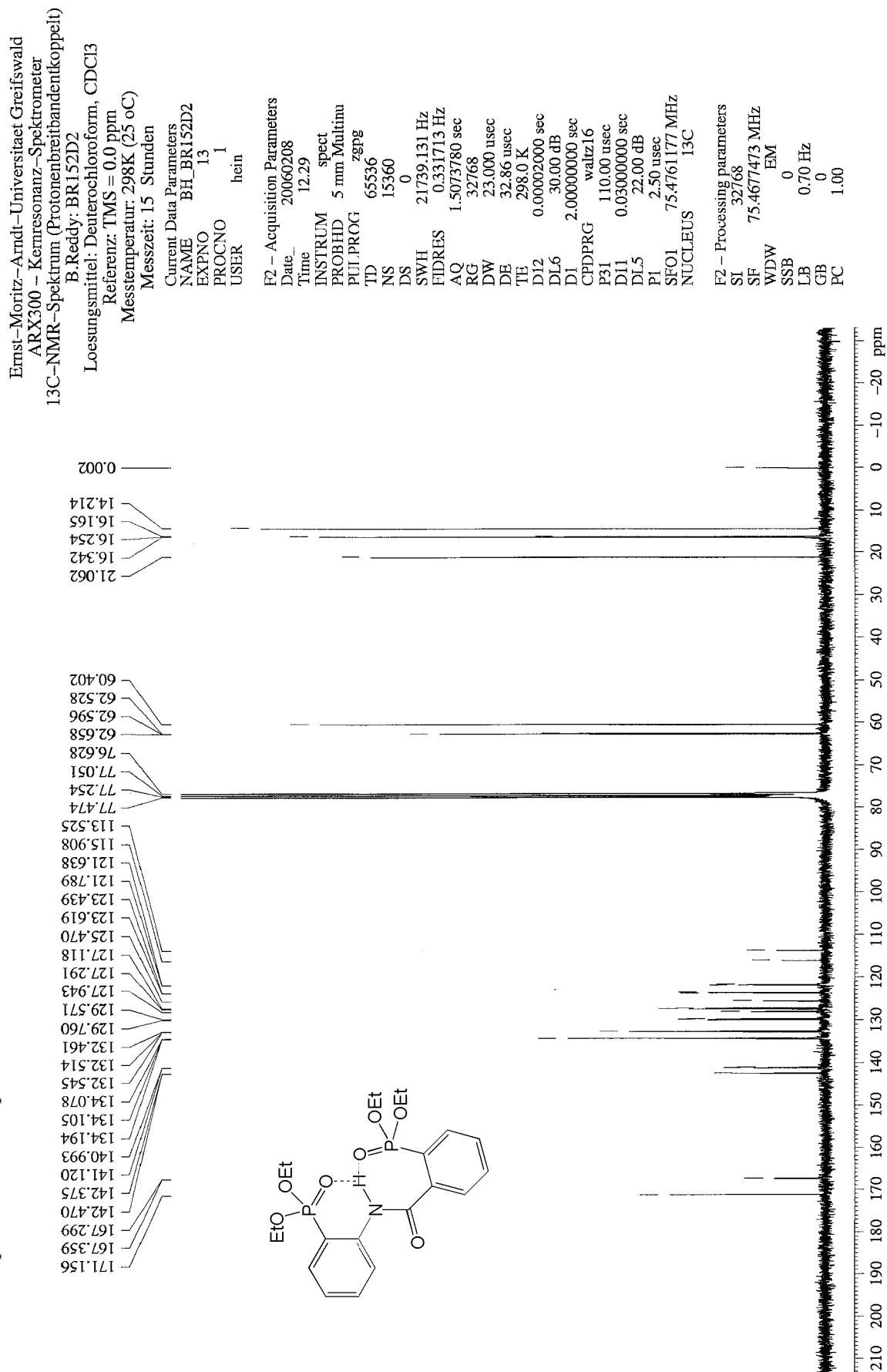
F2 – Processing parameters
SI 16384
SF 75.4677525 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 0.80



¹³C NMR Spectrum of compound 2h.



¹³C NMR Spectrum of compound 2i.



¹³C NMR Spectrum of compound 2j.

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandkoppelt)
B. Reddy: BH_BR105-3P

Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = 77.0 ppm
Mess temperatur: 298K (25 oC)
Messzeit: 16 Stunden

