

Supporting Information

Ligand-free Suzuki–Miyaura coupling reaction of aryl chloride using a continuous irradiation type microwave and palladium nanoparticle catalyst: effect of a co-existing solid

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General consideration

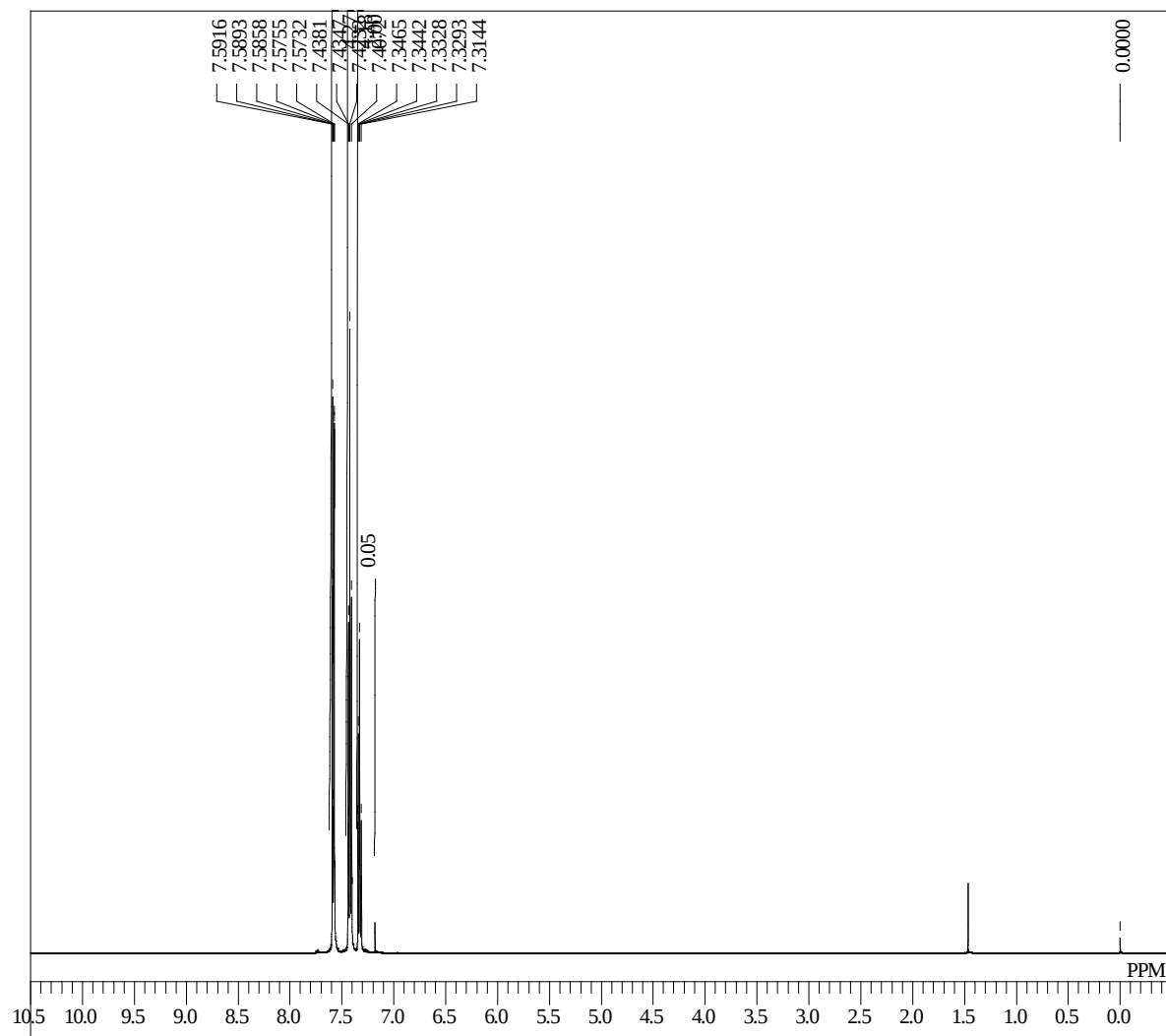
¹H and ¹³C NMR spectra were recorded on JEOL JNM-AL-400 (400 MHz) with tetramethylsilane as an internal standard. ICP-S spectra were obtained on an AGLIRNT ICP-MS 7500 CS. HPLC was carried out using a Mightysil RP-18 GP 150-4.6 (3 μm, KANTO CHEMICAL CO., INC.; column), and HPLC spectra were by JASCO PU-986 Plus (pump), JASCO MD-2010 Plus (detector), JASCO LC-NET II/ADC (system controller) and GL Science CO 705 (column oven). Chemistation (TOKYO RIKAKIKAI CO., LTD, PPS-CTRL) was used for heating reactions. Microwave heating reactions were carried out using a single mode microwave generator (Anton Paar, Monowave 300) or semiconductor microwave generator (TOKYO RIKAKIKAI CO., LTD, GPS-1000C). Au (mesh) was purchased from Sanwa Metal CO. LTD. Column chromatography was carried out with silica gel (Kanto Chemical Co. Inc., Silica Gel 60 N, spherical neutral) unless otherwise stated.

Unless otherwise indicated, all reactions were carried out with magnetic stirring and under argon atmosphere. Reactions were monitored by thin layer chromatography.

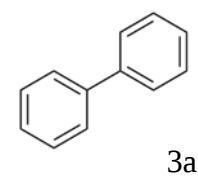
Experiment materials

Solvent were dried by molecular sieves 3A or 4A. Commercial reagents and Argon gas were used as received. TLC (Merck silica gel 60 F²⁵⁴) was used for monitoring reactions.

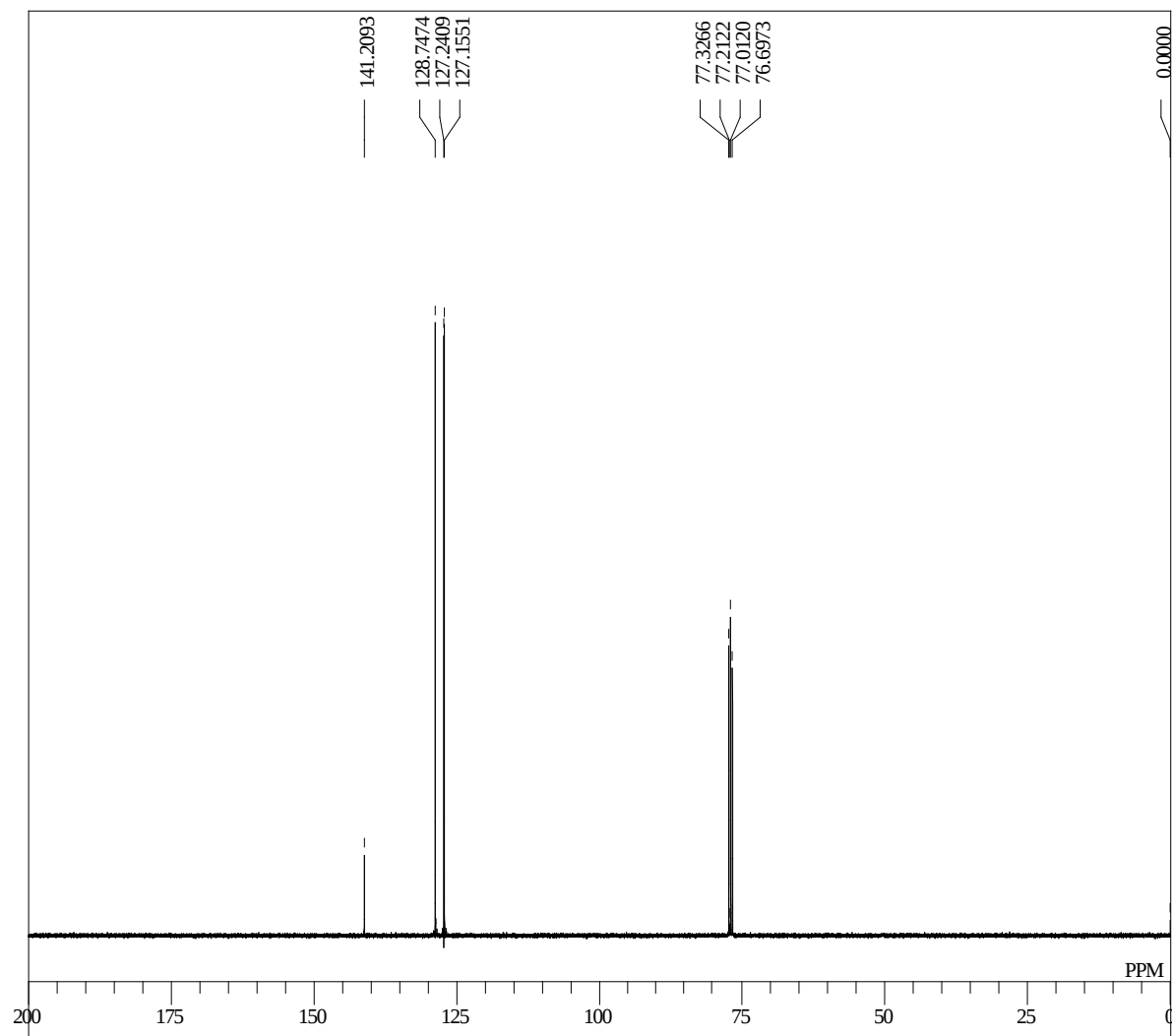
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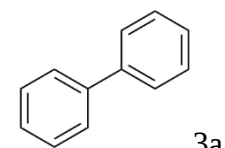
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EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30



