

Supporting Information of
Synthesis of a Pentacoordinate
Germanium Compound Possessing a γ -Lactone
and a Dative-Bonding Carboxylic Acid

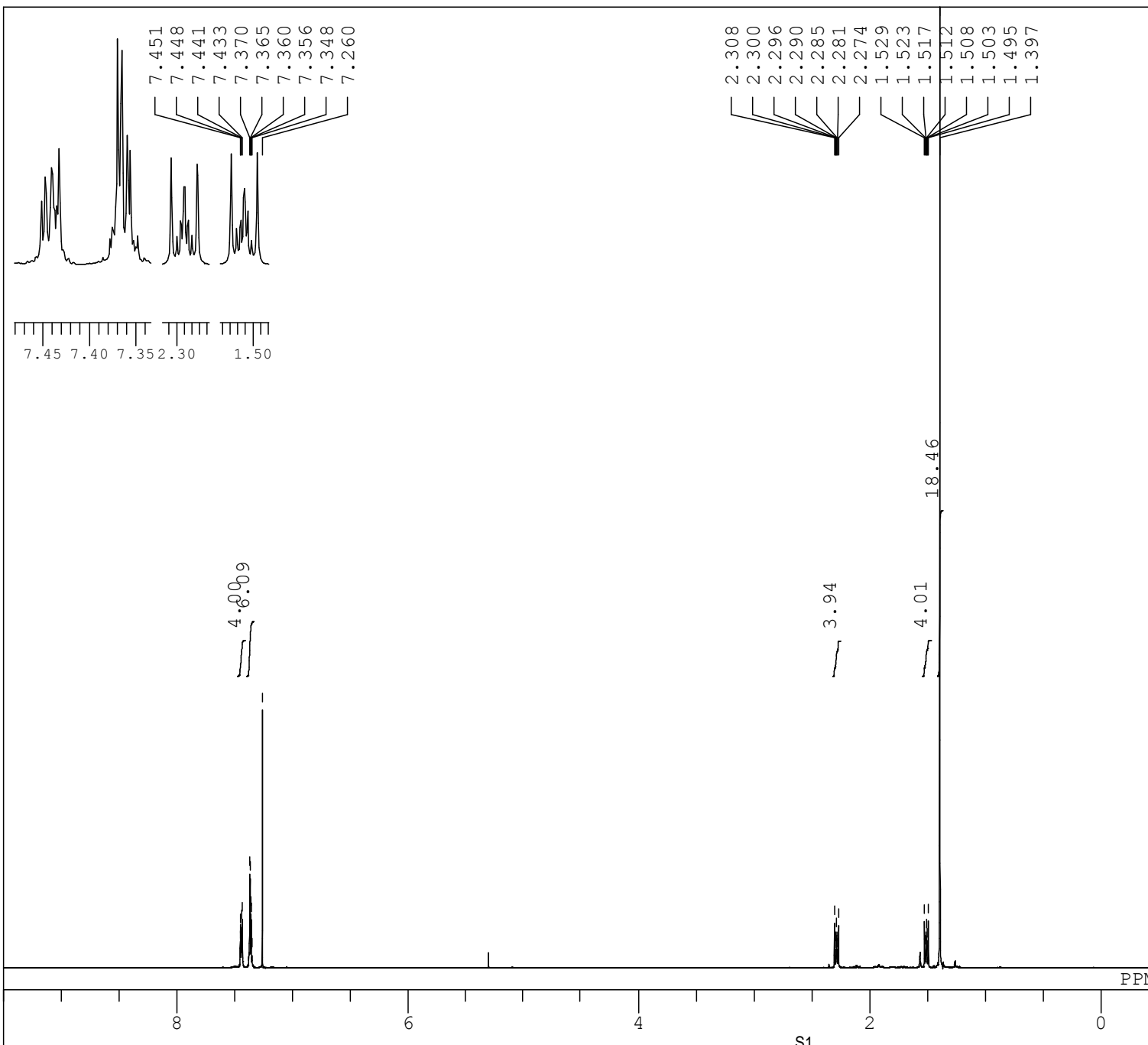
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Division of Materials Science, Graduate School of Science and Technology
Nara Institute of Science and Technology (NAIST)

