

**STEREOSELECTIVE APPROACH TOWARD OPHIODILACTONES BASED ON  
AN INTRAMOLECULAR [2 + 2] CYCLOADDITION REACTION**

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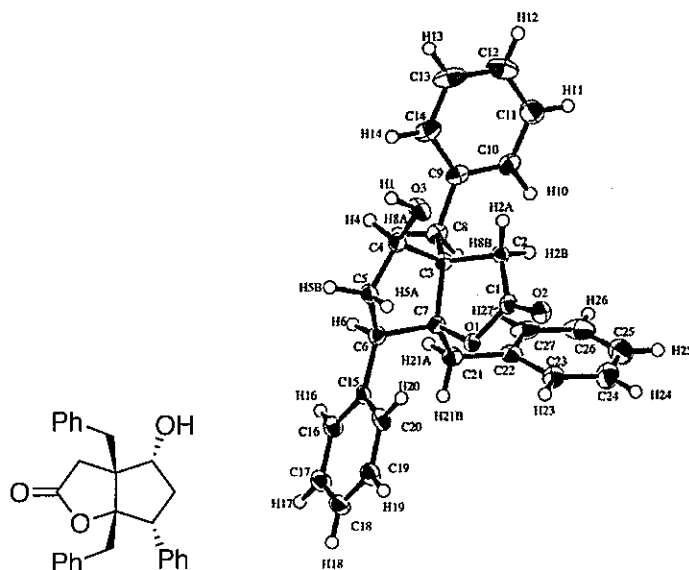
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**Supporting Information**

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## X-ray Crystallographic Data of 18 $\alpha$

The crystallographic data (CCDC 1002959) can be obtained free of charge from the Cambridge Crystallographic Data centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).



A colorless block crystal of  $C_{27}H_{26}O_3$  having approximate dimensions of 0.180 x 0.160 x 0.100 mm was mounted on a glass fiber. All measurements were made on a Rigaku Saturn724 diffractometer using multi-layer mirror monochromated Mo-K $\alpha$  radiation. The crystal-to-detector distance was 44.98 mm.

### A. Crystal Data

Empirical Formula	$C_{27}H_{26}O_3$
Formula Weight	398.50
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.180 x 0.160 x 0.100 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	$a = 10.769(6) \text{ \AA}$ $b = 9.920(6) \text{ \AA}$ $c = 11.266(7) \text{ \AA}$ $\beta = 115.494(6)^\circ$ $V = 1086.4(10) \text{ \AA}^3$
Space Group	$P2_1$ (#4)

Z value	2
$D_{\text{calc}}$	1.218 g/cm <sup>3</sup>
$F_{000}$	424.00
m(MoKa)	0.780 cm <sup>-1</sup>

## B. Intensity Measurements

Diffractometer	Saturn724
Radiation	MoKa ( $\lambda = 0.71075 \text{ \AA}$ ) multi-layer mirror monochromated
Voltage, Current	50kV, 24mA
Temperature	-179.8 °C
Detector Aperture	70 x 70 mm
Data Images	720 exposures
$\omega$ oscillation Range	-110.0 - 70.0°
Exposure Rate	16.0 sec./°
Detector Swing Angle	-20.05°
$\omega$ oscillation Range	-110.0 - 70.0°
Exposure Rate	16.0 sec./°
Detector Swing Angle	-20.05°
Detector Position	44.98 mm
Pixel Size	0.141 mm
$2\theta_{\text{max}}$	55.0°
No. of Reflections Measured	Total: 8974 Unique: 4804 ( $R_{\text{int}} = 0.0322$ ) Friedel pairs: 2180
Corrections	Lorentz-polarization Absorption (trans. factors: 0.835 - 0.992)