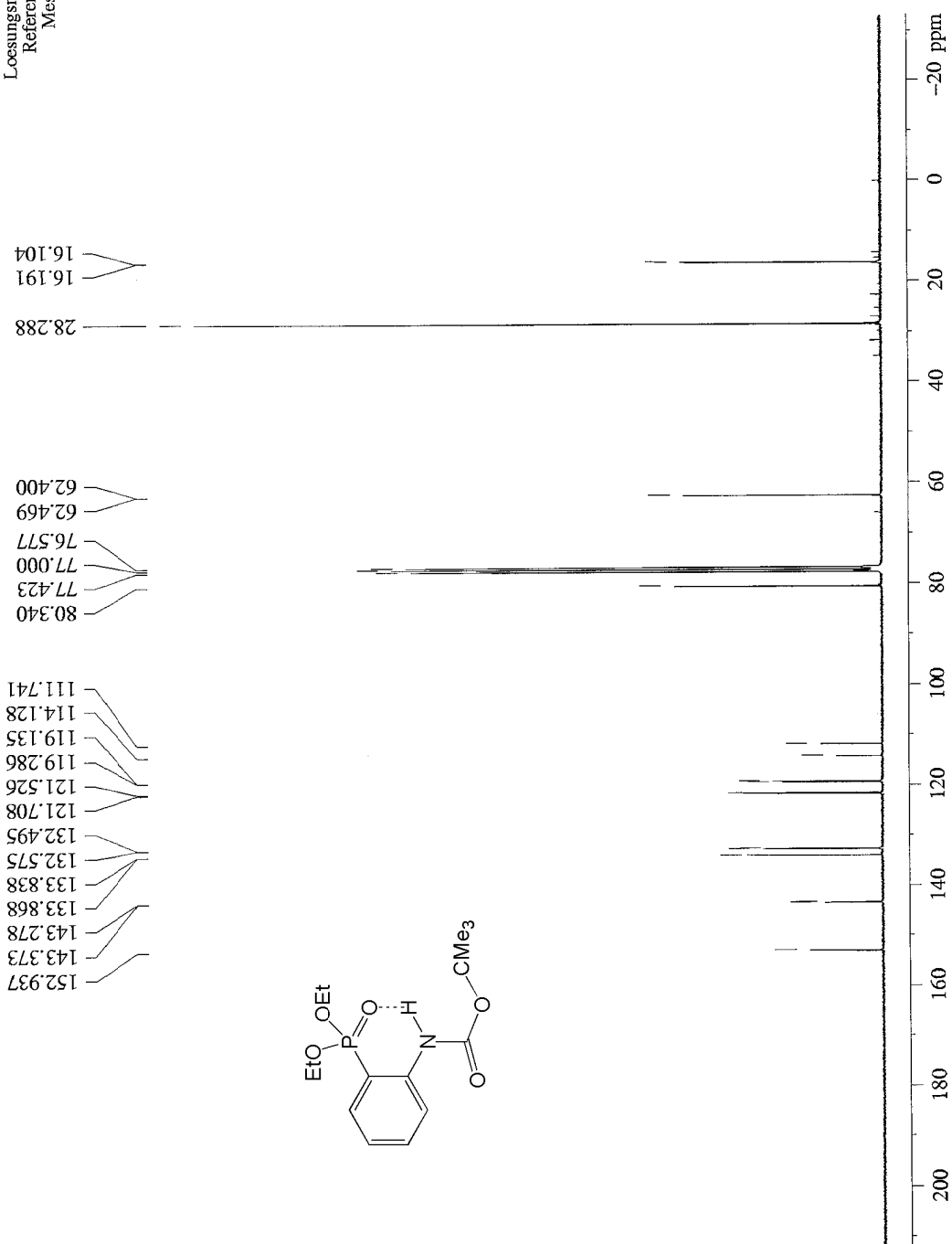
Current Data Parameters
NAME: BIL_BR105-3P
EXPNO: 13
PROCNO: 1
USER: hein

F2 - Acquisition Parameters

Date_: 20051017
Time: 12.23
INSTRUM: spect
PROBHD: 5 mm Multinu
PULPROG: zgpg
TD: 65536
NS: 16384
DS: 0
SWH: 21739.131 Hz
FIDRES: 0.331713 Hz
AQ: 1.5073780 sec
RG: 32768
DW: 23.000 usec
DE: 32.86 usec
TE: 298.0 K
D12: 0.00002000 sec
DL6: 30.00 dB
D1: 2.00000000 sec
CPDPRG: waltz16
P31: 110.00 usec
D11: 0.03000000 sec
DL5: 22.00 dB
P1: 2.50 usec
SFO1: 75.4761177 MHz
NUCLEUS: ¹³C

F2 - Processing parameters

SI: 32768
SF: 75.4677505 MHz
WDW: EM
SSB: 0
LB: 0.70 Hz
GB: 0
PC: 0.80



¹³C NMR Spectrum of compound 2k.

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandentkoppelt)