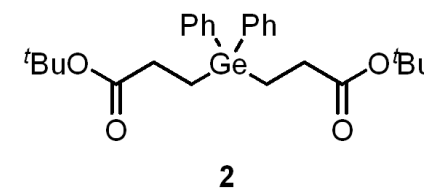
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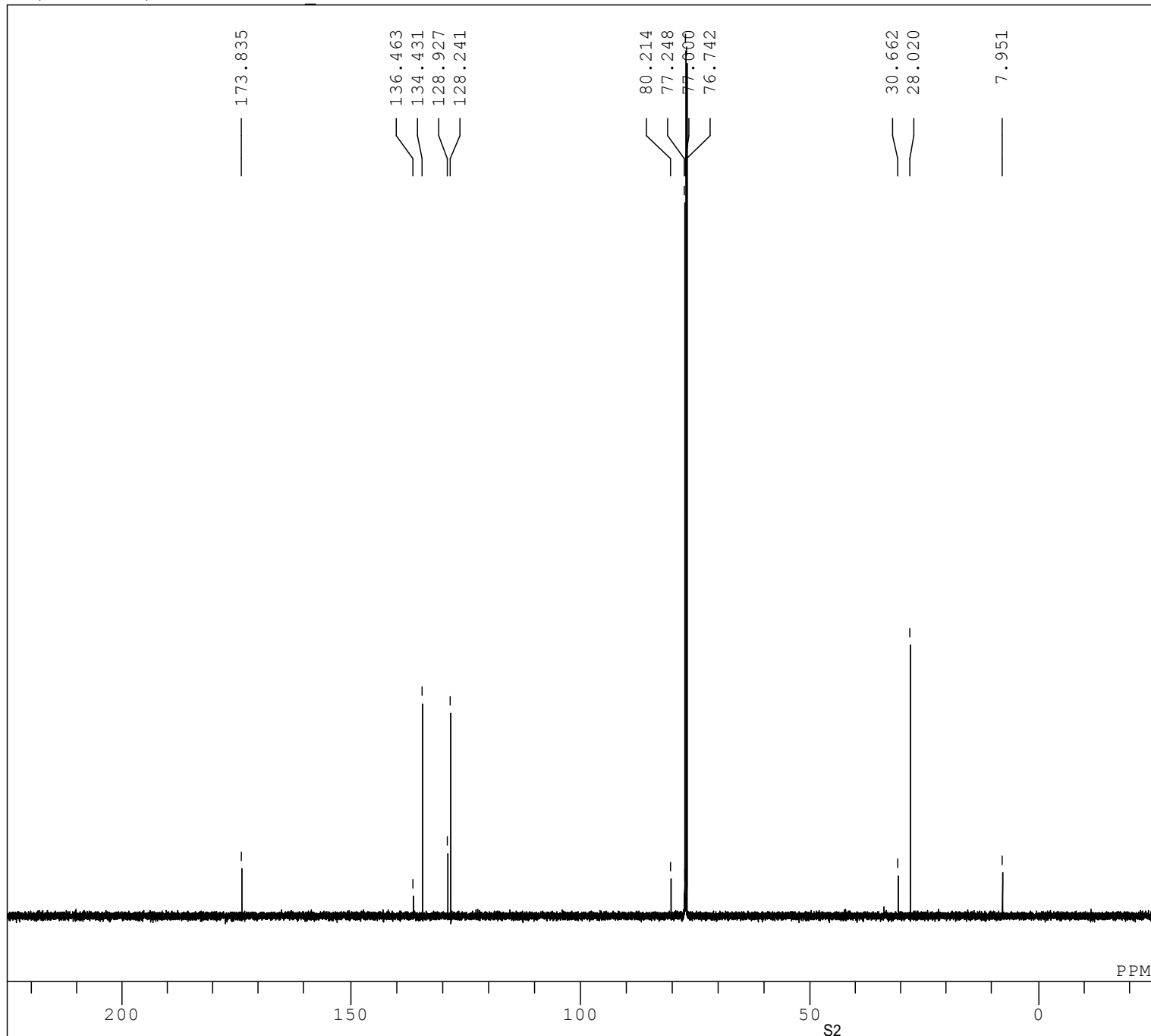
[1] ^1H , ^{13}C NMR Spectra	S001
[2] X-ray Crystallographic Analysis Information	S011



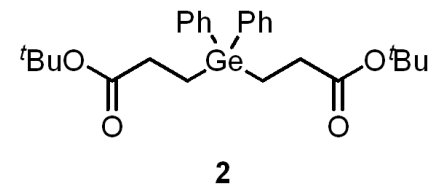
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 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
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 PD 5.0000 sec
 PW1 6.85 usec
 IRNUC 1H
 CTEMP 15.7 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.10 Hz
 RGAIN 44



Z:\NMR data\td-01-077-063_Carbon-1-1.als



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EXMOD carbon.jxp
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OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
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IRNUC 1H
CTEMP 15.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 60

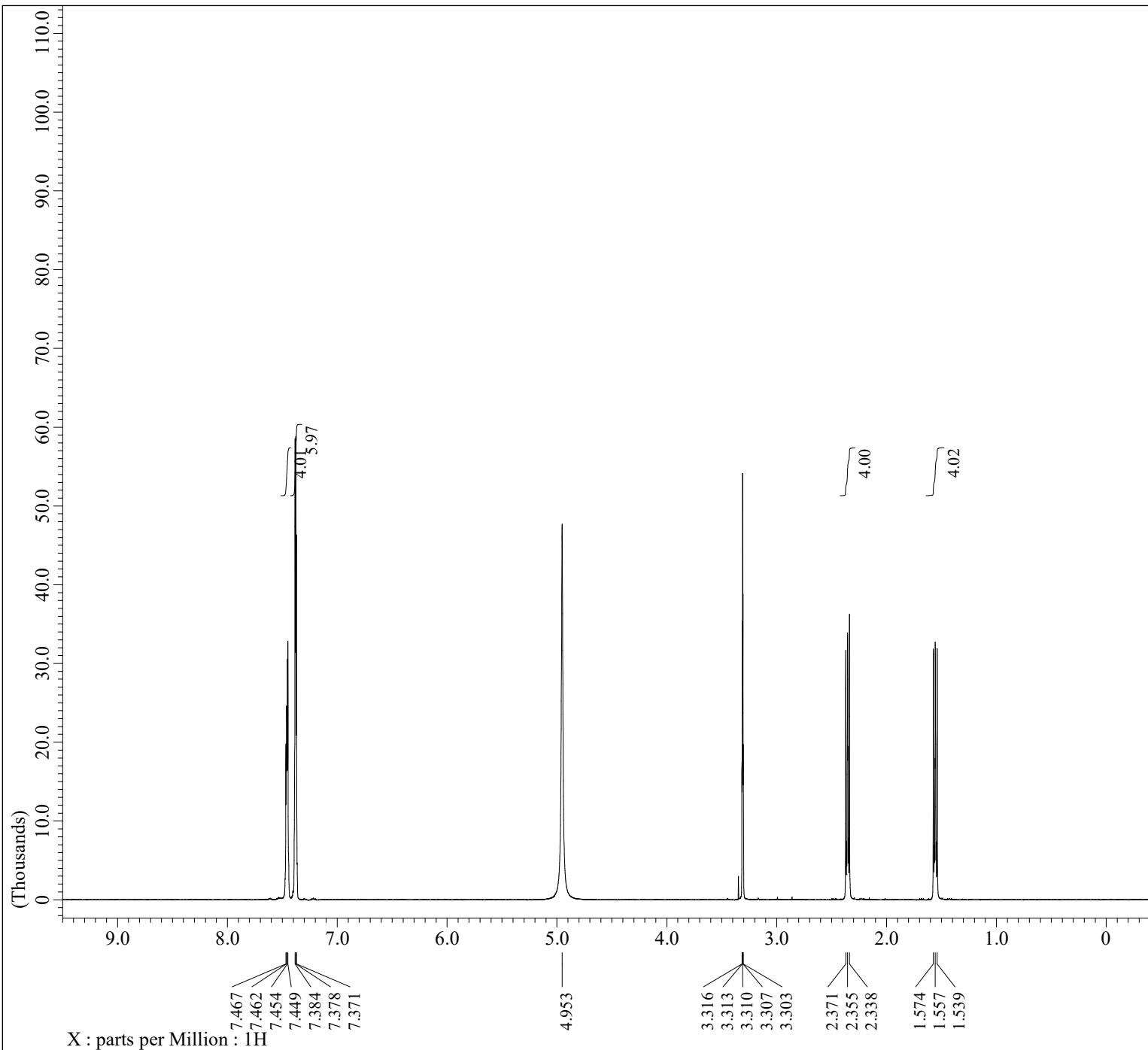
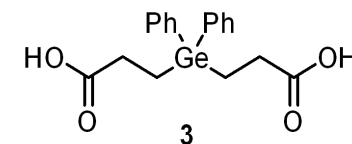