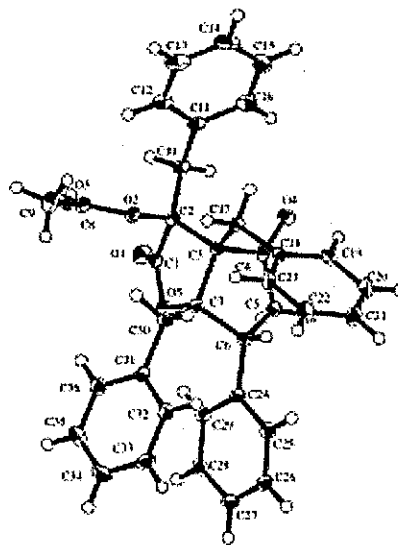
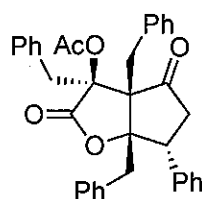
## C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on $F^2$

Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [ s^2(F_o^2) + (0.0437 \cdot P)^2 + 0.0000 \cdot P ]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2) / 3$
$2\theta_{\text{max}}$ cutoff	55.0°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4804
No. Variables	276
Reflection/Parameter Ratio	17.41
Residuals: R1 ( $I > 2.00\sigma(I)$ )	0.0455
Residuals: R (All reflections)	0.0654
Residuals: wR2 (All reflections)	0.0925
Goodness of Fit Indicator	0.923
Flack Parameter (Friedel pairs = 2180)	0.0(11)
Max Shift/Error in Final Cycle	1.099
Maximum peak in Final Diff. Map	0.16 e-/Å <sup>3</sup>
Minimum peak in Final Diff. Map	-0.18 e-/Å <sup>3</sup>

### X-ray Crystallographic Data of the Acetate of 3

The crystallographic data (CCDC 1002961) can be obtained free of charge from the Cambridge Crystallographic Data centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).



A colorless block crystal of  $C_{36}H_{32}O_5$  having approximate dimensions of 0.160 x 0.130 x 0.090 mm was mounted on a glass fiber. All measurements were made on a Rigaku Saturn724 diffractometer using multi-layer mirror monochromated Mo-K $\alpha$  radiation. The crystal-to-detector distance was 45.00 mm.

#### A. Crystal Data

Empirical Formula	$C_{36}H_{32}O_5$
Formula Weight	544.65
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.160 x 0.130 x 0.090 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	$a = 7.6947(13) \text{ \AA}$ $b = 16.278(3) \text{ \AA}$ $c = 11.450(2) \text{ \AA}$ $\beta = 101.762(3)^\circ$ $V = 1404.0(5) \text{ \AA}^3$
Space Group	$P2_1$ (#4)

Z value	2
$D_{\text{calc}}$	1.288 g/cm <sup>3</sup>
$F_{000}$	576.00
m(MoKa)	0.848 cm <sup>-1</sup>