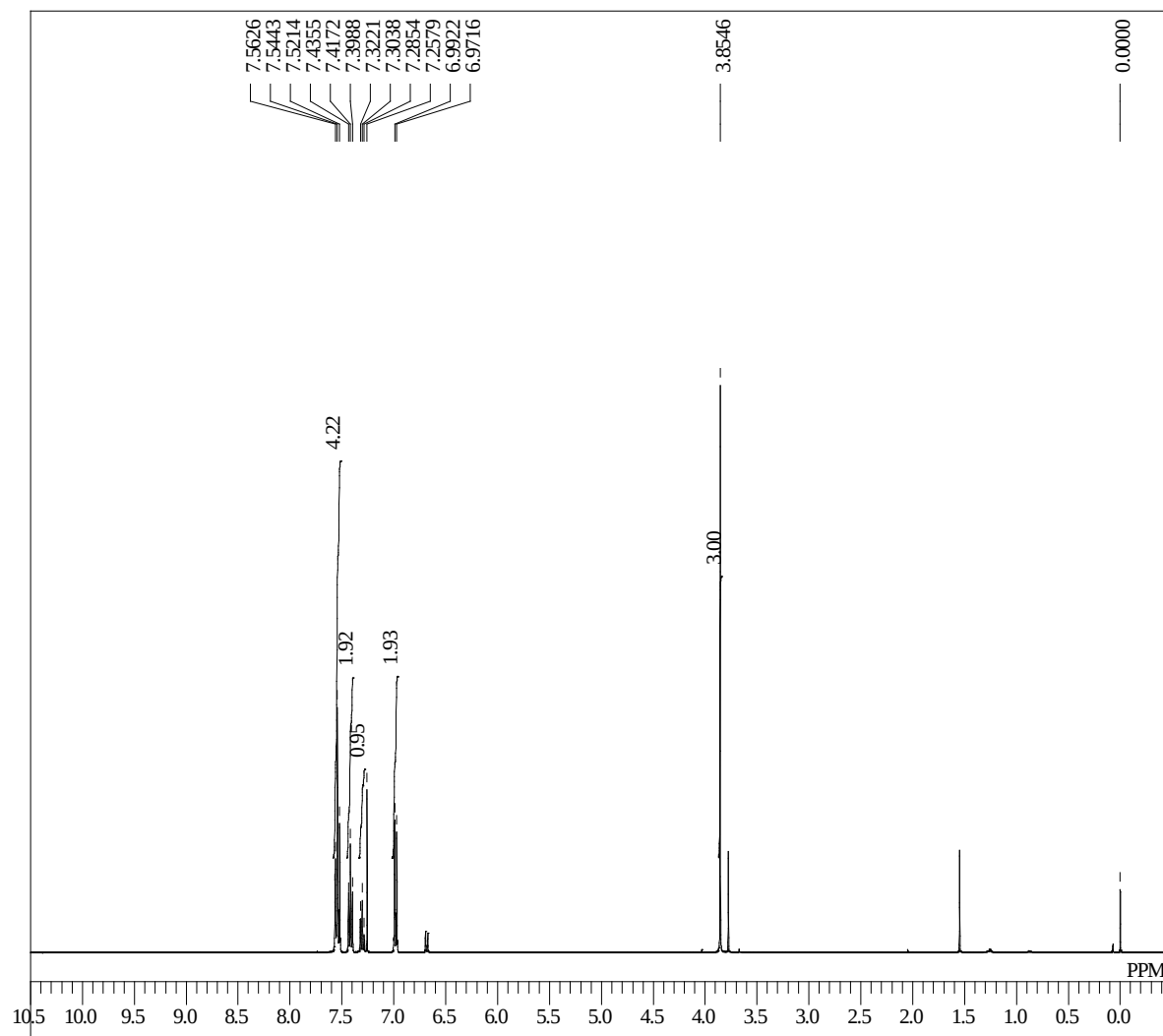
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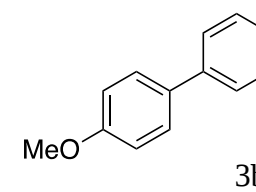
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POINT 26214
FREQU 25125.63 Hz
SCANS 1024
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PW1 3.17 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60



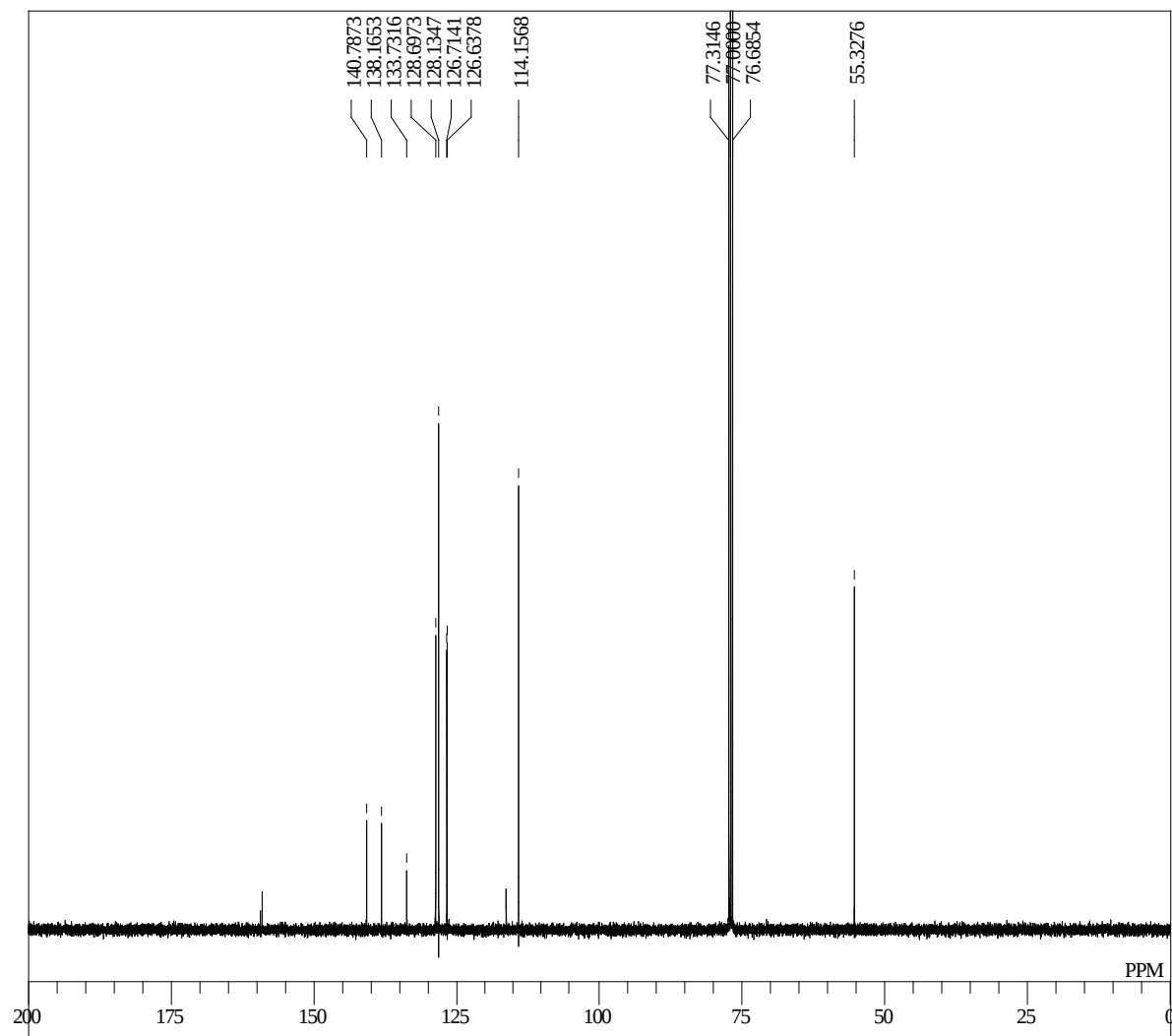
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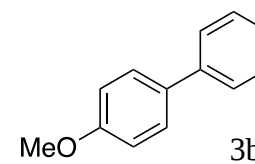
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 POINT 13107
 FREQU 6002.40 Hz
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 ACQTIM 2.1837 sec
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 RGAIN 50