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 Solvent = CD3OD
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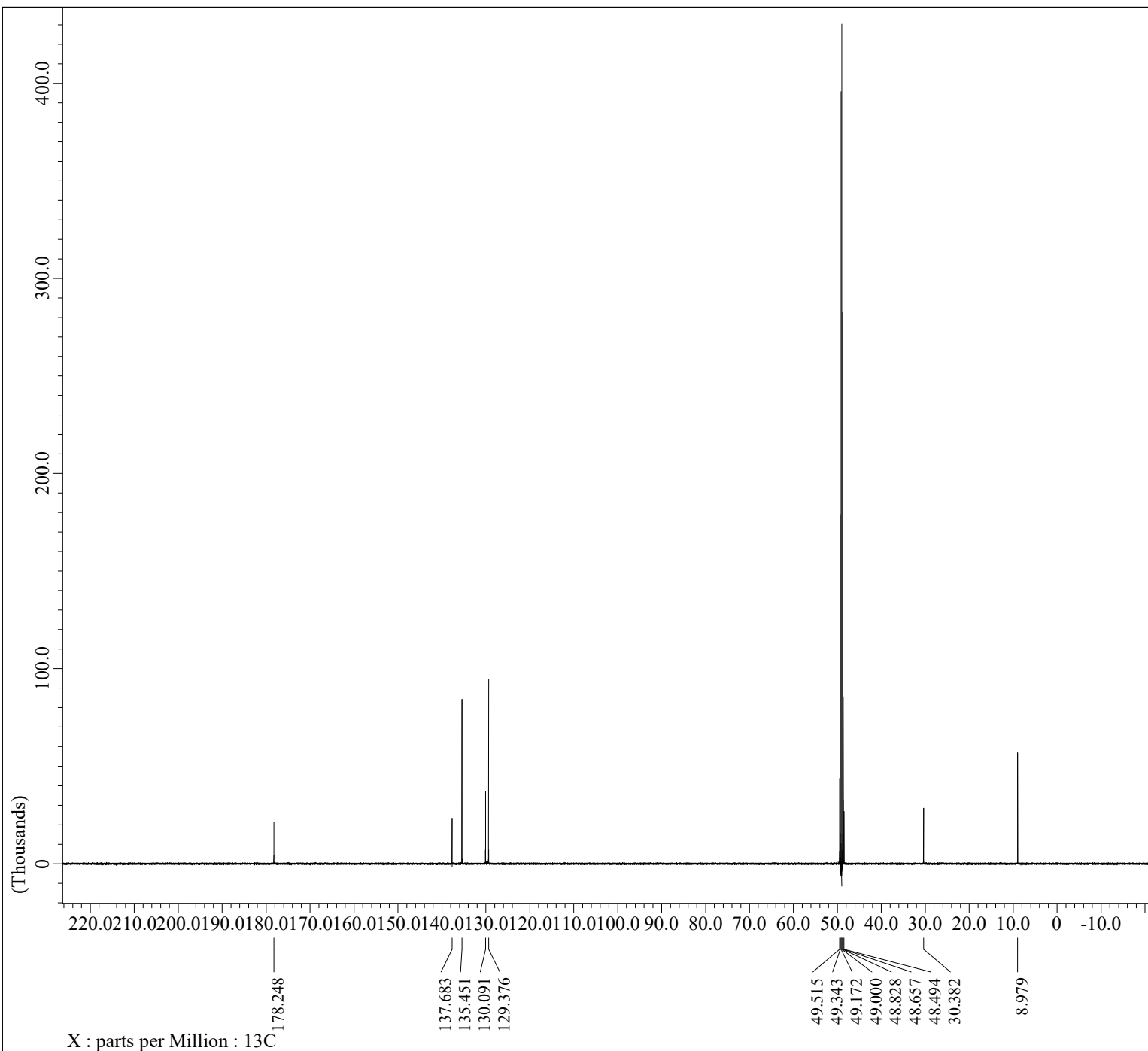
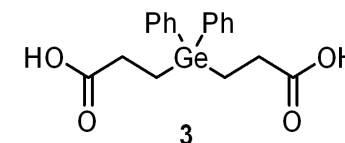