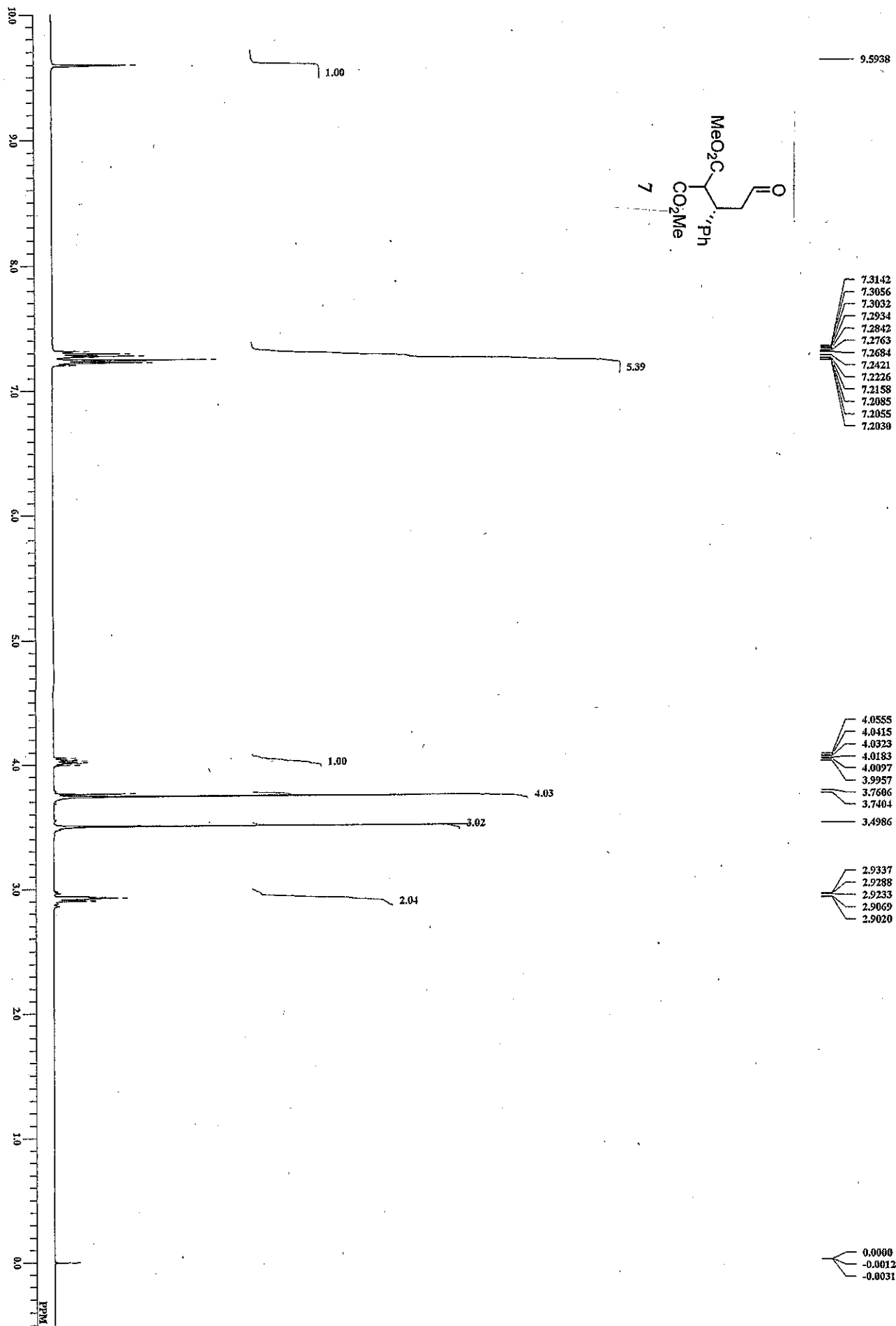
## B. Intensity Measurements

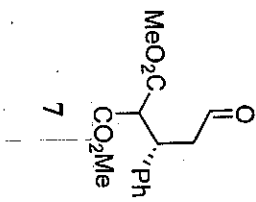
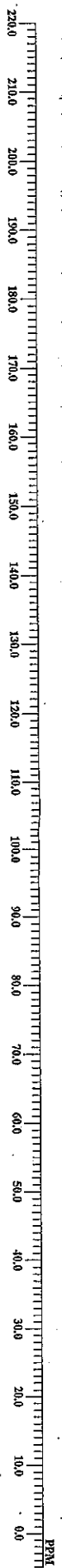
Diffractometer	Saturn724
Radiation	MoKa ( $\lambda = 0.71075 \text{ \AA}$ ) multi-layer mirror monochromated
Voltage, Current	50kV, 24mA
Temperature	-179.8 °C
Detector Aperture	70 x 70 mm
Data Images	720 exposures
$\omega$ oscillation Range	-110.0 - 70.0°
Exposure Rate	16.0 sec./°
Detector Swing Angle	-20.07°
$\omega$ oscillation Range	-110.0 - 70.0°
Exposure Rate	16.0 sec./°
Detector Swing Angle	-20.07°
Detector Position	45.00 mm
Pixel Size	0.141 mm
$2\theta_{\text{max}}$	54.9°
No. of Reflections Measured	Total: 11772 Unique: 6198 ( $R_{\text{int}} = 0.0236$ ) Friedel pairs: 2884
Corrections	Lorentz-polarization Absorption (trans. factors: 0.928 - 0.992)