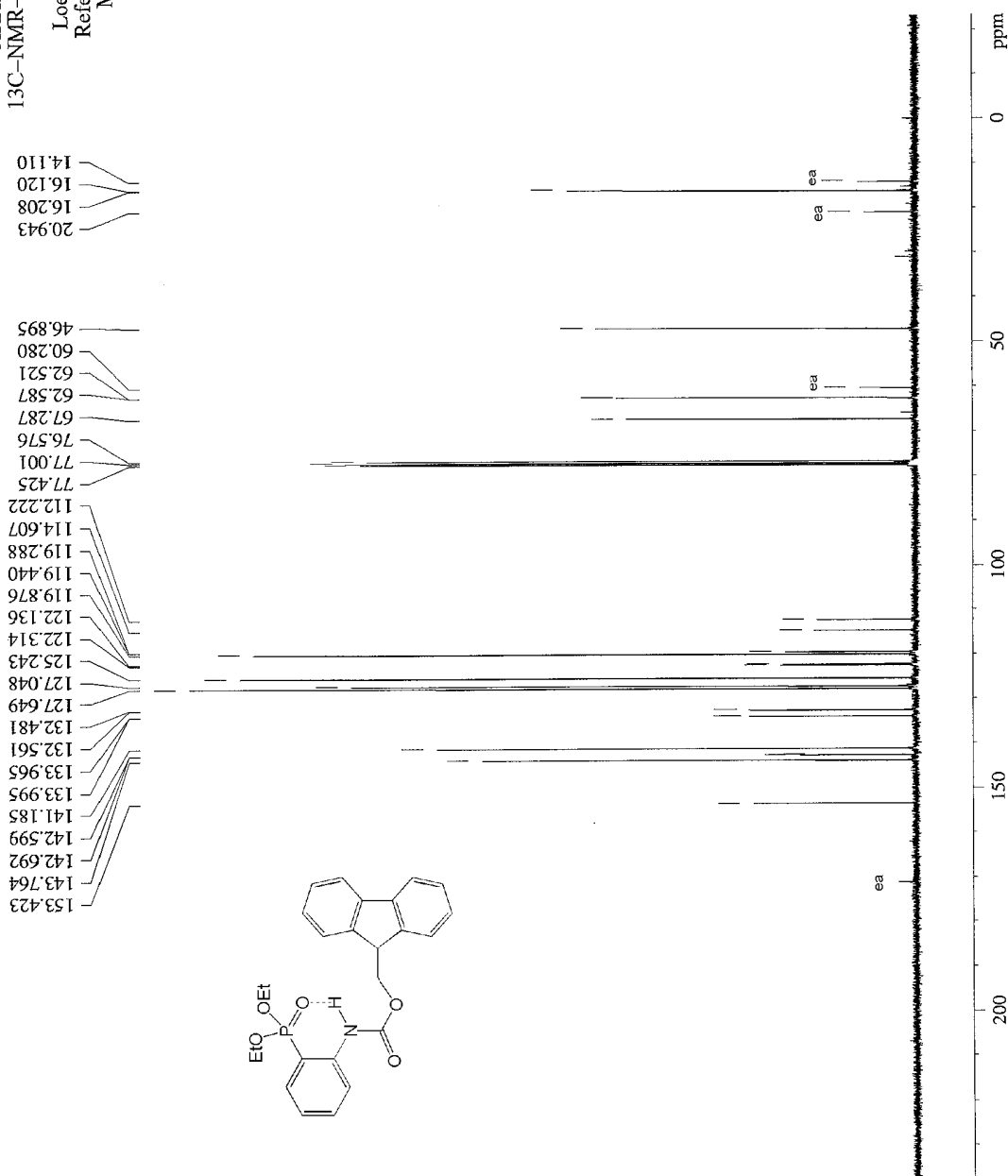
B.Reddy: BL1P
Lösungsmittel: Deuteriochloroform
Referenz: Loesungsmittel = 77.0 ppm
Mess temperatur: 298K (25 oC)