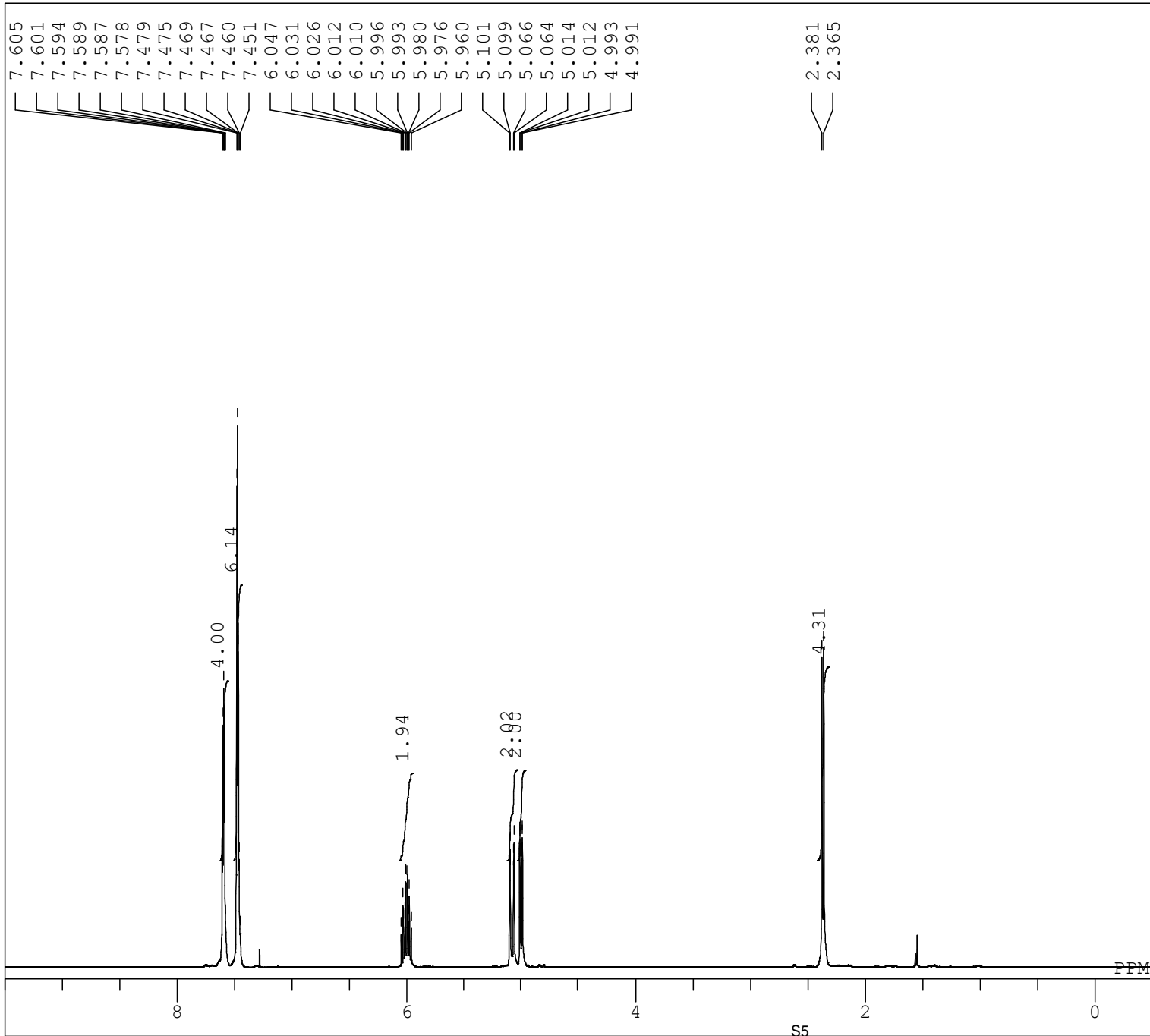
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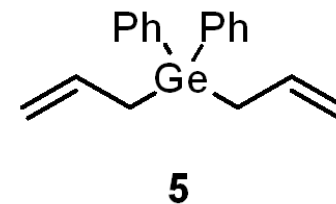
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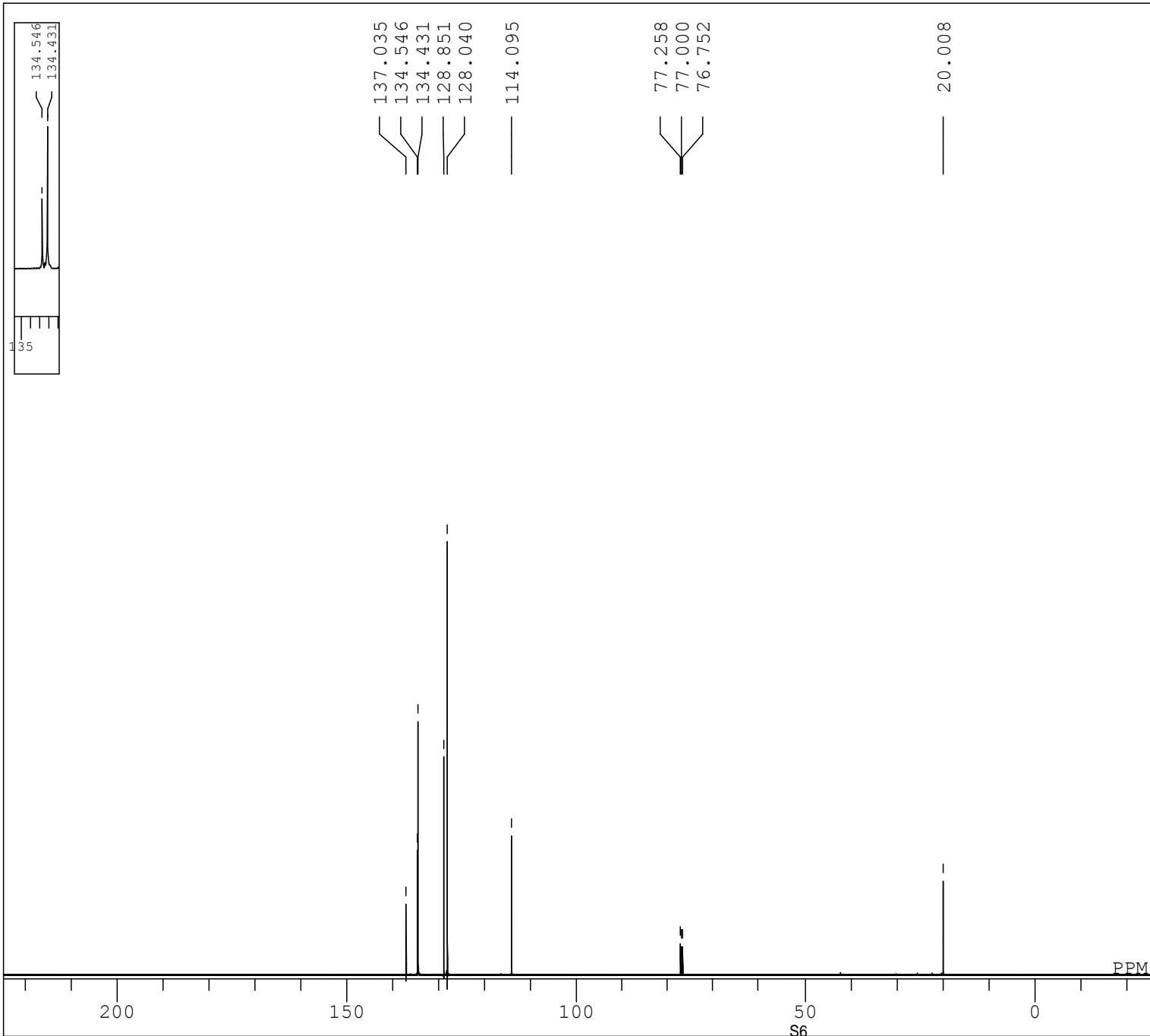
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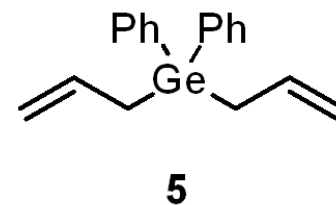