## C. Structure Solution and Refinement

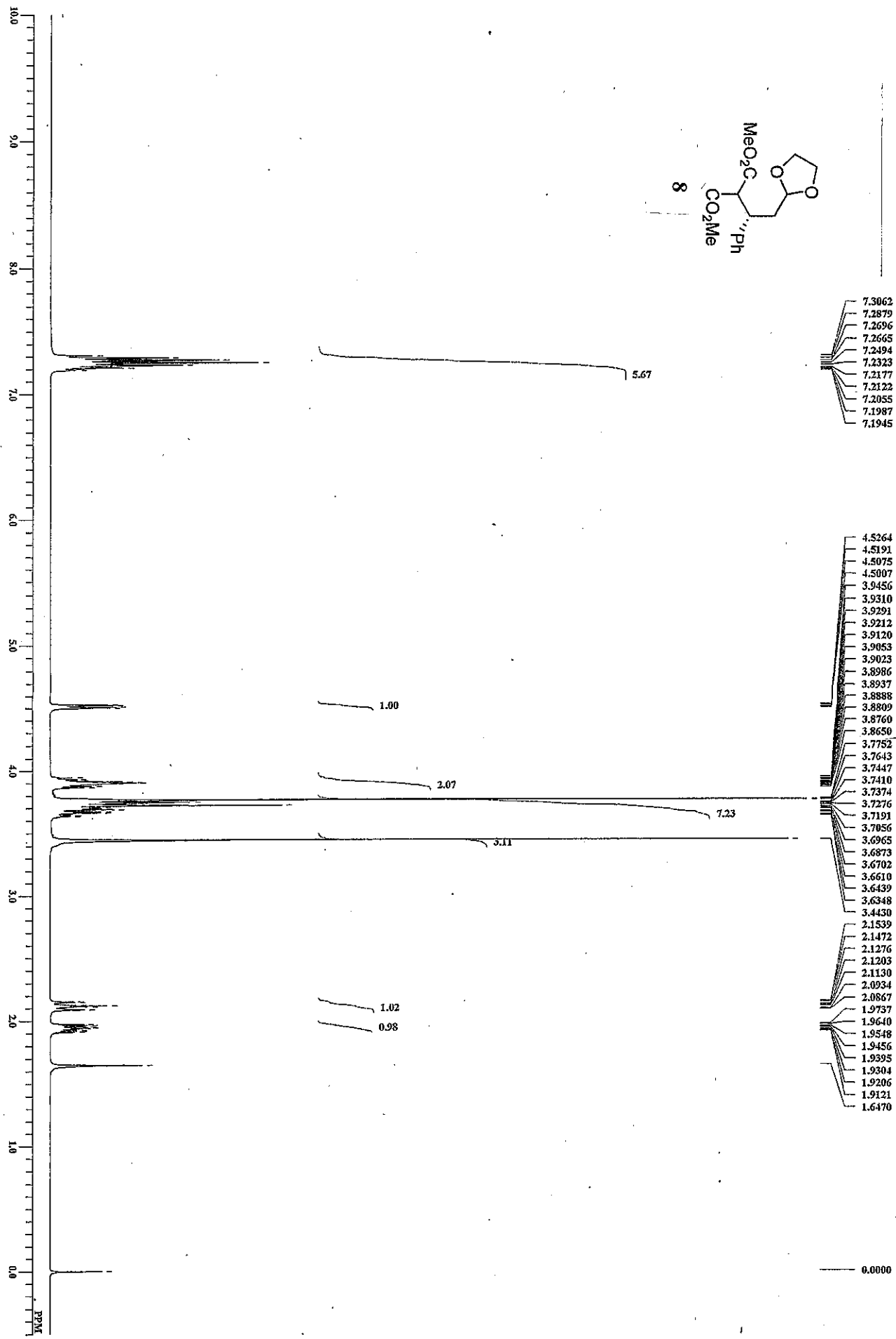
Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on $F^2$