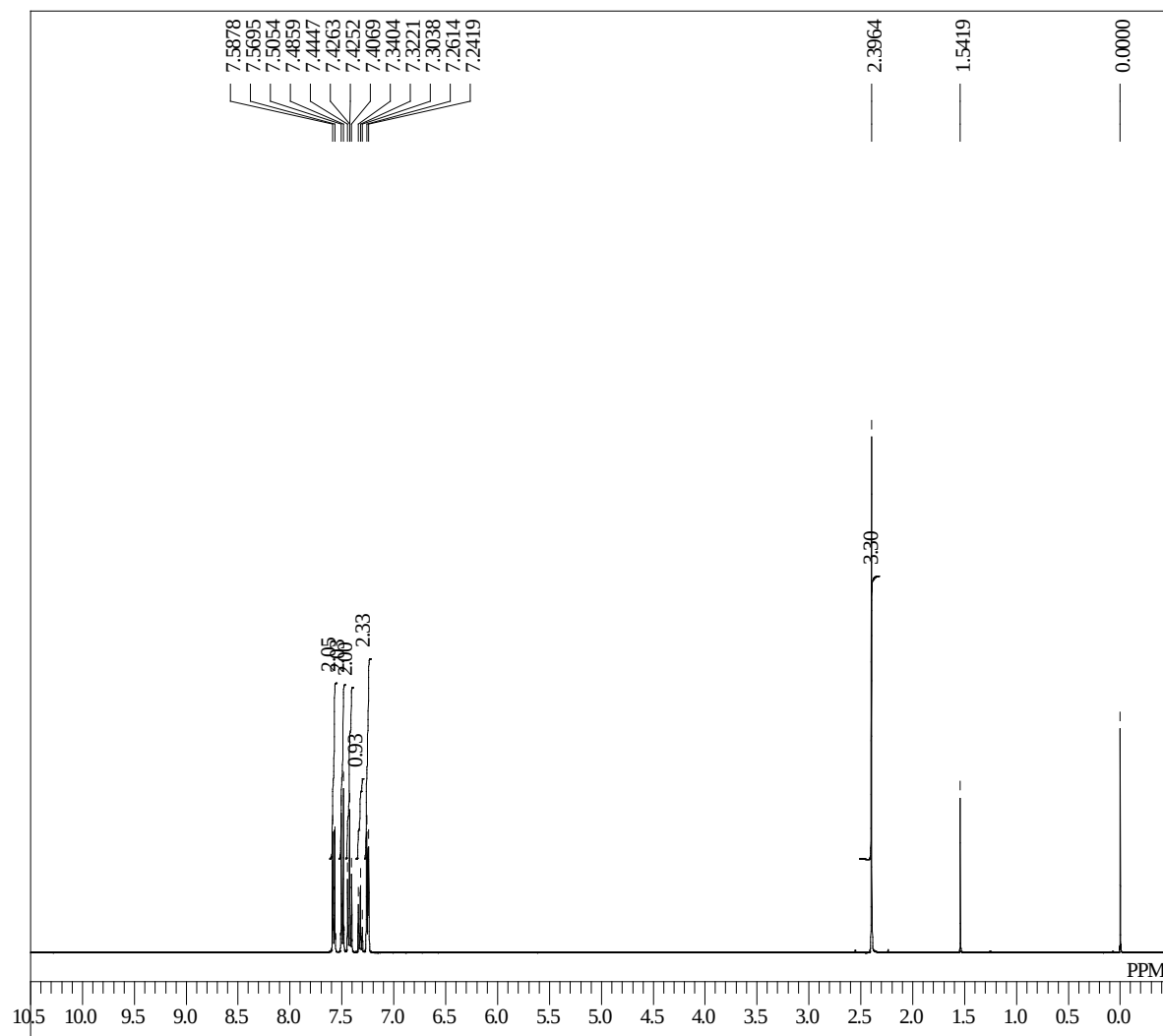
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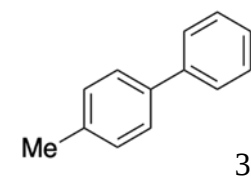
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 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 1030
 ACQTM 1.0433 sec
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 CTEMP 19.0 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
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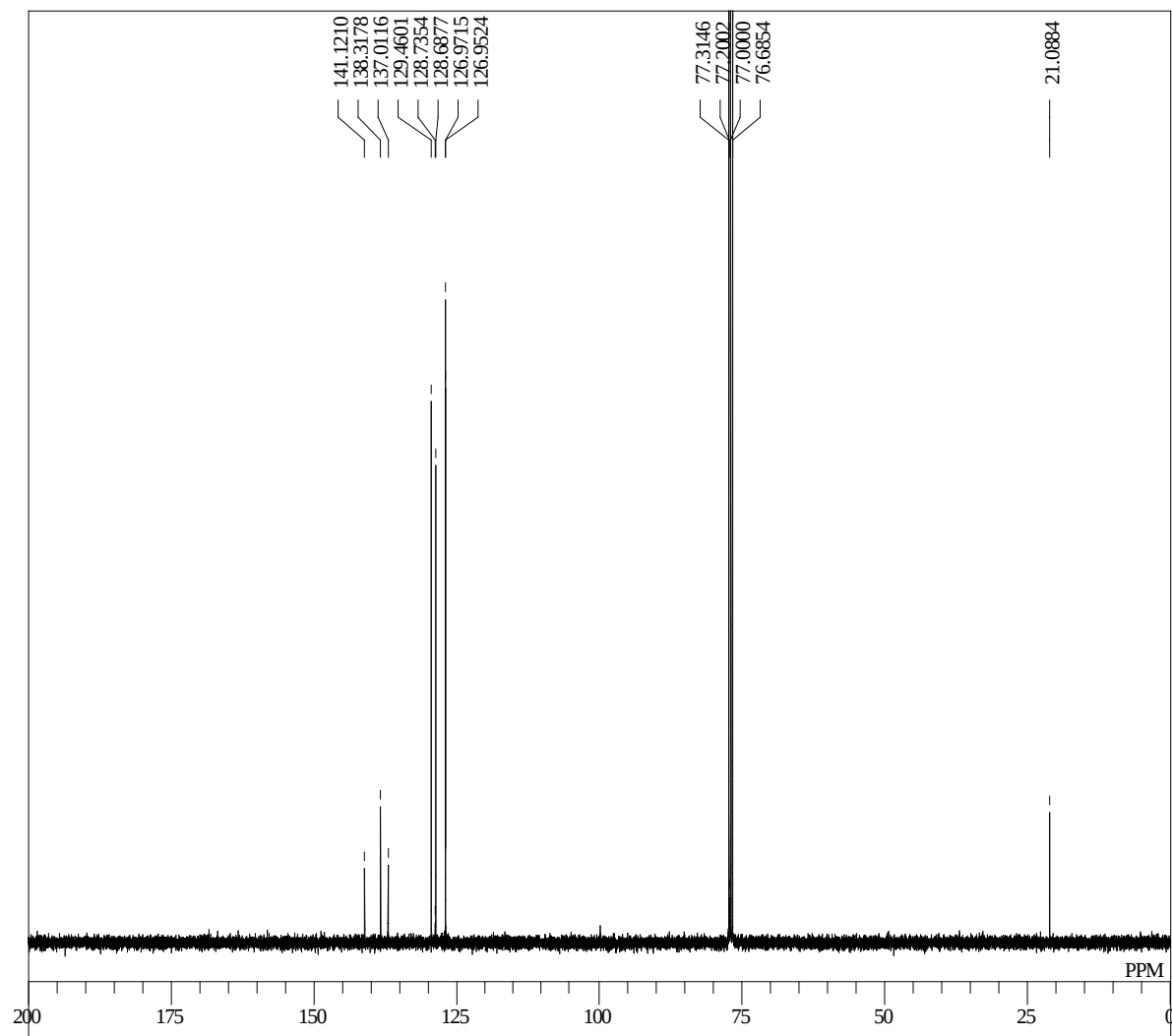
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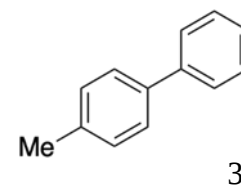
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 POINT 13107
 FREQU 6002.40 Hz
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 ACQTM 2.1837 sec
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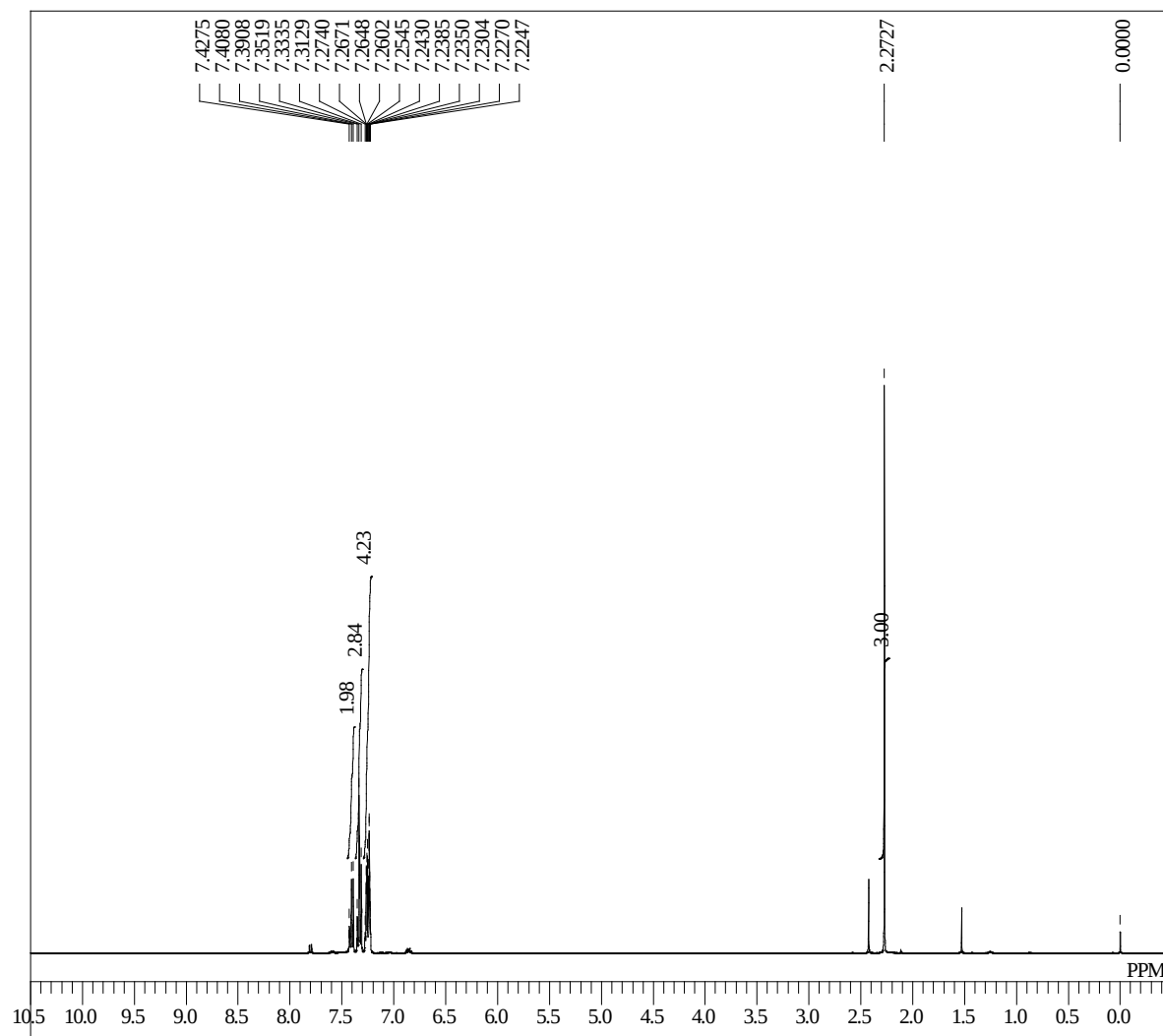
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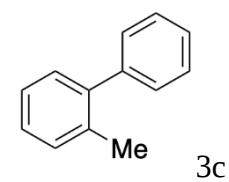
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 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 1030
 ACQTM 1.0433 sec
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 IRNUC 1H
 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 60