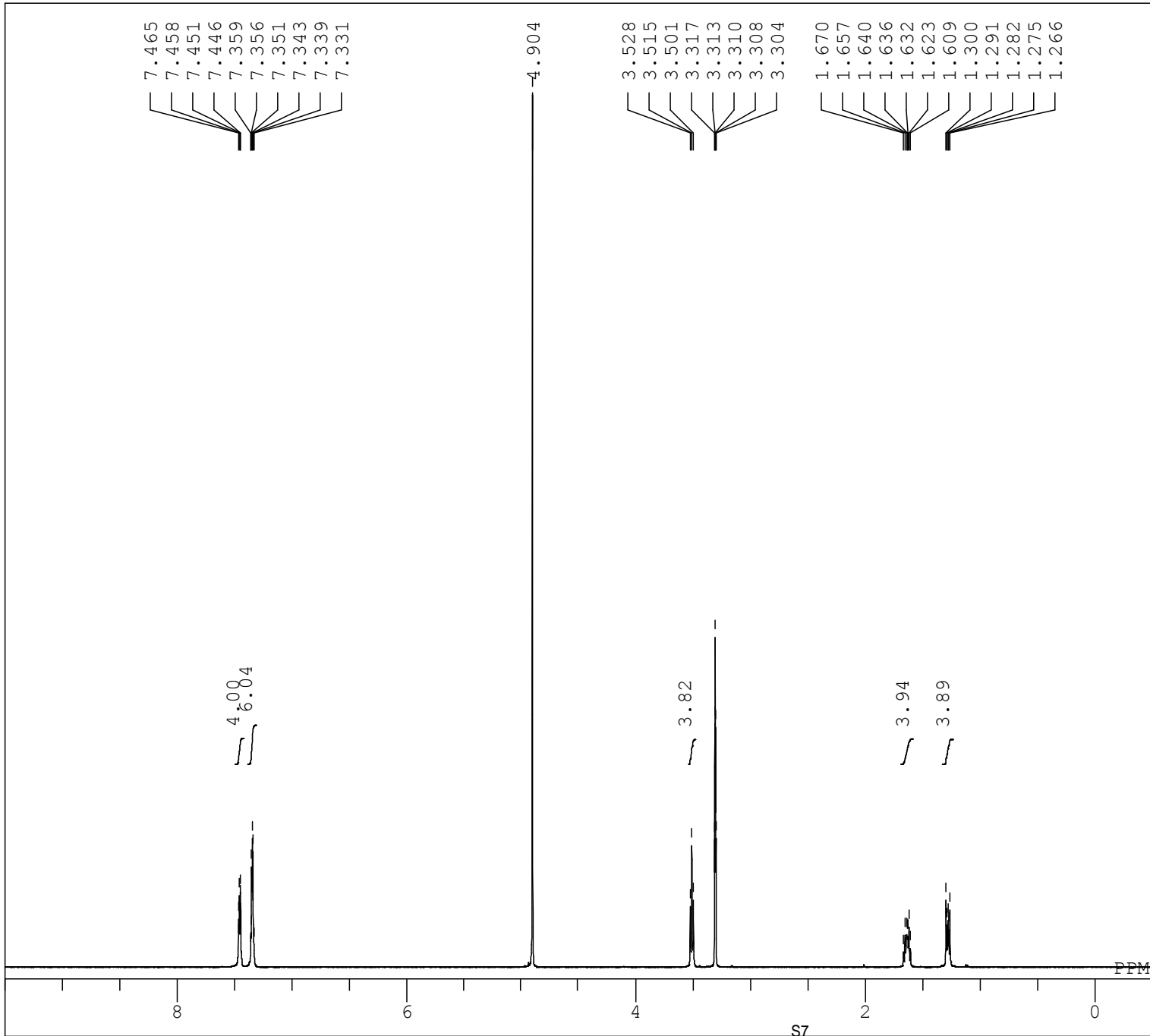
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 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 6.85 usec
 IRNUC 1H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 2.05 ppm
 BF 0.10 Hz
 RGAIN 20