Current Data Parameters
NAME BH_BL1P
EXPNO 13
PROCNO 1
USER hein

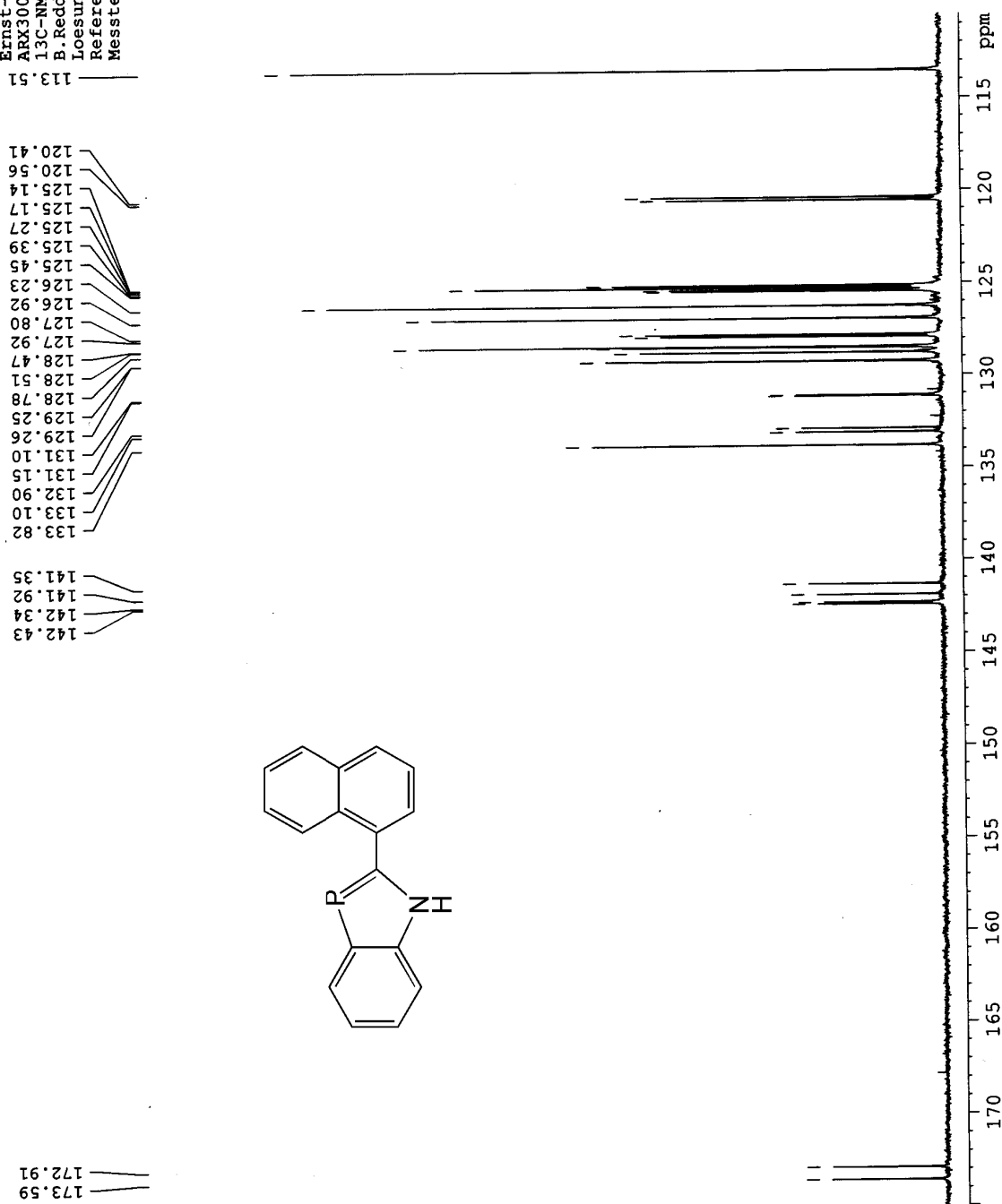
F2 - Acquisition Parameters

Date_ 20060223
Time 11.54
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 65536
NS 2048
DS 0
SWH 25000.000 Hz
FIDRES 0.381470 Hz
AQ 1.3107700 sec
RG 32768
DW 20.000 usec
DE 25.00 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4784953 MHz
NUCLEUS ¹³C

F2 - Processing parameters
SI 32768
SF 75.4677572 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 0.60



¹³C NMR Spectrum of compound **3b**.



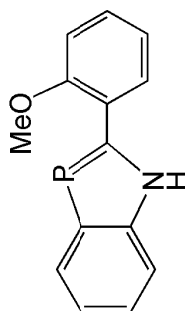
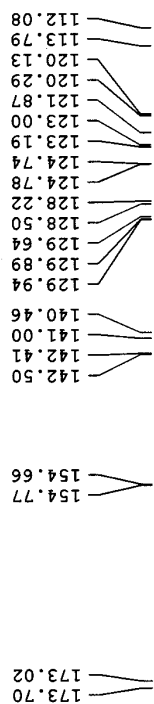
Ernst-Moritz-Arndt-Universitaet Greifswald
 ARX300 - Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitbandenkoppelt)
 B.Reddy: BR82D
 Loesungsmittel: Deuteriochloroform, CDCl3
 Referenz: LM = 77.0 ppm
 Messtemperatur: 298K (25 oC)

Current Data Parameters BH_BR82D
 NAME
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20051205
 Time 11:25
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 4096
 DS 4
 FIDRES 21739.131 Hz
 AQ 0.331713 Hz
 RG 1.5073780 sec
 DW 22800
 DE 23.000 usec
 TE 32.86 usec
 D12 298.0 K
 D16 0.000200 sec
 D1 30.00 dB
 D15 2.0000000 sec
 CDPRG waltz16
 P31 110.00 usec
 D11 0.03000000 sec
 DL5 22.00 dB
 F1 2.50 usec
 SFO1 75.476117 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 65536
 SF 75.4677572 MHz
 WDW EM
 SSB 0
 LB 0
 GB 0
 PC 1.00

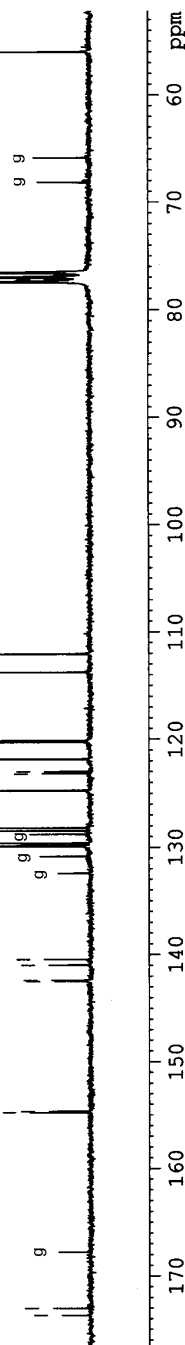
¹³C NMR Spectrum of compound **3c**.