Function Minimized	$S w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [ s^2(F_o^2) + (0.0397 \cdot P)^2 + 0.0000 \cdot P ]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2) / 3$
$2q_{\text{max}}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	6198
No. Variables	371
Reflection/Parameter Ratio	16.71
Residuals: R1 ( $I > 2.00s(I)$ )	0.0357
Residuals: R (All reflections)	0.0462
Residuals: wR2 (All reflections)	0.0761
Goodness of Fit Indicator	0.998
Flack Parameter (Friedel pairs = 2884)	0.4(7)
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.18 e-/Å <sup>3</sup>
Minimum peak in Final Diff. Map	-0.18 e-/Å <sup>3</sup>

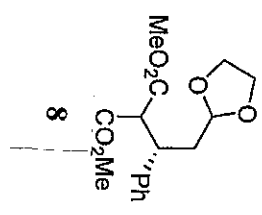
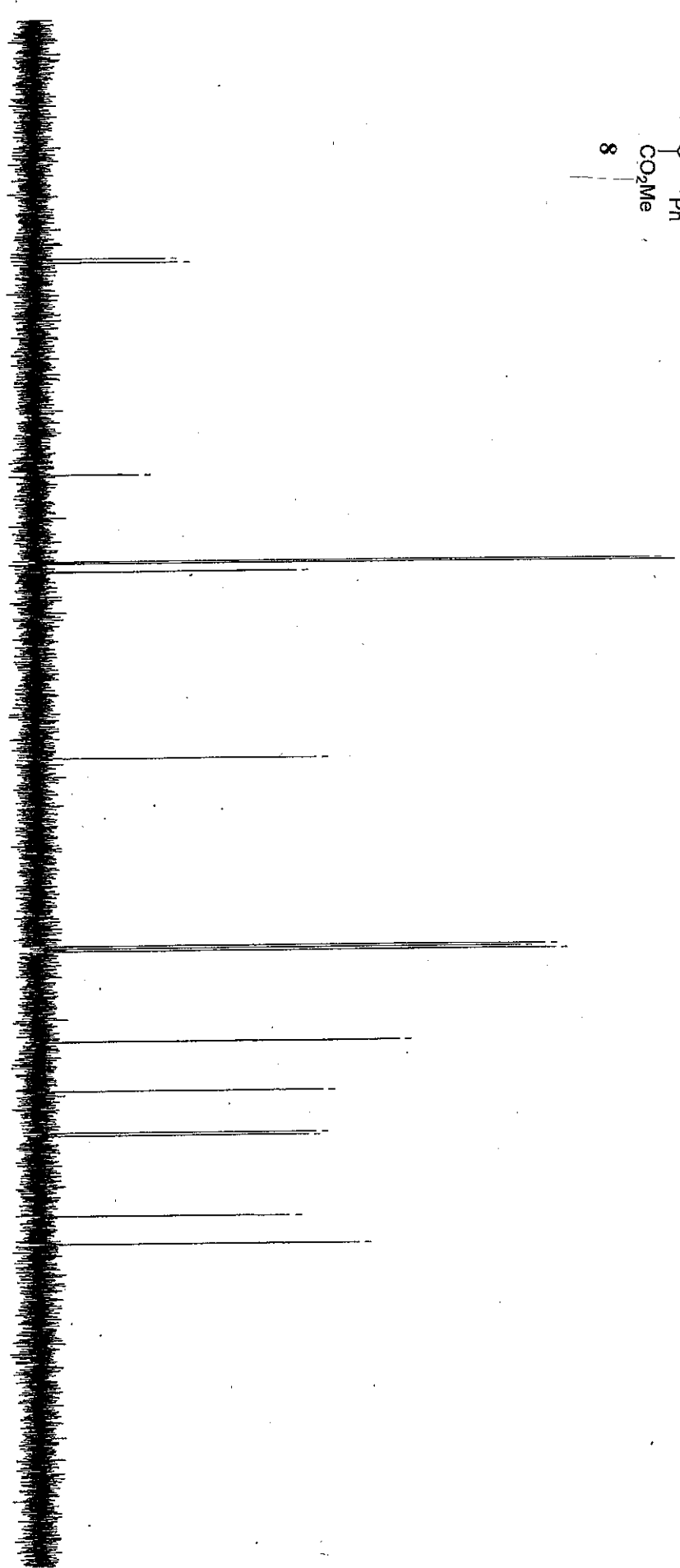