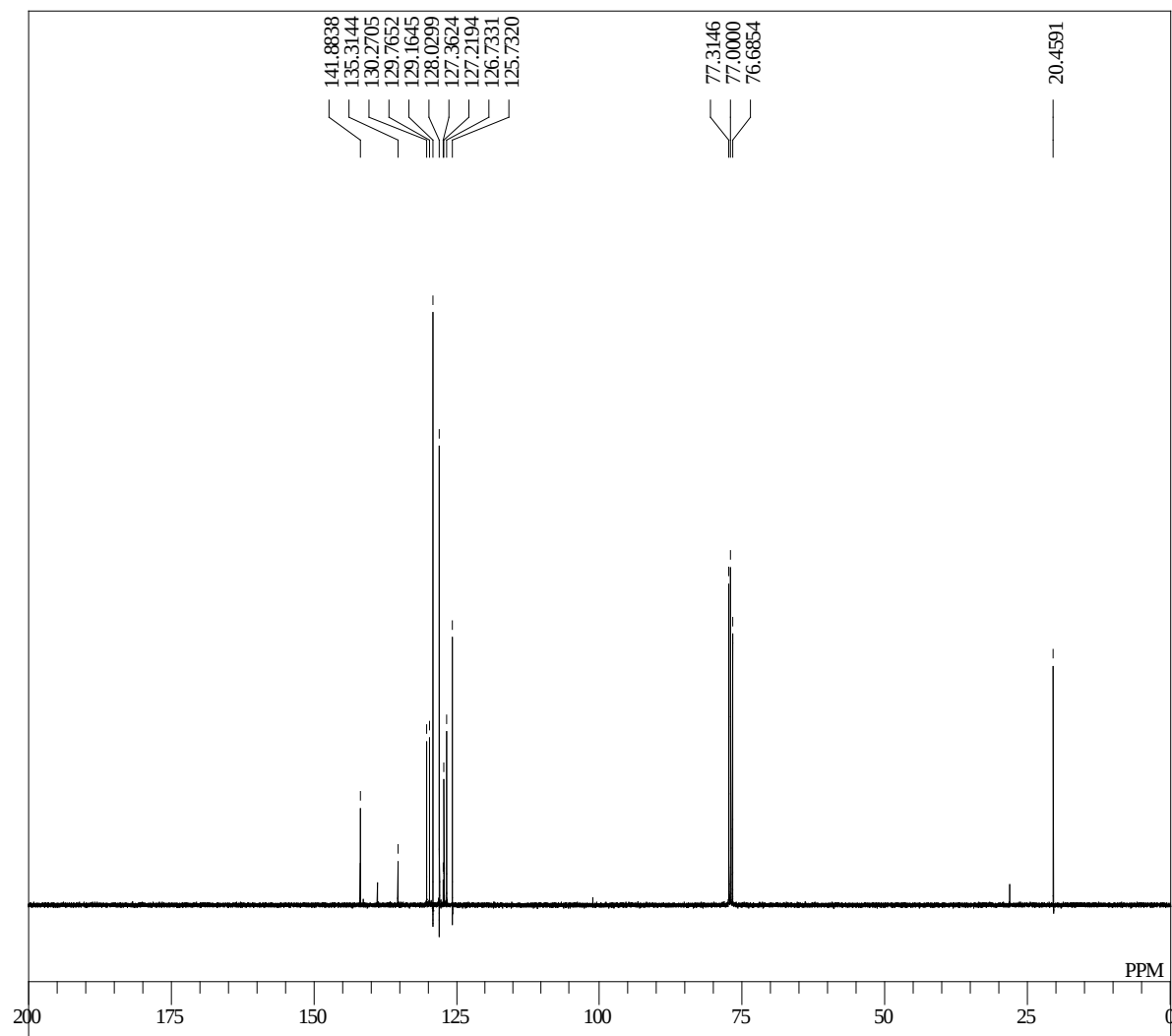
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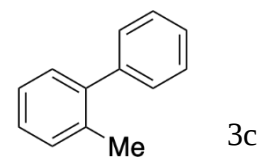
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 OBFIN 7.29 Hz
 POINT 13107
 FREQU 6002.40 Hz
 SCANS 16
 ACQTIM 2.1837 sec
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 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 40



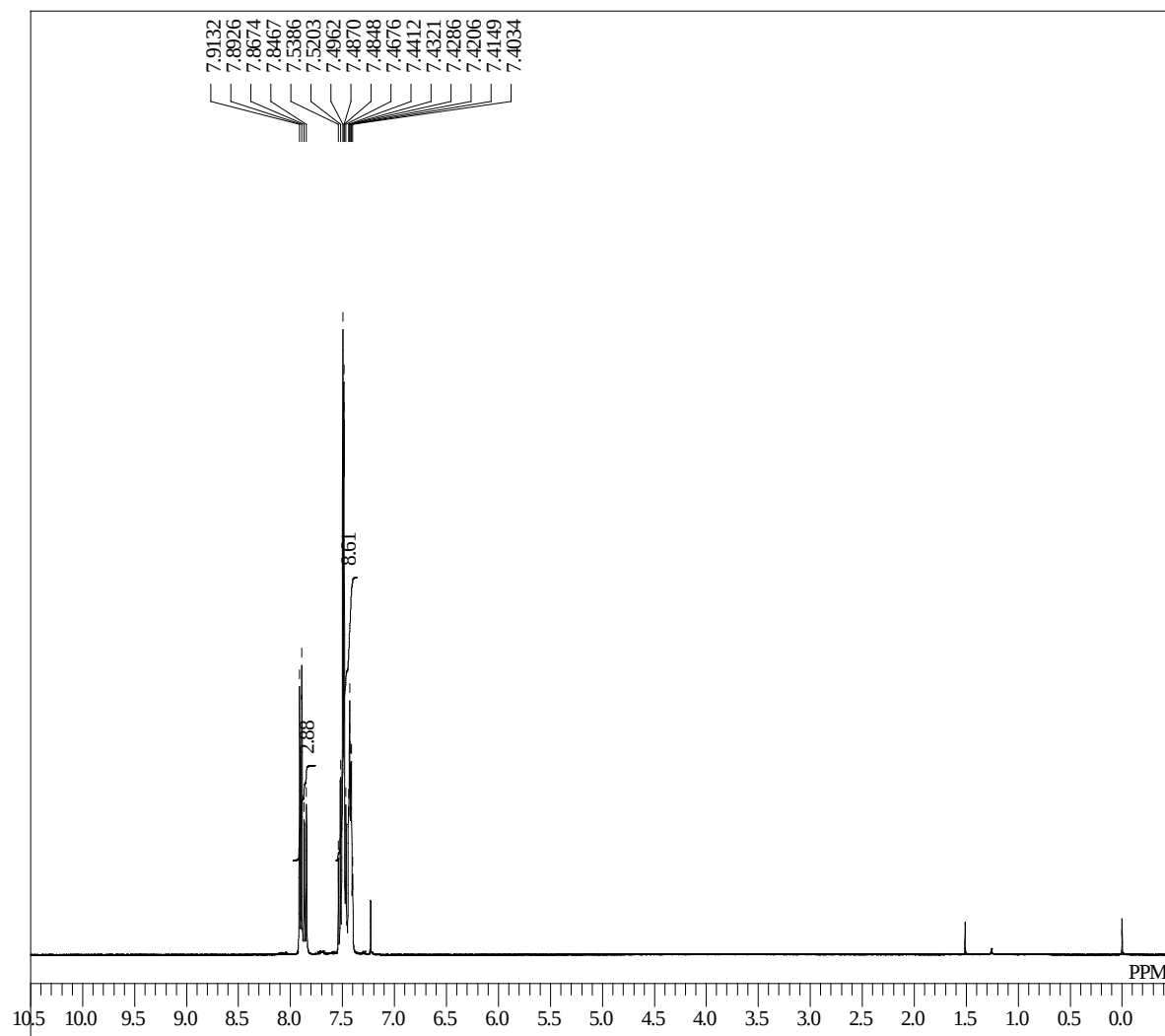
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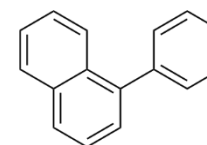
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 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 1024
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
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 CTEMP 19.1 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 60



single_pulse

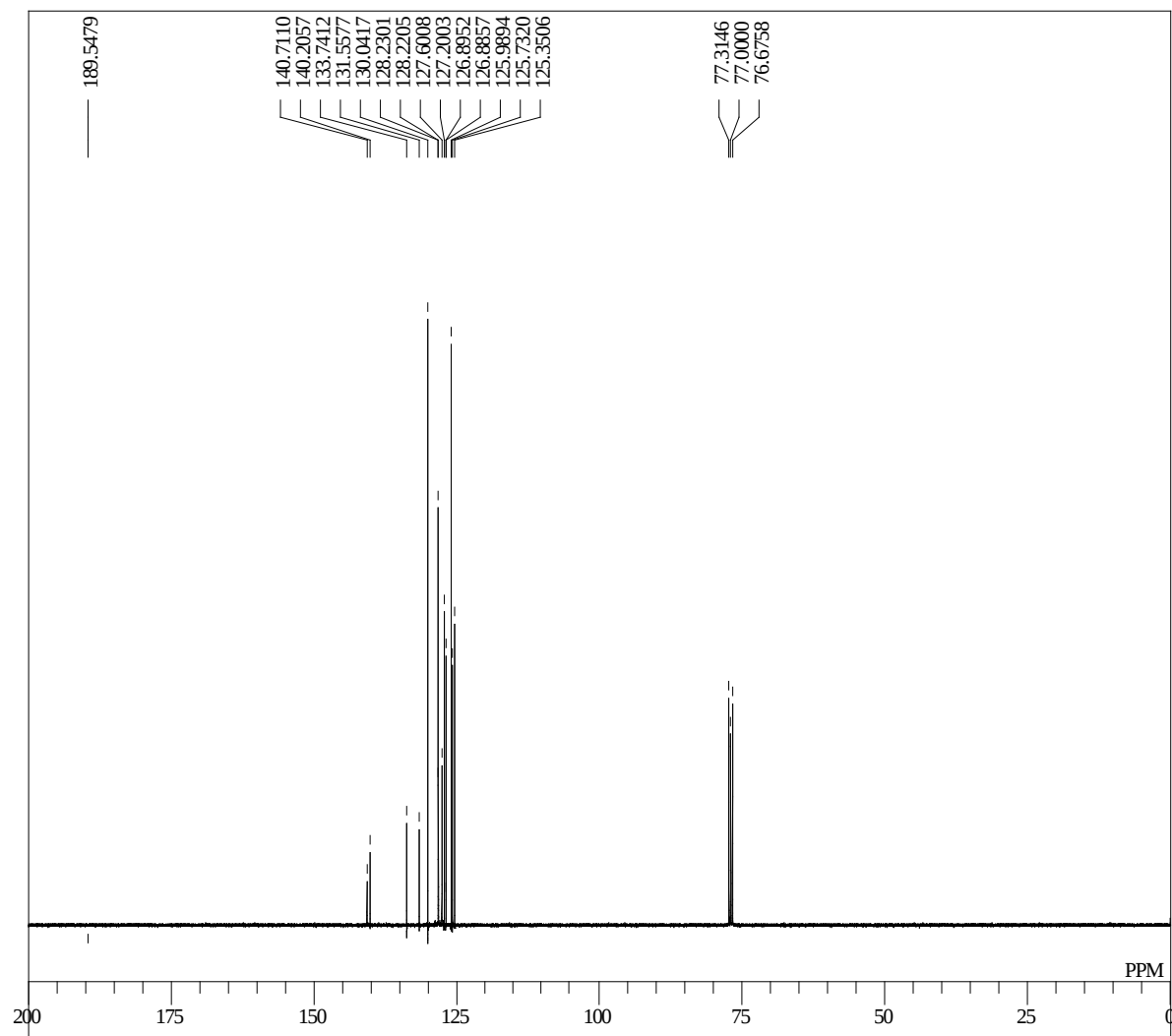


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FREQU 6002.40 Hz
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CTEMP 19.1 c
SLVNT CDCL3
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BF 0.12 Hz
RGAIN 40