Current Data Parameters
Name: BH165P1
EXPNO: 13
PROCNO: 1

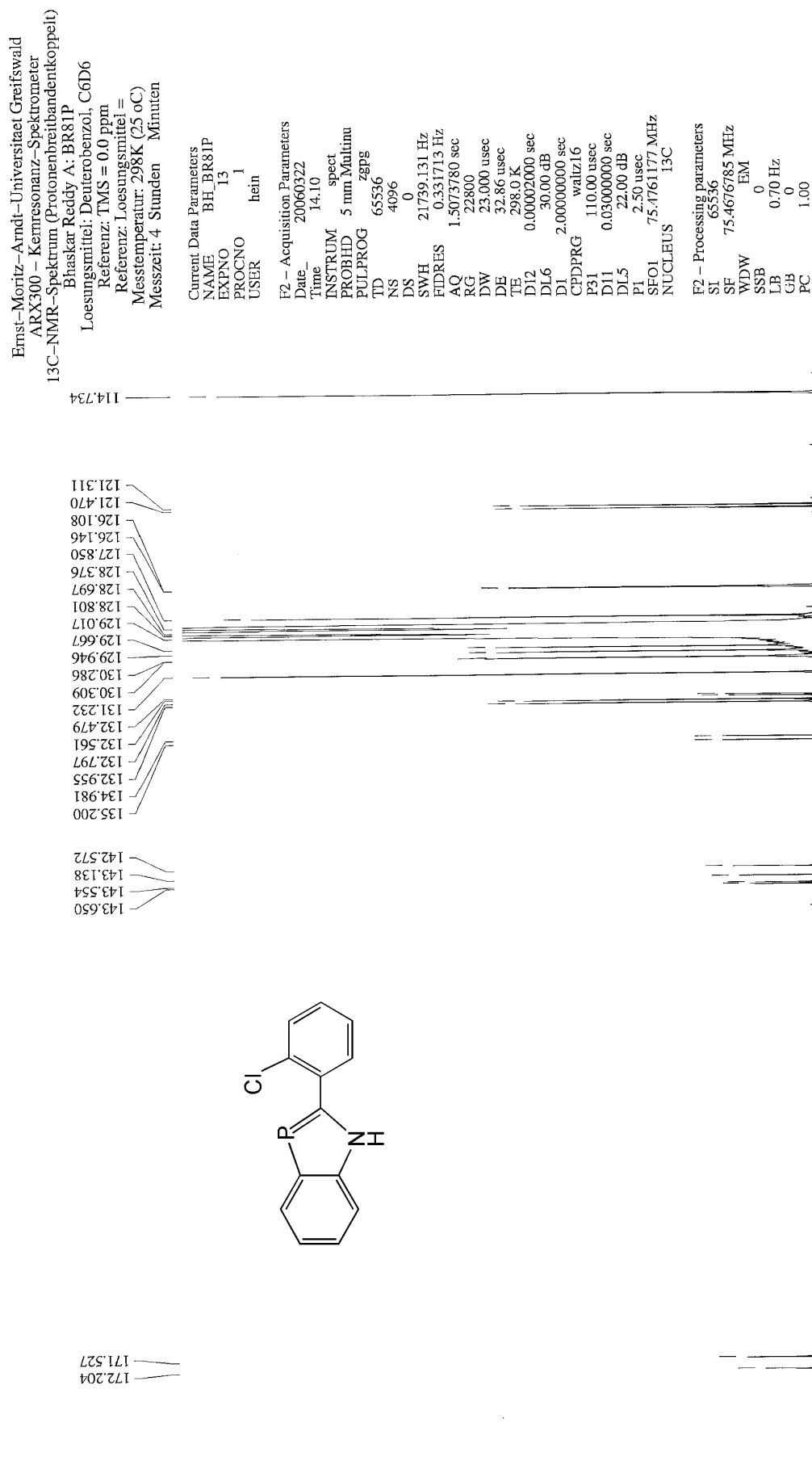
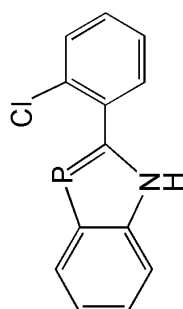
F2 - Acquisition Parameters
Date_ Time: 20060405 17:52
INSTRUM: spect
PROBHD: 5 mm Multinu
PULPROG: zgpg
TD: 65536
SOLVENT: CDCl3
NS: 1228
DS: 0
SWH: 25000.000 Hz
FIDRES: 0.381470 Hz
AQ: 1.3107700 sec
RG: 32768
DM: 20.000 usec
DE: 25.00 usec
TE: 298.0 K
D12: 0.0002000 sec
DL6: 30.00 dB
D1: 0.0002000 sec
CHRG: 11.00 usec
F31: 0.03000000 sec
D11: 22.00 dB
D15: 2.50 usec
F1: 75.4784953 MHz
SF01: 13C
NUCLEUS: 13C

F2 - Processing parameters
SI: 32768
SF: 75.4677479 MHz
WDW: EM
SSB: 0
LB: 0.70 Hz
GB: 0
PC: 0.60