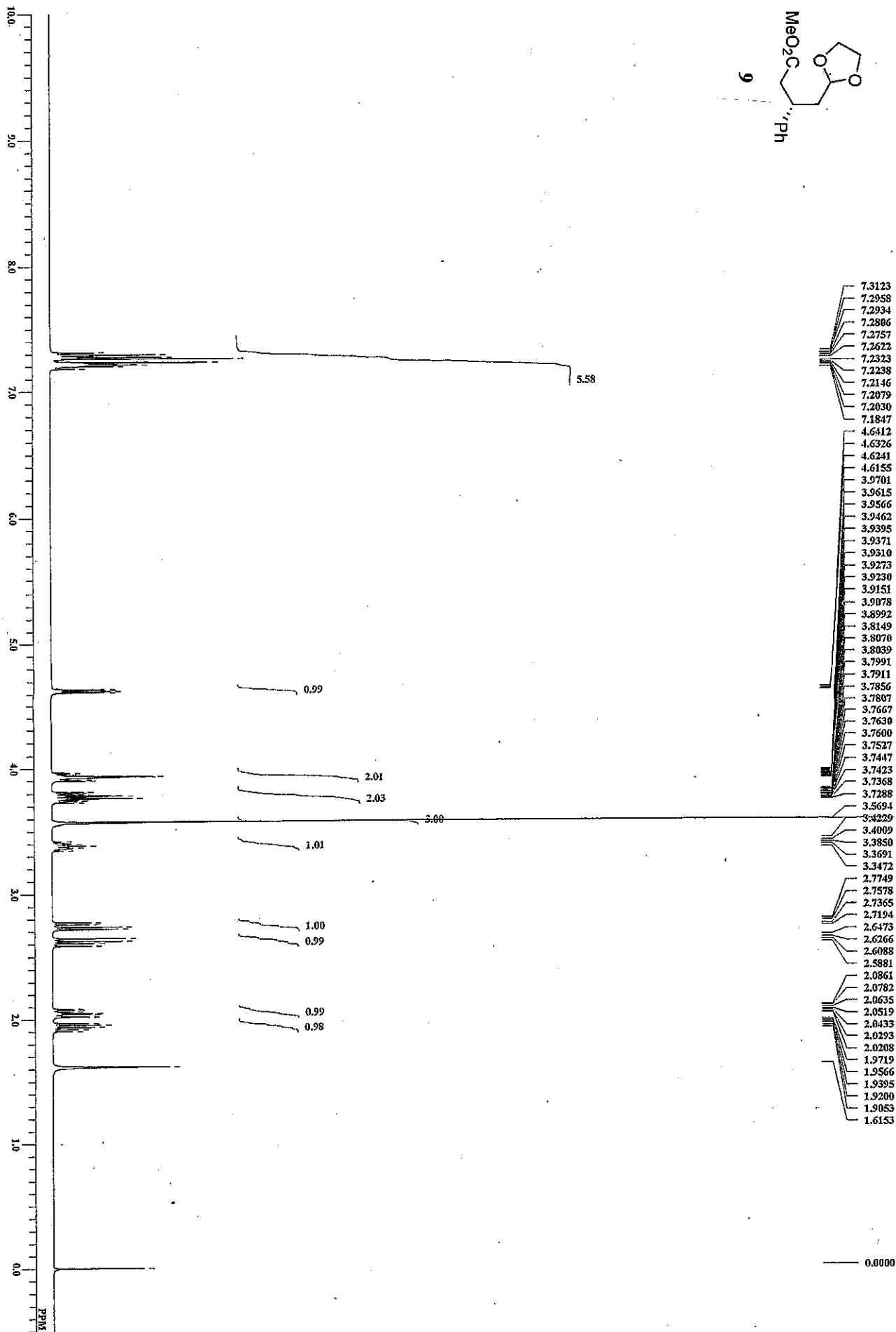
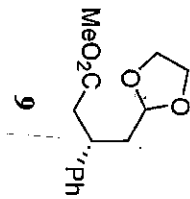
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