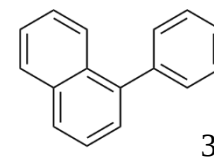


3j

single pulse decoupled gated NOE

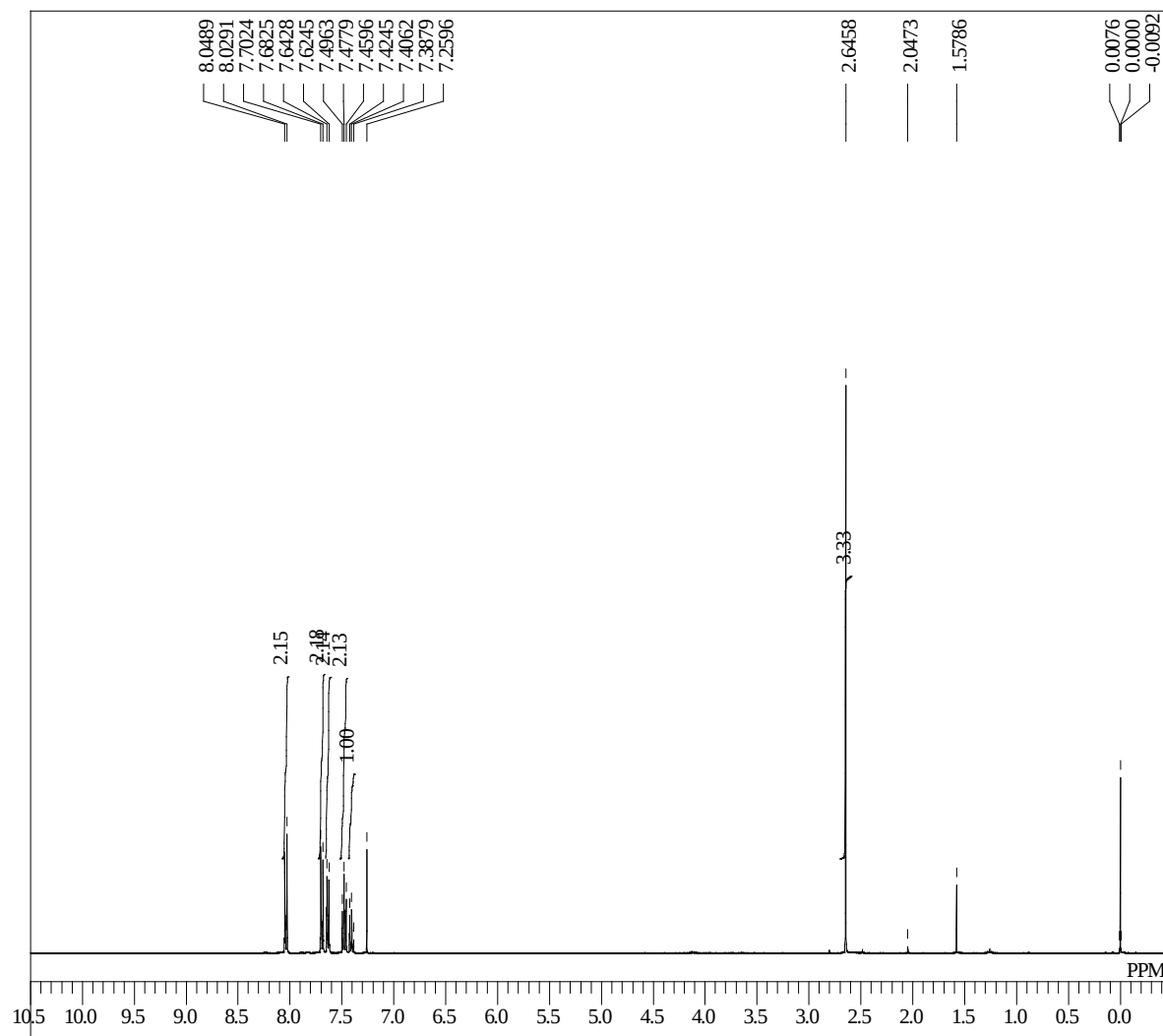


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OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
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ACQTM 1.0433 sec
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PW1 3.17 usec
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SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

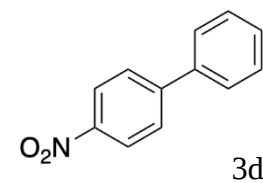


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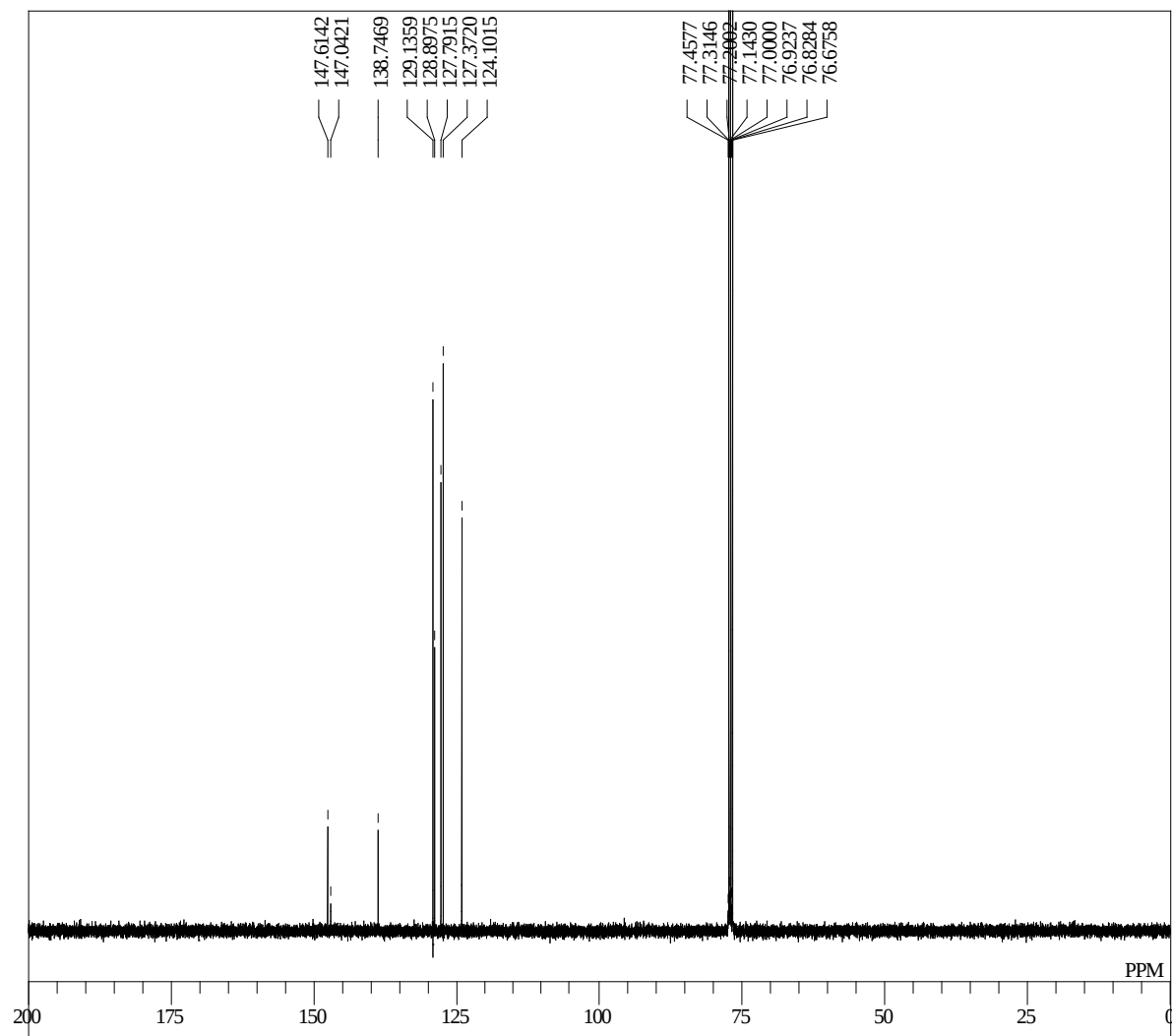
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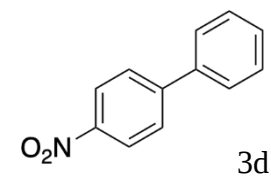
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 POINT 13107
 FREQU 8000.00 Hz
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 ACQTM 1.6384 sec
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 BF 0.12 Hz
 RGAIN 46