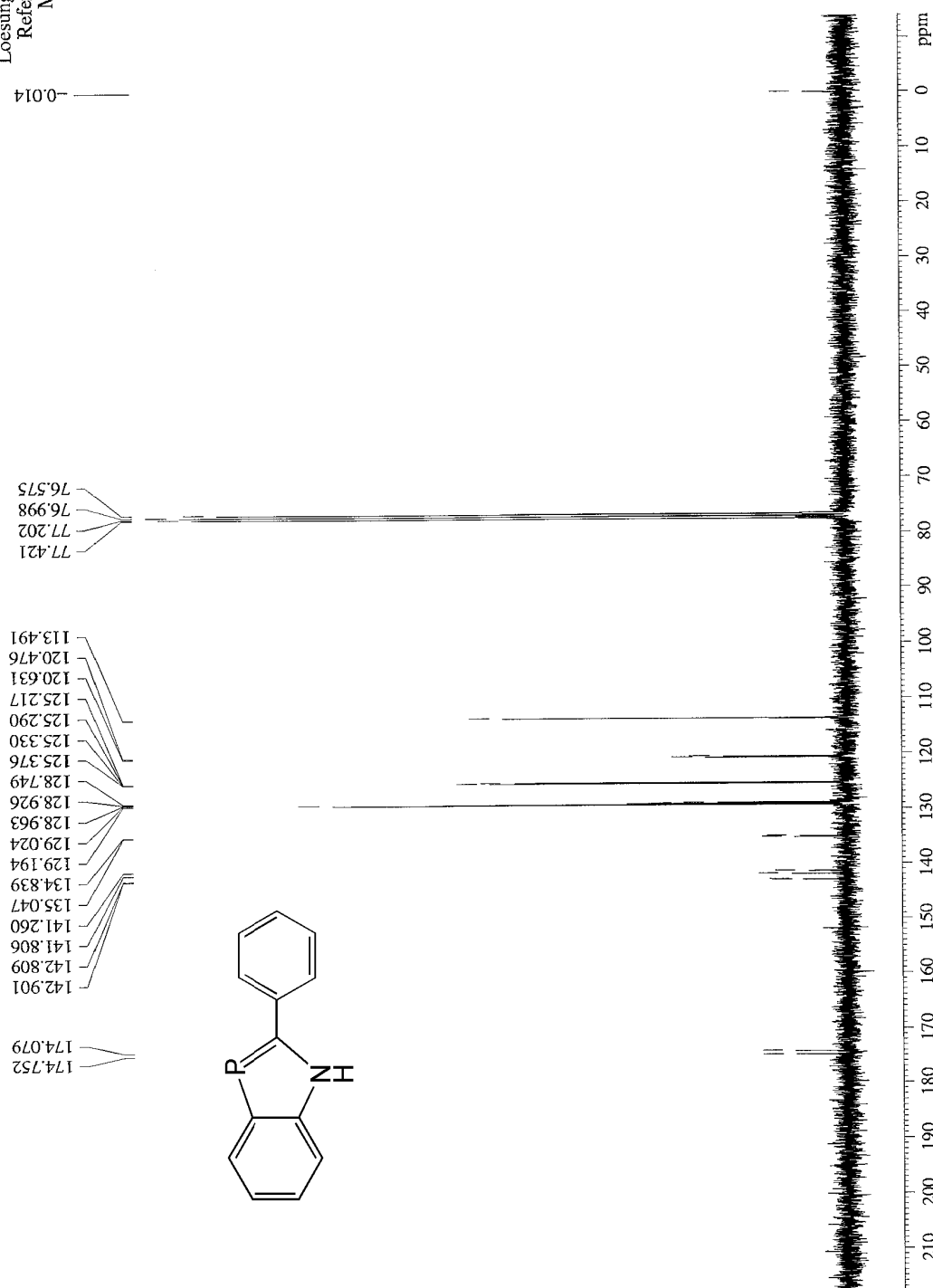


g signals of phthalate ester grease

¹³C NMR Spectrum of compound 3d.



¹³C NMR Spectrum of compound **3a** (by reductive cyclization of **2e**).



Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandenkoppelt)
B.Reddy: BR155c
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = 77.0 ppm
Mess temperatur: 298K (25 oC)

Current Data Parameters
NAME BH_BR155c
EXPNO 13
PROCNO 1
USER hein

F2 – Acquisition Parameters
Date_ 20060202
Time 8.51
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 65536
SOLVENT CDCl₃
NS 584
DS 0
SWH 21739.131 Hz
FIDRES 0.331713 Hz
AQ 1.5073780 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS ¹³C

F2 – Processing parameters
SI 32768
SF 75.4677512 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00

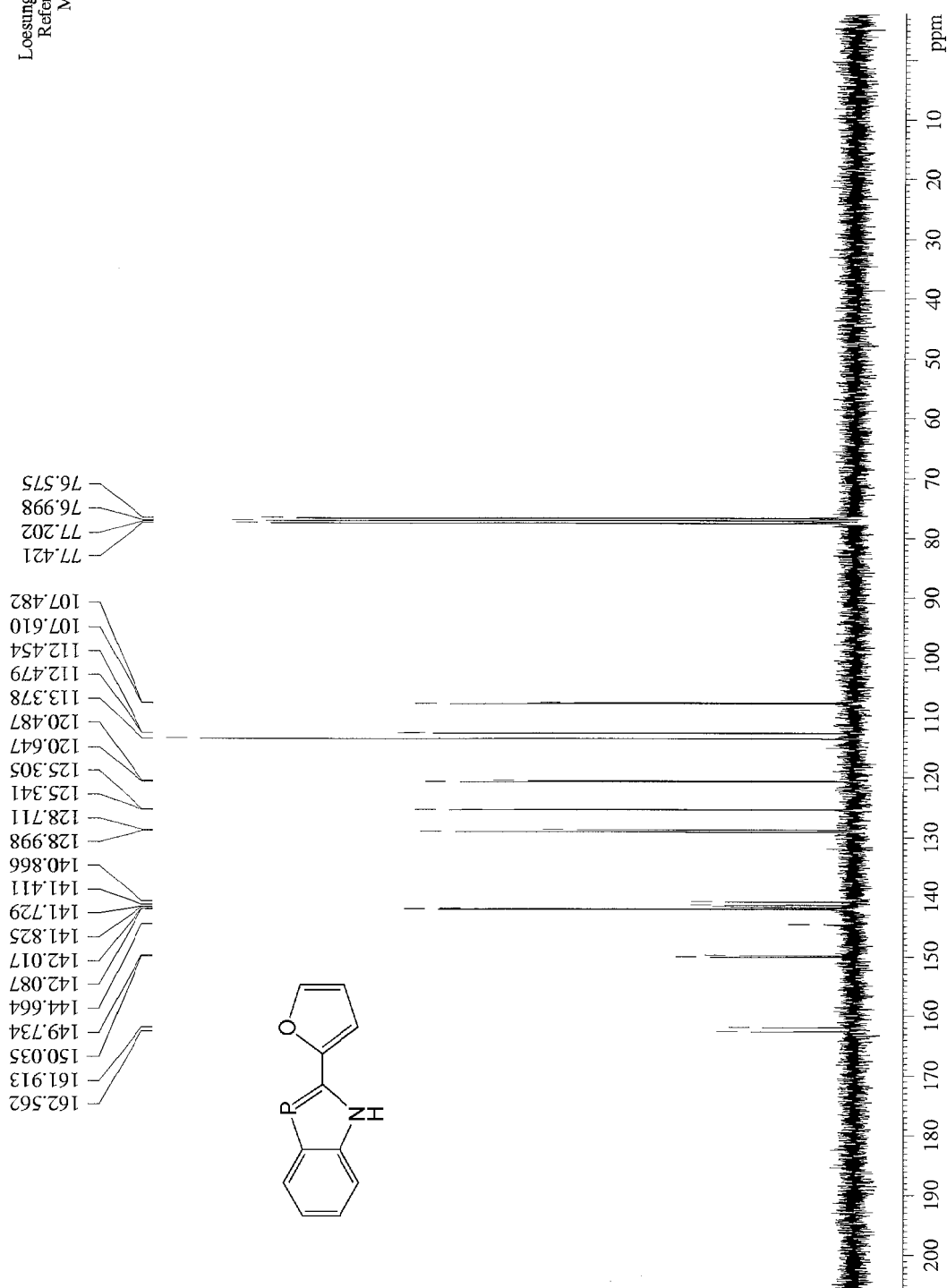
¹³C NMR Spectrum of compound 3f.