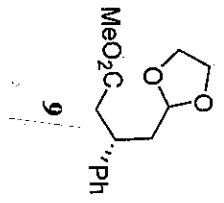
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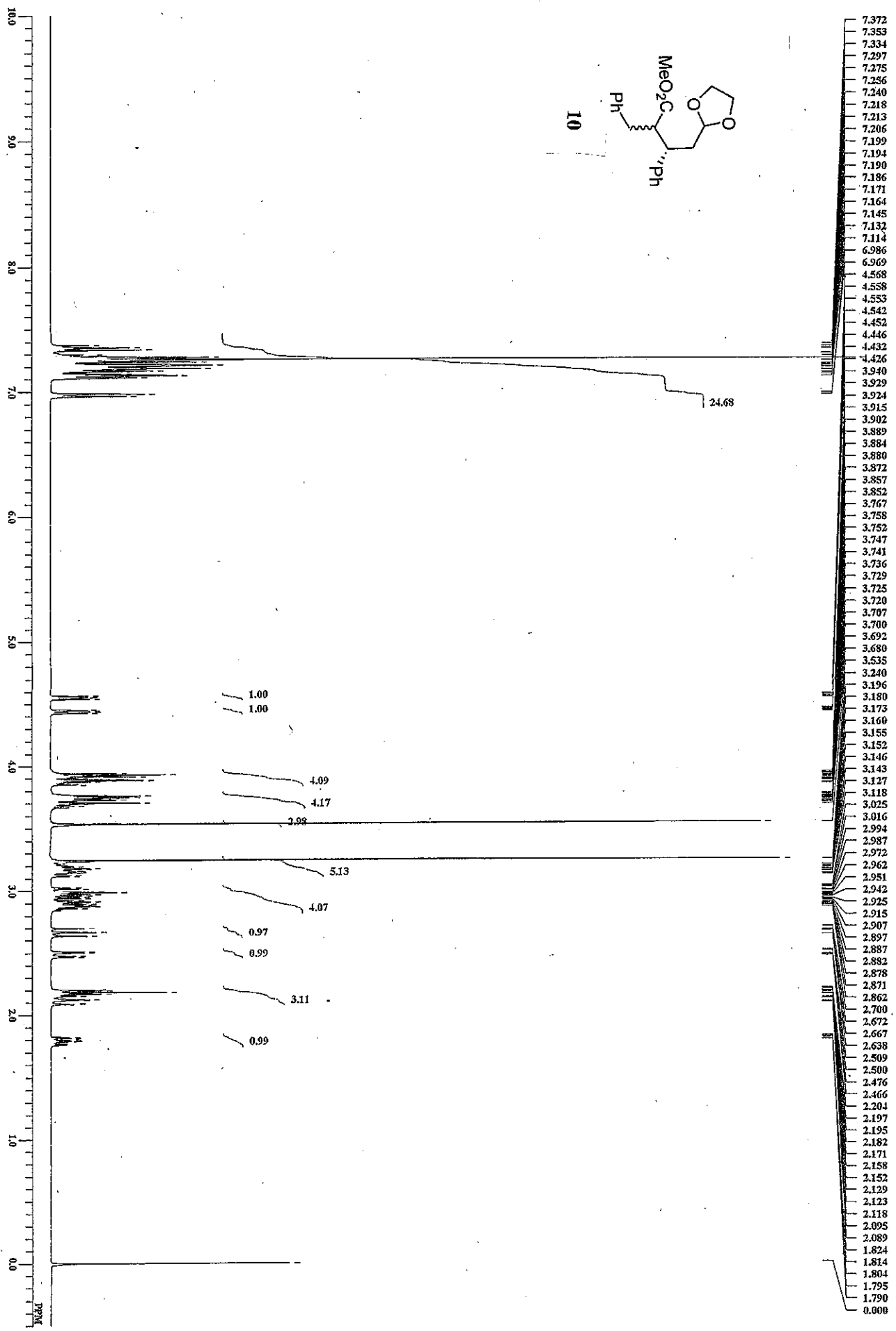