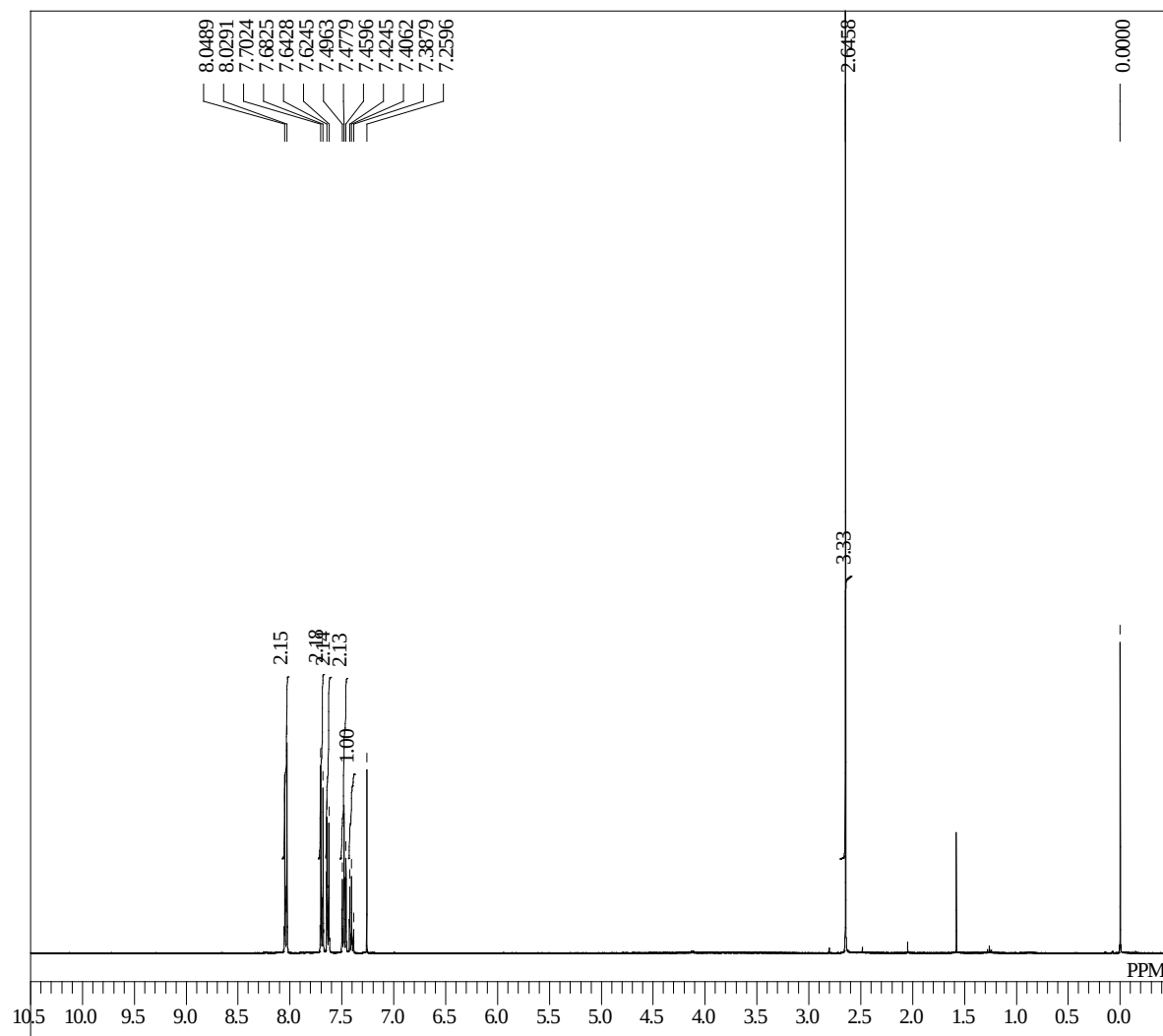
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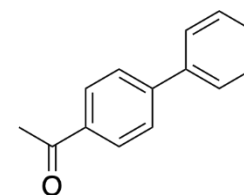
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 EXMOD carbon.jxp
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 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 1120
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
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 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 60



single_pulse

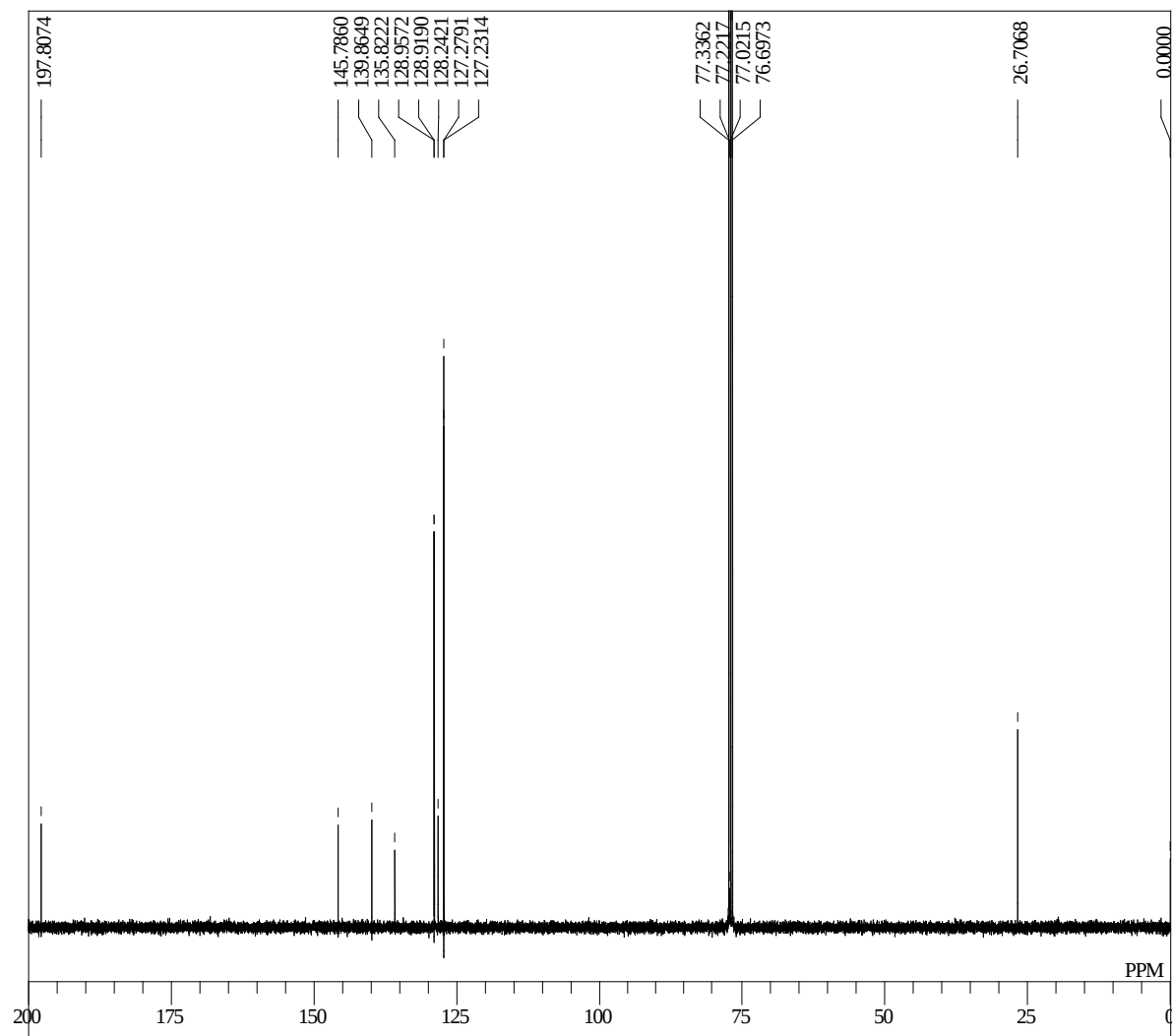


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 FREQU 8000.00 Hz
 SCANS 16
 ACQTIM 1.6384 sec
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 BF 0.12 Hz
 RGAIN 46

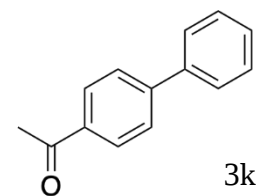


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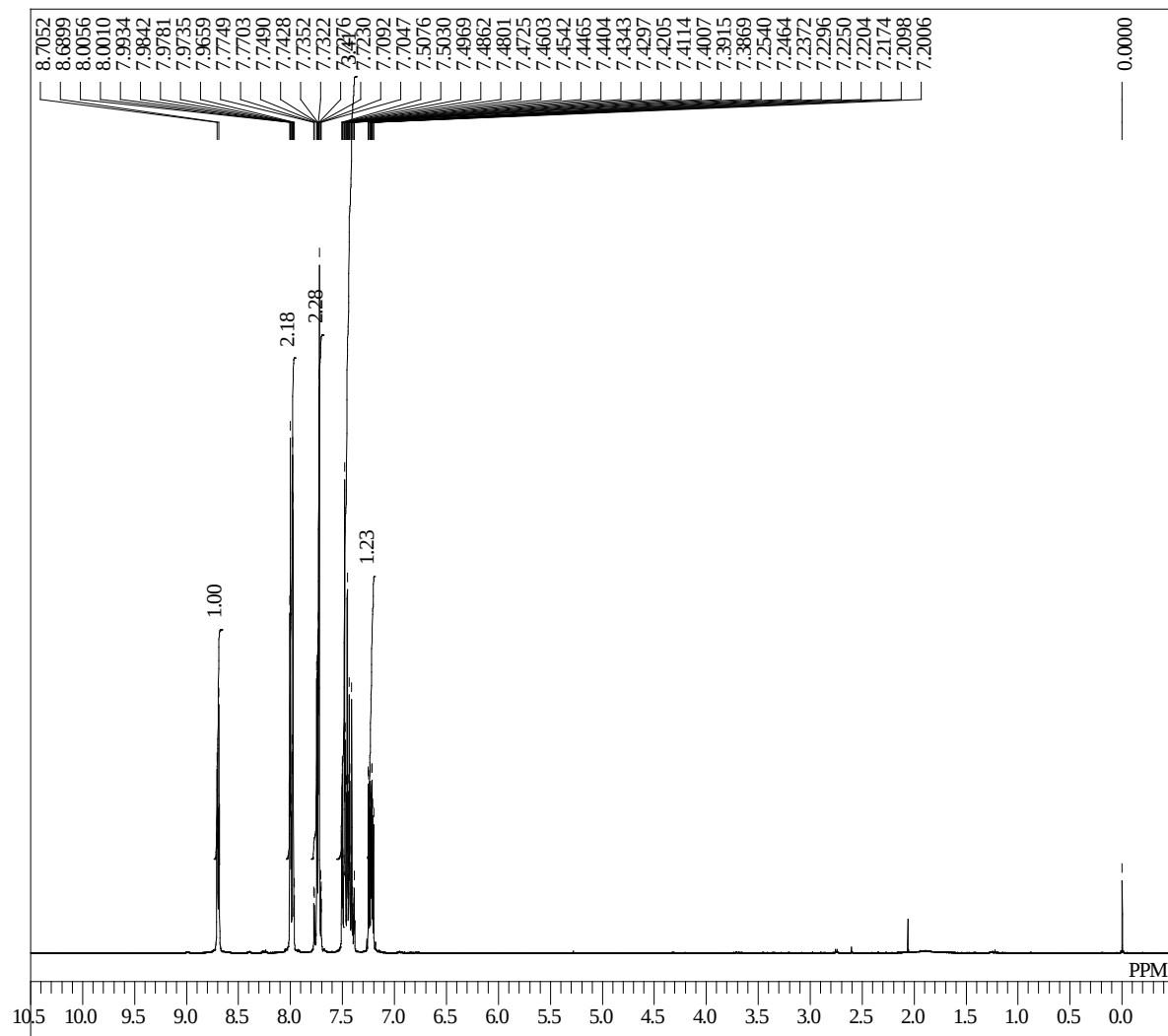
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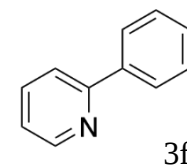
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POINT 26214
FREQU 25125.63 Hz
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RGAIN 60