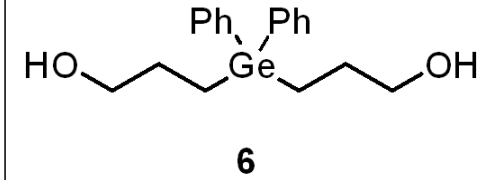


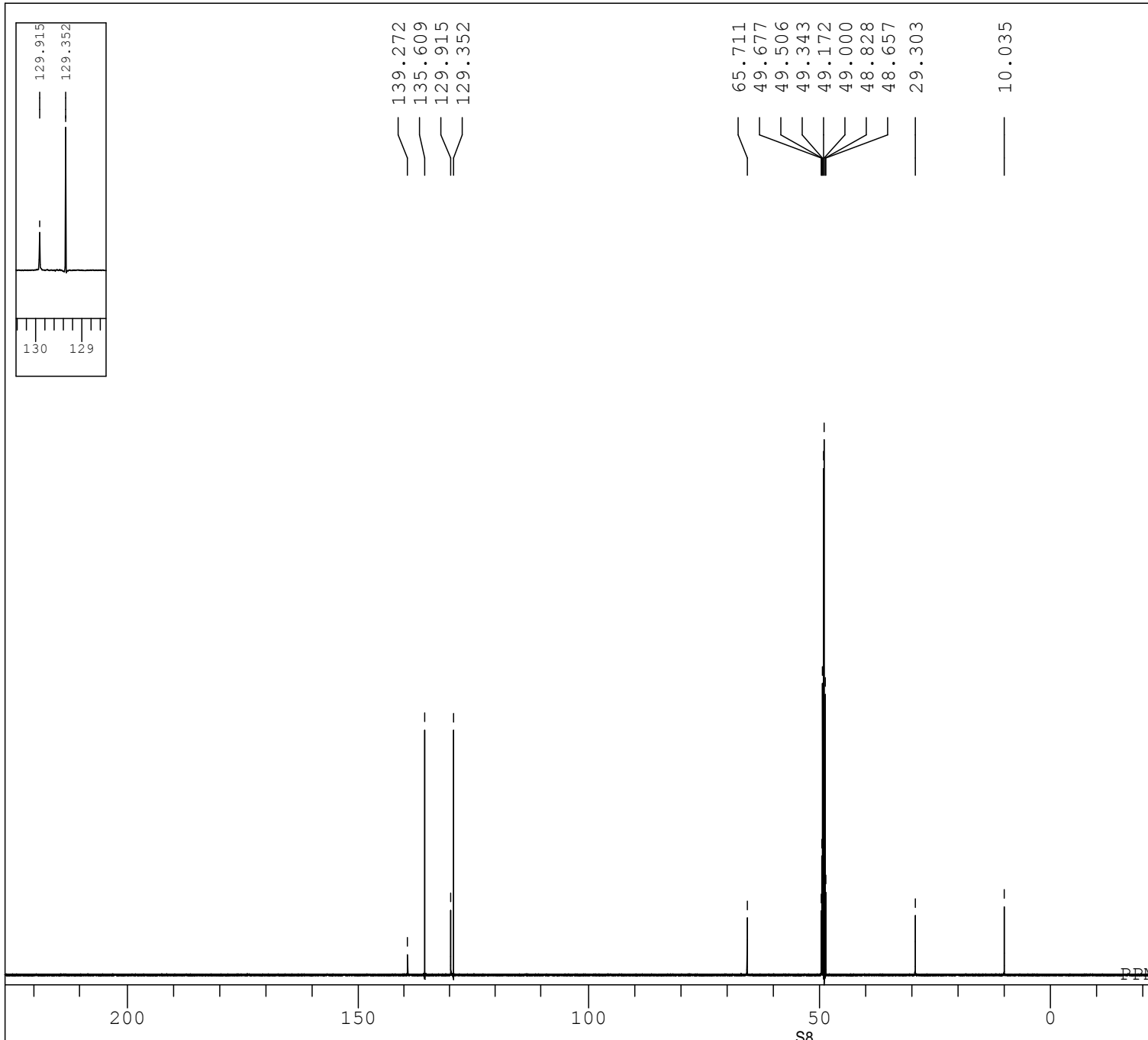
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 POINT 26214
 FREQU 31446.54 Hz
 SCANS 1024
 ACQTM 0.8336 sec
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 IRNUC 1H
 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.10 Hz
 RGAIN 60



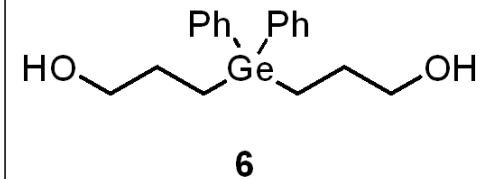


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 FREQU 7507.51 Hz
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 ACQTM 1.7459 sec
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 PW1 6.85 usec
 IRNUC 1H
 CTEMP 18.5 c
 SLVNT CD3OD
 EXREF 3.31 ppm
 BF 0.10 Hz
 RGAIN 44