Ernst-Moritz-Arndt-Universitaet Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandentkoppelt)
Bhaskar Reddy A: BL35C
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Loesungsmittel 77.0 = ppm
Messtemperatur: 298K (25 oC)

Current Data Parameters
NAME BH_BL35C
EXPNO 13
PROCNO 1
USER hein

F2 – Acquisition Parameters
Date_ 20060630
Time 8.21
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 65536
NS 264
DS 0
SWH 21739.131 Hz
FIDRES 0.331713 Hz
AQ 1.5073780 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.00002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters
SI 32768
SF 75.4677552 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00



¹³C NMR Spectrum of compound 3g.

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandentkoppelt)

Bhaskar Reddy BL36UD

Lösungsmittel: Deuteriochloroform, CDCl₃

Referenz: TMS = 0.0 ppm

Messtemperatur: 298K (25 °C)

Messzeit: 17 Stunden

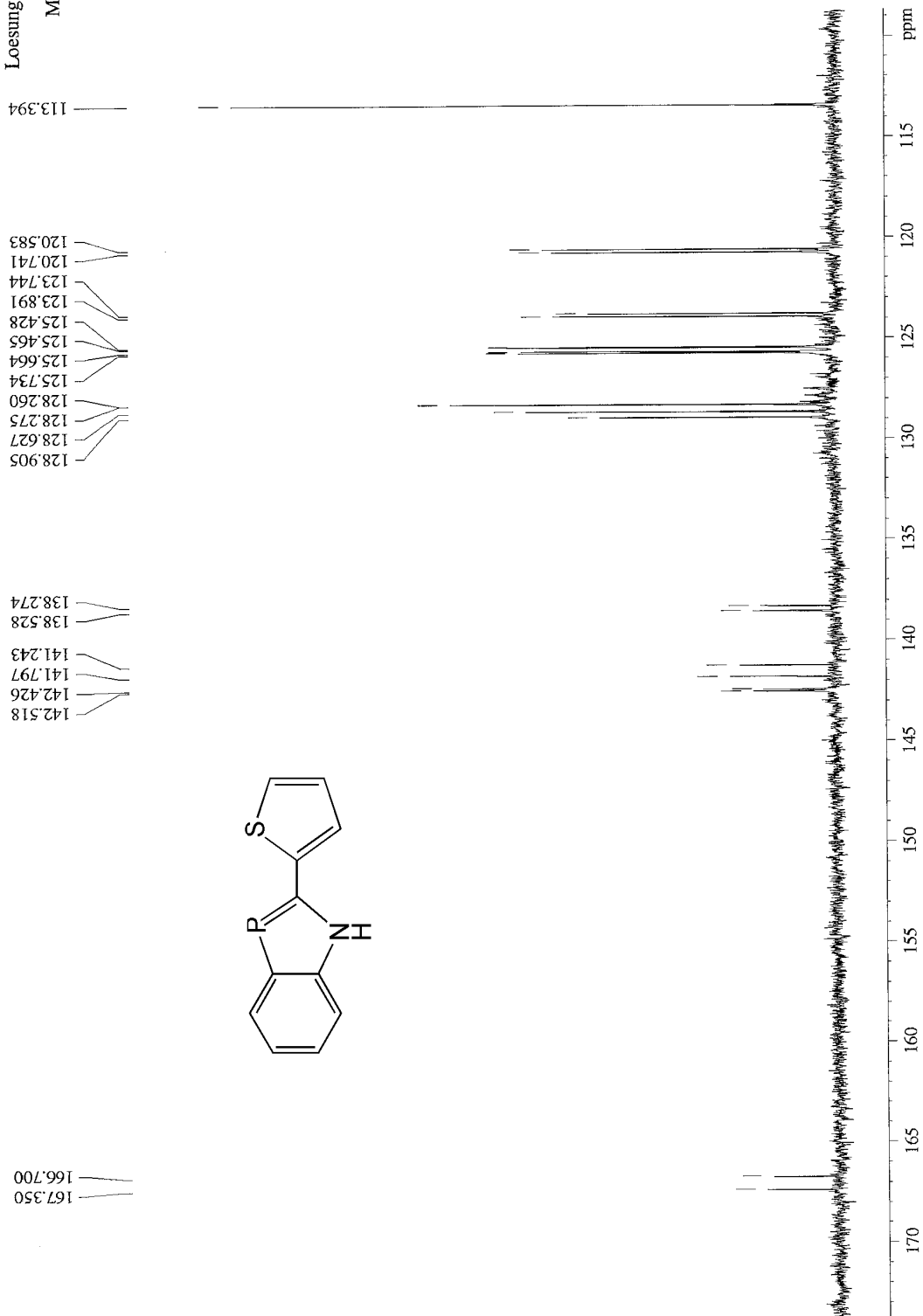
Current Data Parameters
NAME BH BL36UD
EXPNO 13
PROCNO 1
USER hein