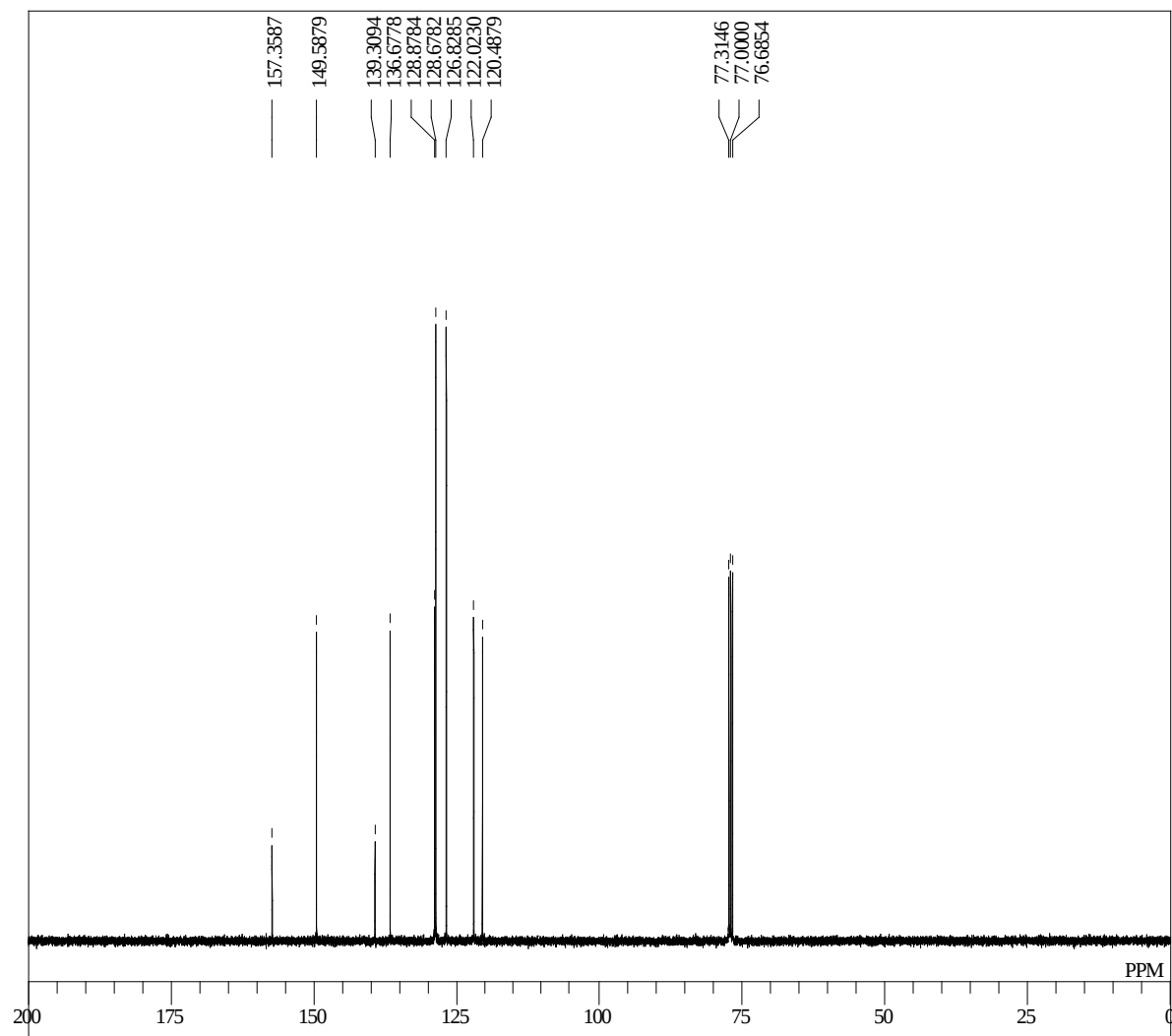
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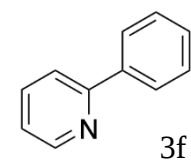
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 FREQU 6016.85 Hz
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 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 36



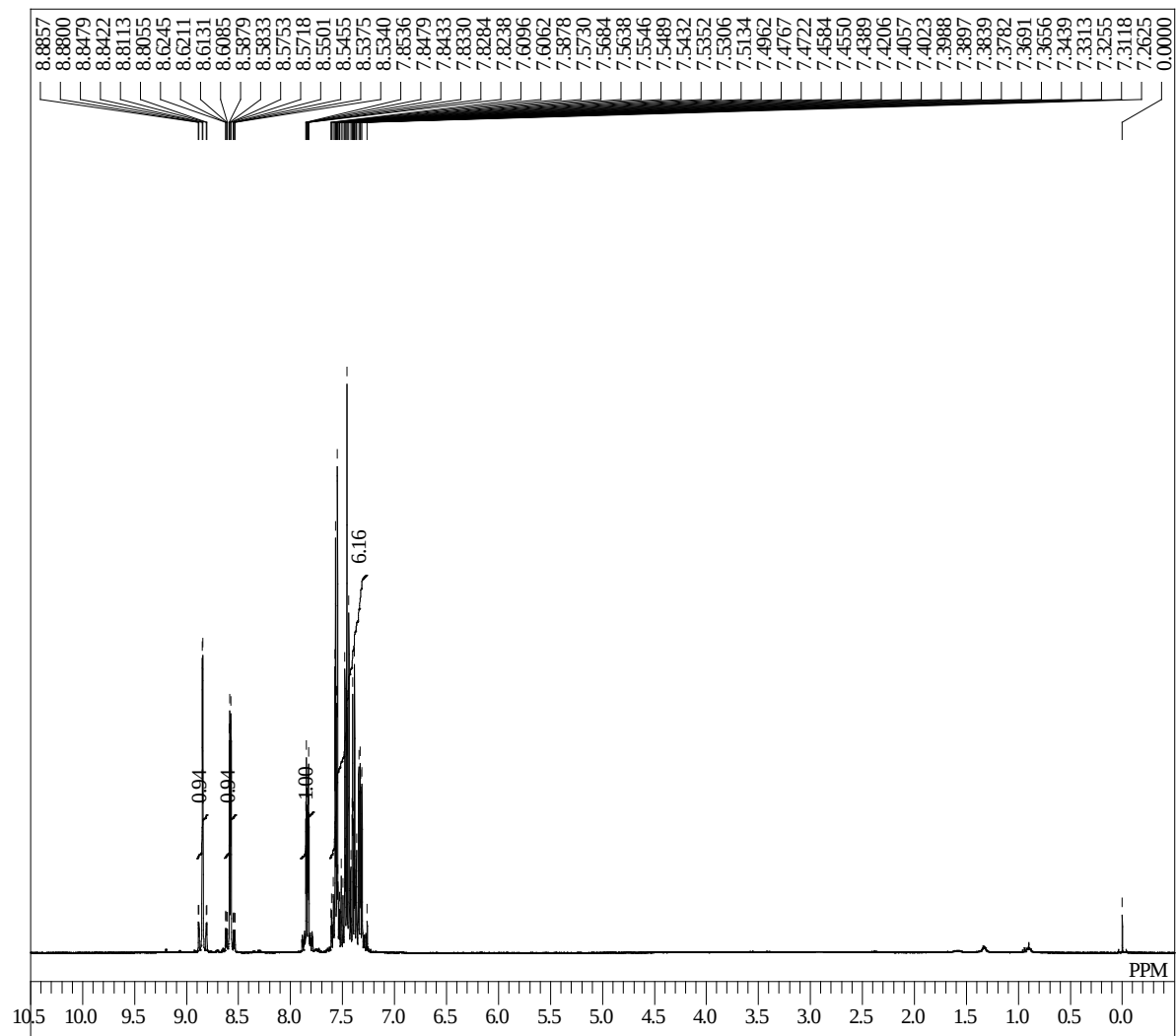
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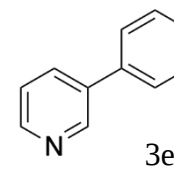
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 OBFIN 5.86 Hz
 POINT 26214
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 SCANS 1024
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 60