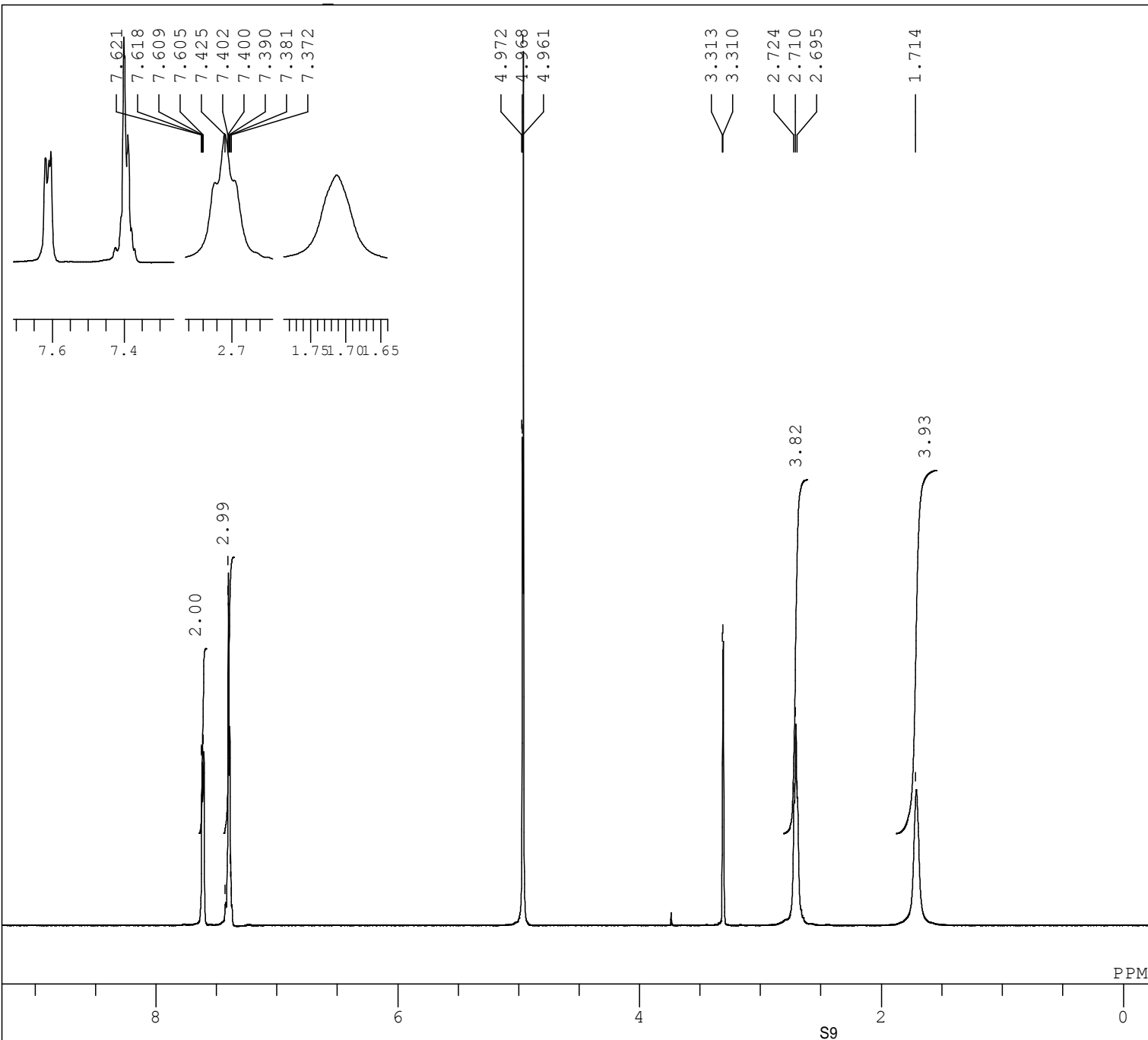




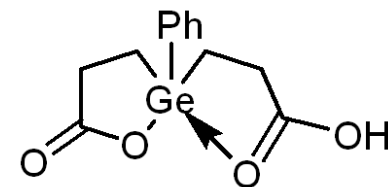
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 POINT 26214
 FREQU 31446.54 Hz
 SCANS 1024
 ACQTM 0.8336 sec
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 PW1 3.12 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.10 Hz
 RGAIN 58

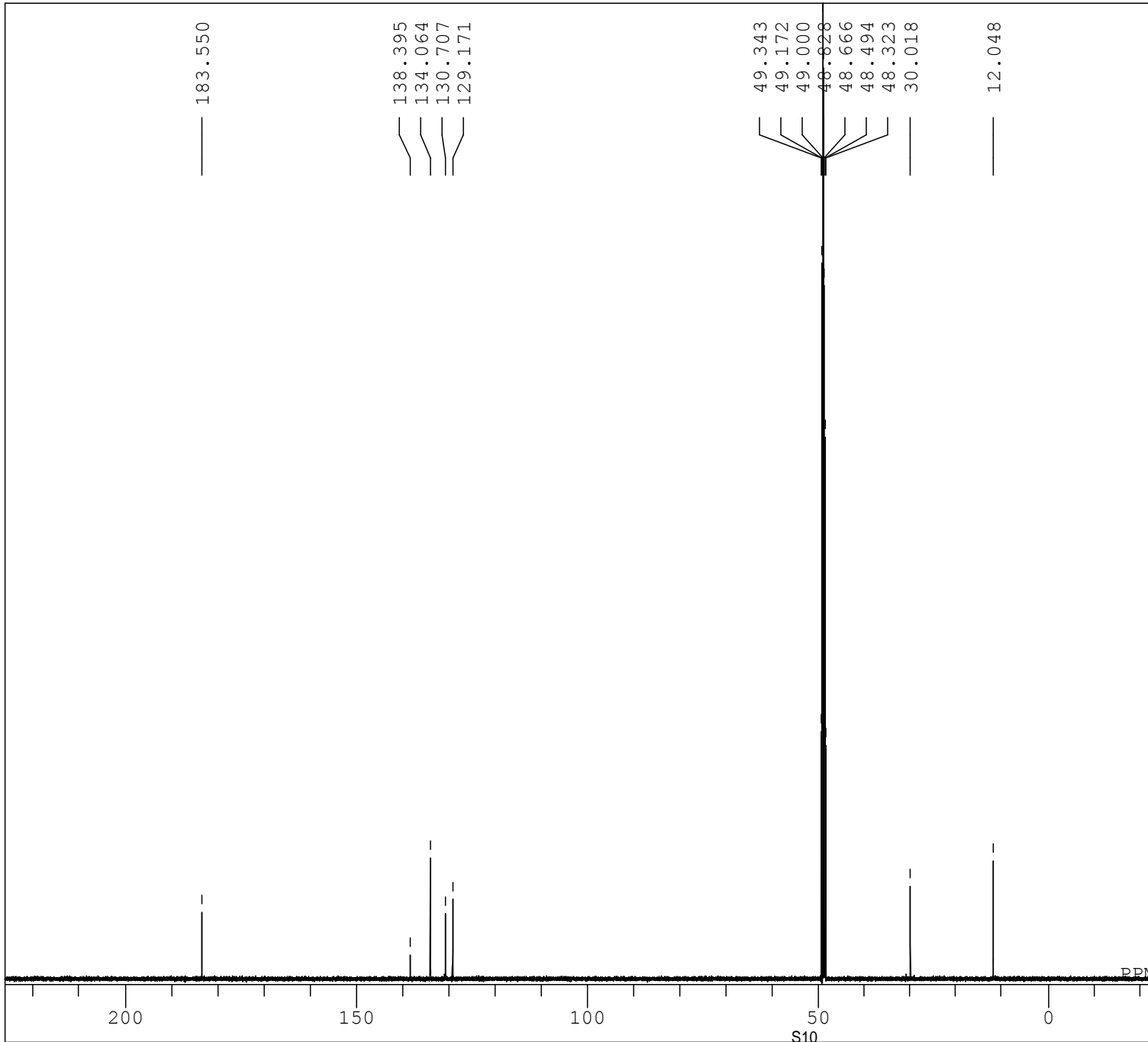


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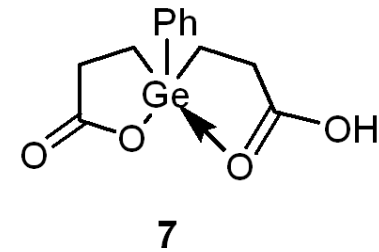