F2 – Acquisition Parameters

Date_ 20060814
Time 12.38
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg
TD 65536
NS 17408
DS 0
SWH 21739.131 Hz
FIDRES 0.331713 Hz
AQ 1.5075780 sec
RG 32768
DW 23.000 usec
DE 32.86 usec
TE 298.0 K
D12 0.0002000 sec
DL6 30.00 dB
D1 2.00000000 sec
CPDPRG waltz16
P31 110.00 usec
D11 0.03000000 sec
DL5 22.00 dB
P1 2.50 usec
SFO1 75.4761177 MHz
NUCLEUS 13C

F2 – Processing parameters

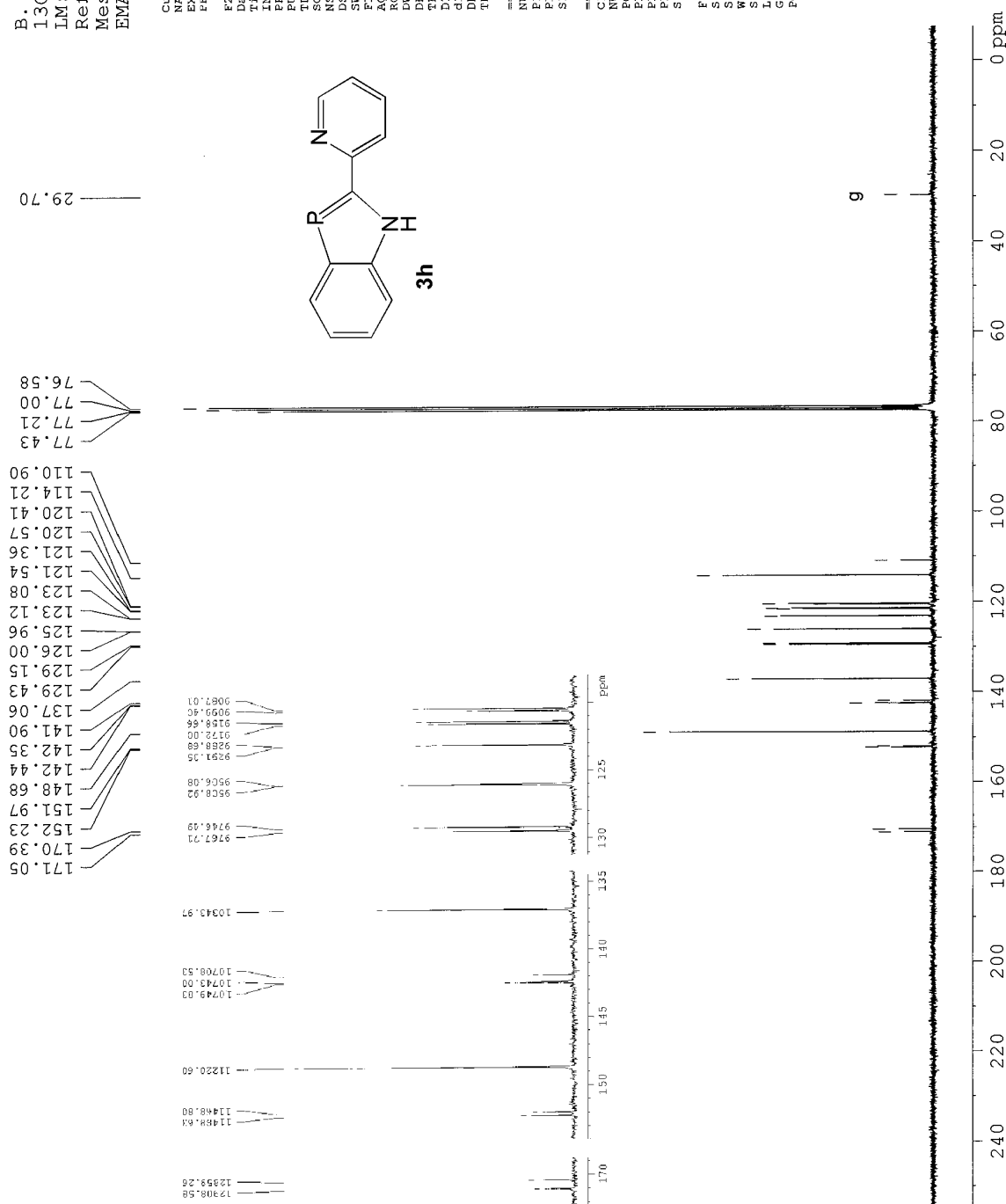
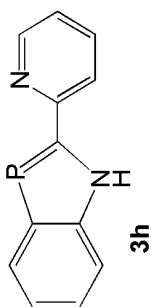
SF 32768
SF 75.4677486 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 0.60



¹³C NMR Spectrum of compound 3h.

B. NIAZ:BN-72C
13C{1H}-NMR-Spektrum
LM: CDCl₃
Referenz: LM = 77.0 ppm
Messzeit: 12 Stunden
EMAU, AVANCE II - 300

Current Data Parameters
NAME NI_BN-72c
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100603
Time 8.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl₃
NS 18343
DSH 21739.131 Hz
SFR 0.663426 Hz
FIDRES 0.7537140 sec
AQ 32800
RG 23.000 usec
DW 6.00 usec
DE 673.2 K
TE 2.00000000 sec
D1 0.03000000 sec
d11 1.89999998 sec
DELTA 1
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.80 usec
PL1 -1.00 dB
SFO1 75.4761177 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL14 19.00 dB
PL15 19.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 16384
SF 75.4677478 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.20



g - grease impurity