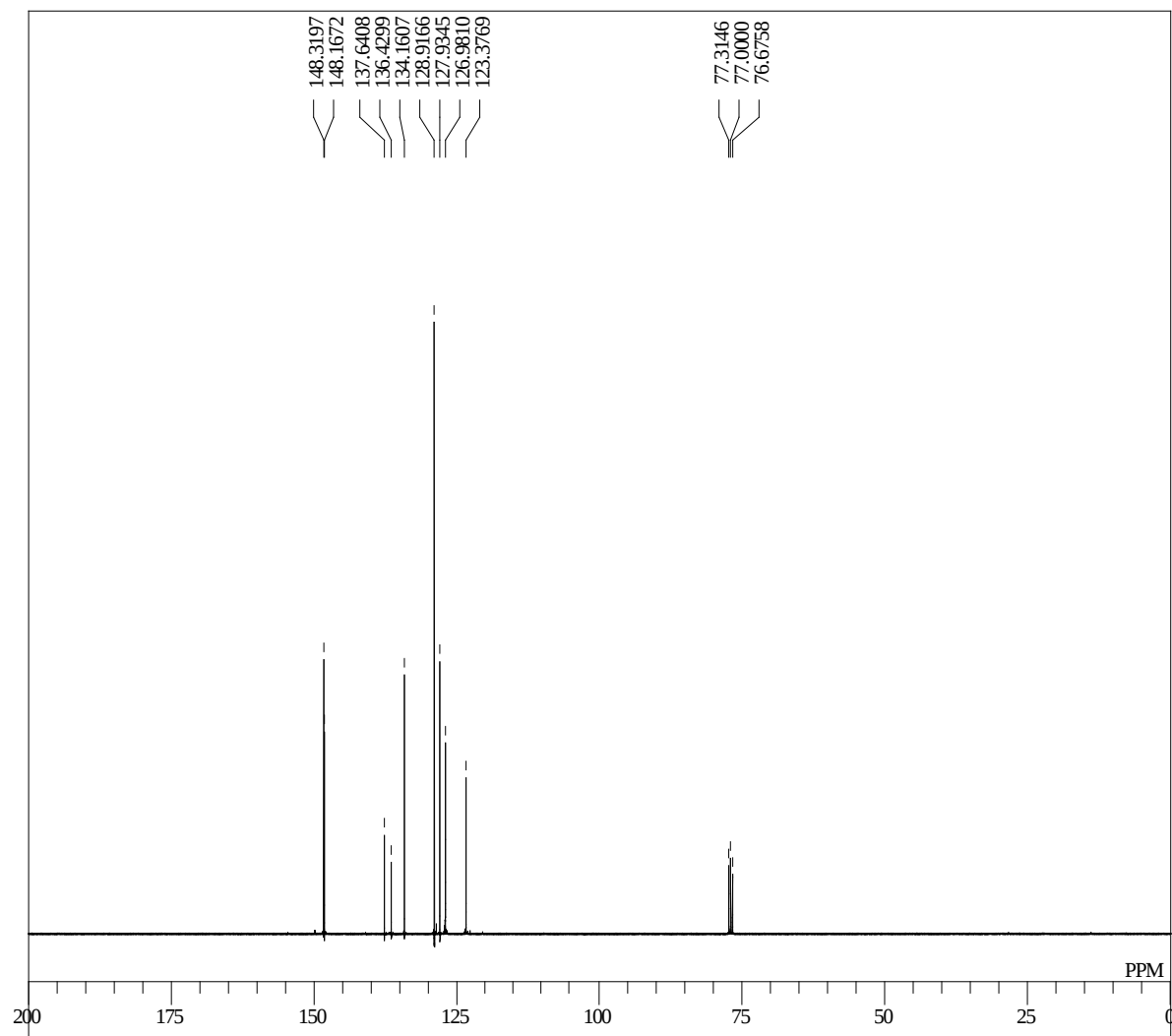
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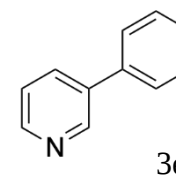
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 OBFIN 7.29 Hz
 POINT 13107
 FREQU 6002.40 Hz
 SCANS 8
 ACQTIM 2.1837 sec
 PD 2.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 19.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28



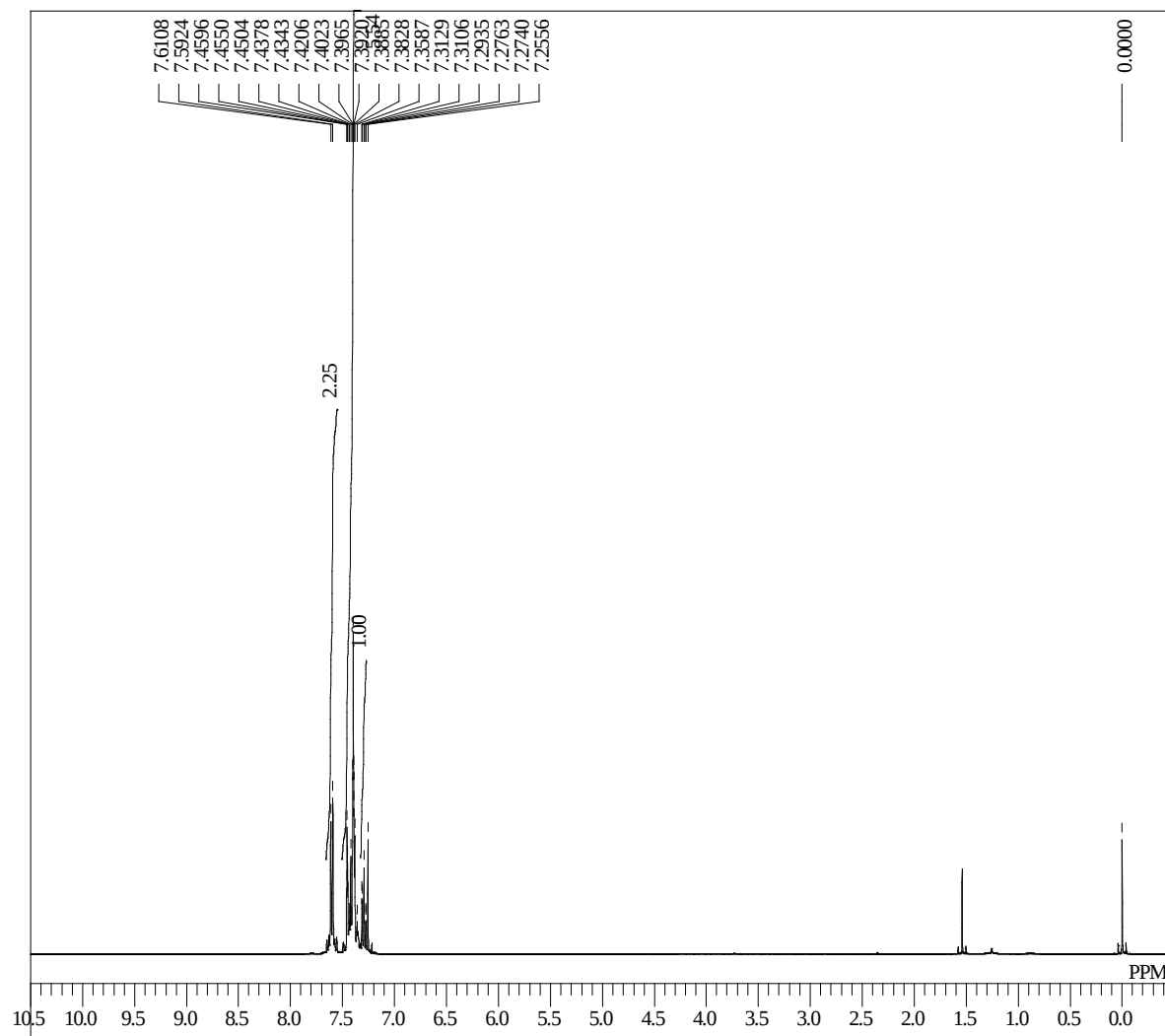
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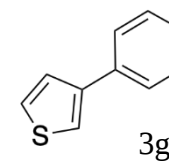
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 EXMOD carbon.jxp
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 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 1024
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.4 c
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 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 60



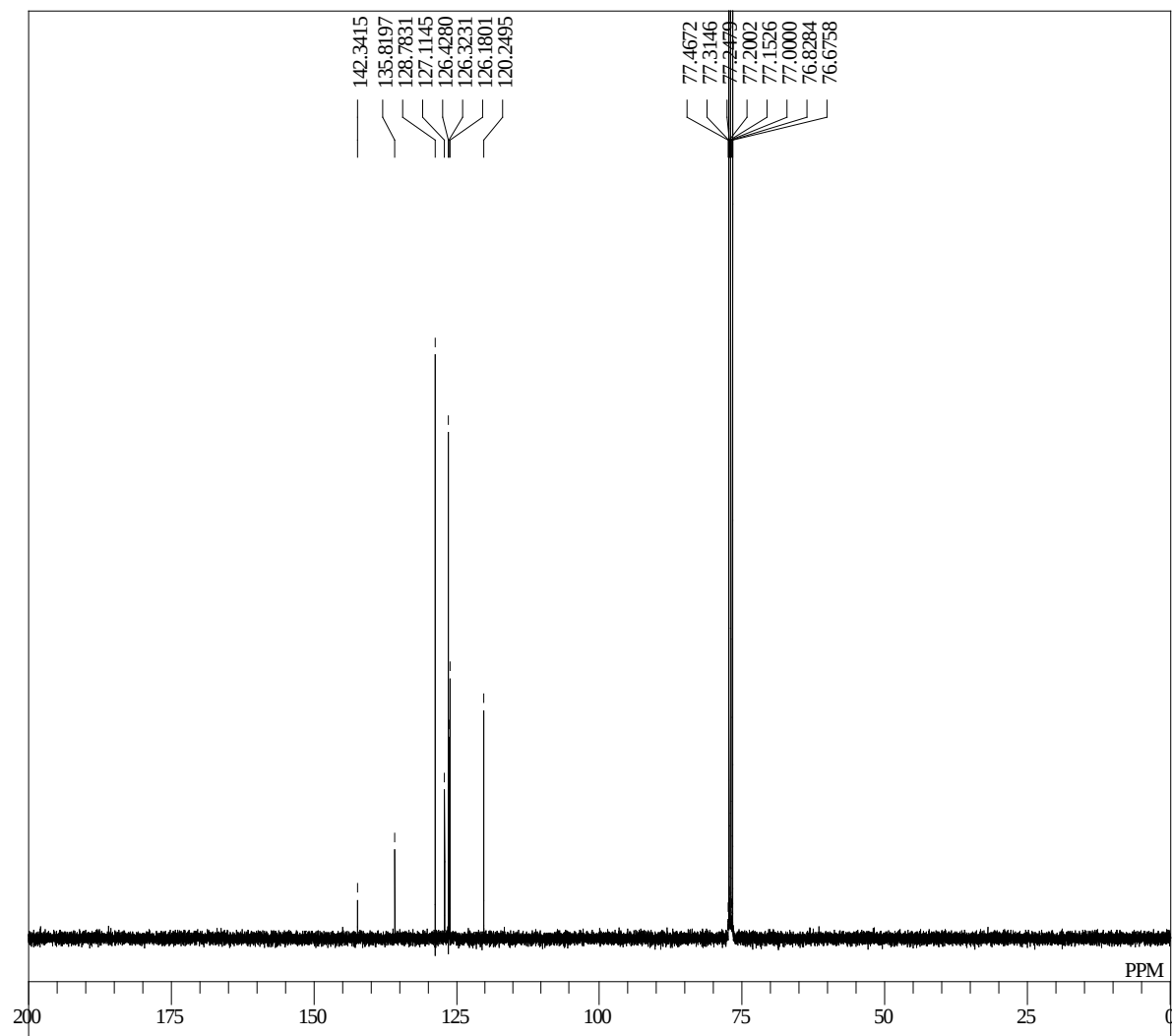
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EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCl₃
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50



single pulse decoupled gated NOE



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 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 1120
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC ¹H
 CTEMP 19.6 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 60