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RGAIN 40





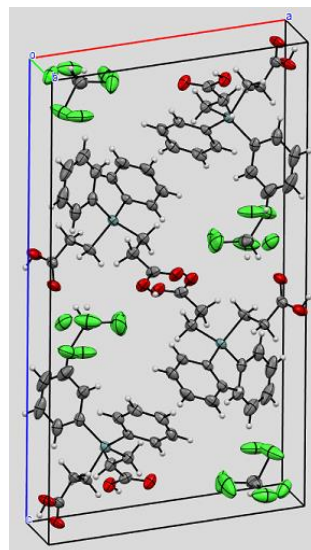
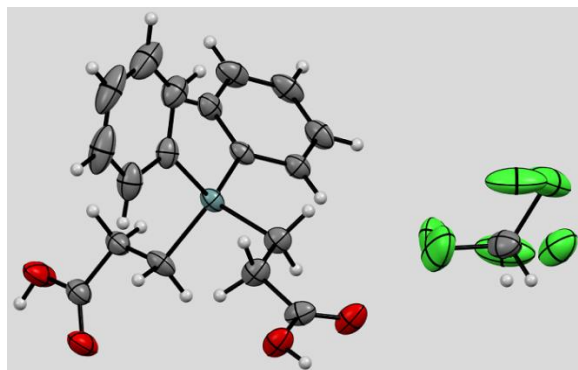
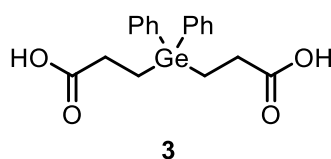
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 RGAIN 60



X-ray Crystallographic Analysis Information

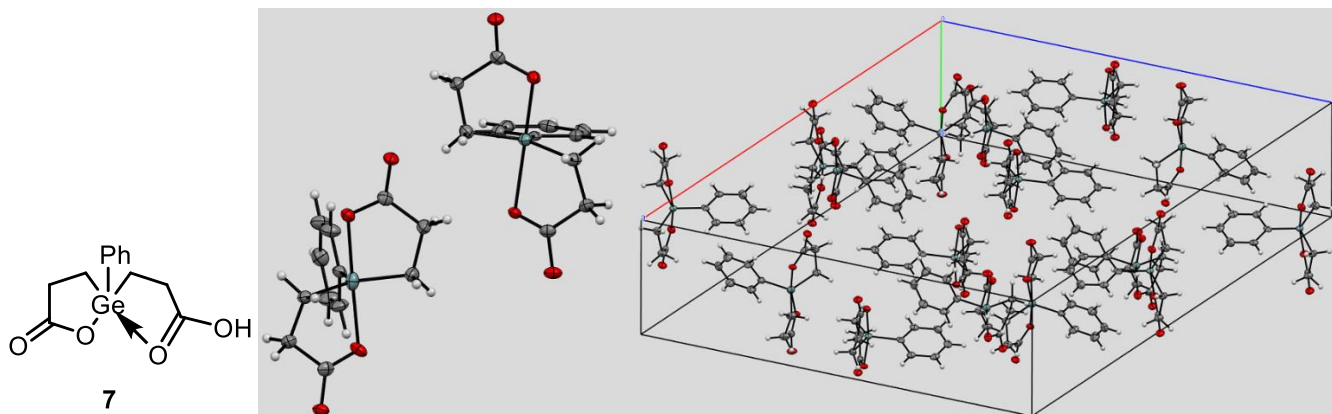
(ORTEP thermal ellipsoids at 50% probability)

(1) Dicarboxylic acid **3** with chloroform disordered (CCDC No. 1993114)



Empirical formula	$C_{19}H_{21}Cl_3GeO_4$
Formula weight	492.32
Temperature	-150 ± 1 °C
Wavelength	MoK α ($\lambda = 0.71075$ Å)
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
Unit cell dimensions	$a = 12.8984(3)$ Å $b = 7.48709(14)$ Å $\beta = 97.788(7)$ ° $c = 23.2436(4)$ Å
Volume	$V = 2223.96(8)$ Å ³
Z	4
Density (calculated)	1.470 g/cm ³
$2\theta_{max}$	55.0 °
Absorption coefficient μ for Mo-K α	17.569 cm ⁻¹
$F(000)$	1000.00
Crystal size	0.210 x 0.200 x 0.100 mm
No. of reflection collected	Total: 36289 Unique: 5101 ($R_{int} = 0.0406$)
Transmission factor	min: 0.666, max: 0.839
Refinement method	Full-matrix least-squares on F^2
Goodness-of-fit on F^2	1.062
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0427$
R indices (all data)	$R_1 = 0.0478$ $wR_2 = 0.1117$
Largest diff. peak and hole	-0.55 and 1.11 e \cdot Å ⁻³

(2) Pentacoordinate germane 7 (CCDC No. 1993115)



Empirical formula	$C_{12}H_{13}GeO_4$
Formula weight	293.82
Temperature	$-150 \pm 1^\circ C$
Wavelength	MoK α ($\lambda = 0.71075 \text{ \AA}$)
Crystal system	monoclinic
Space group	C2/c (#15)
Unit cell dimensions	$a = 24.7253(5) \text{ \AA}$ $b = 9.34850(18) \text{ \AA}$ $\beta = 119.183(8)^\circ$ $c = 24.2034(5) \text{ \AA}$
Volume	$V = 4884.4(4) \text{ \AA}^3$
Z	4
Density (calculated)	1.598 g/cm^3
$2\theta_{\max}$	55.0°
Absorption coefficient μ for Mo-K α	25.066 cm^{-1}
$F(000)$	2384.00
Crystal size	0.240 x 0.050 x 0.020 mm
No. of reflection collected	Total: 39631 Unique: 5598 ($R_{\text{int}} = 0.0569$)
Transmission factor	min: 0.638, max: 0.951
Refinement method	Full-matrix least-squares on F^2
Goodness-of-fit on F^2	1.285
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0635$
R indices (all data)	$R_1 = 0.0689$ $wR_2 = 0.1497$
Largest diff. peak and hole	-0.93 and $0.95 \text{ e} \cdot \text{\AA}^{-3}$