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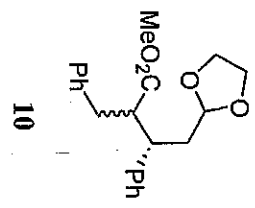
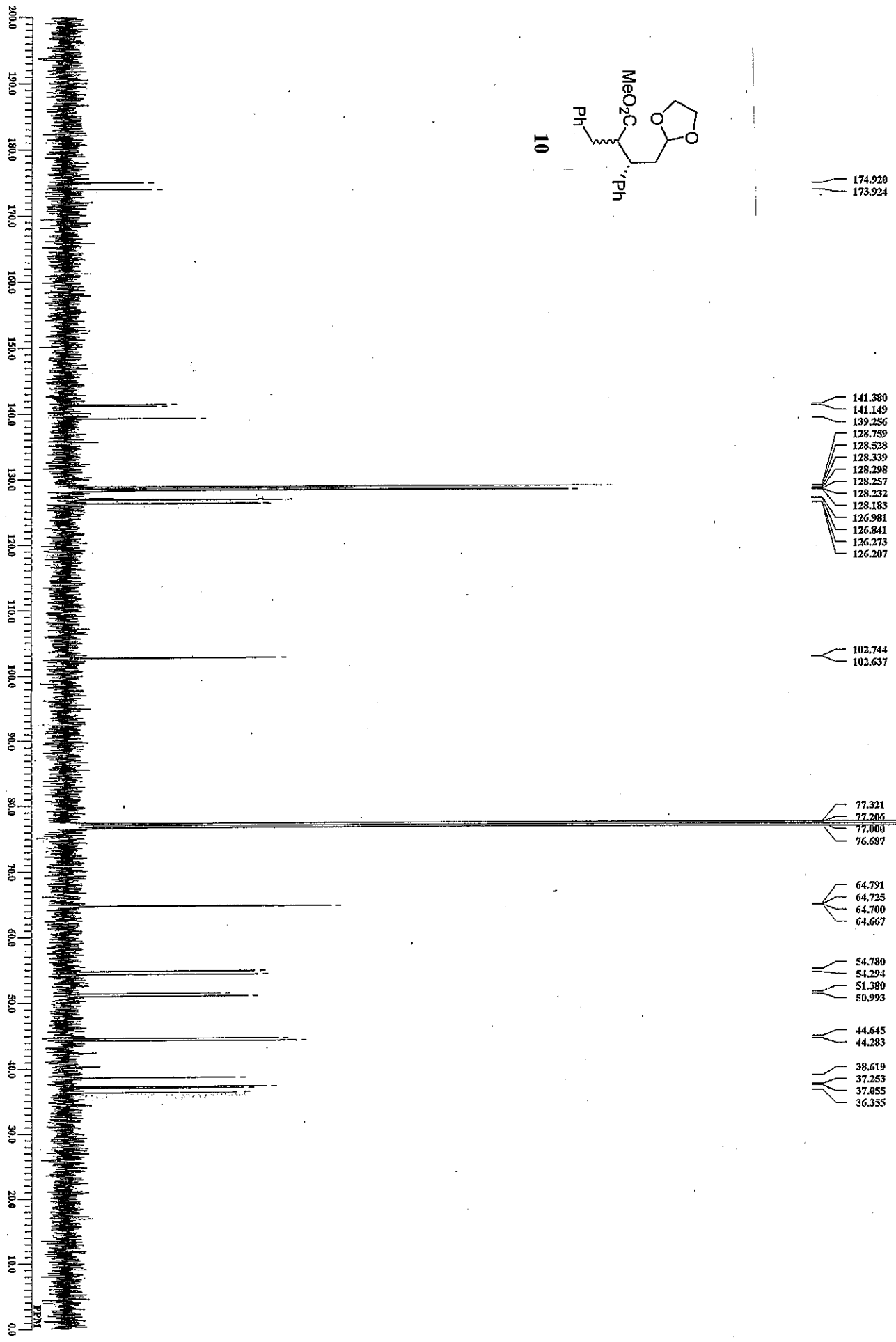


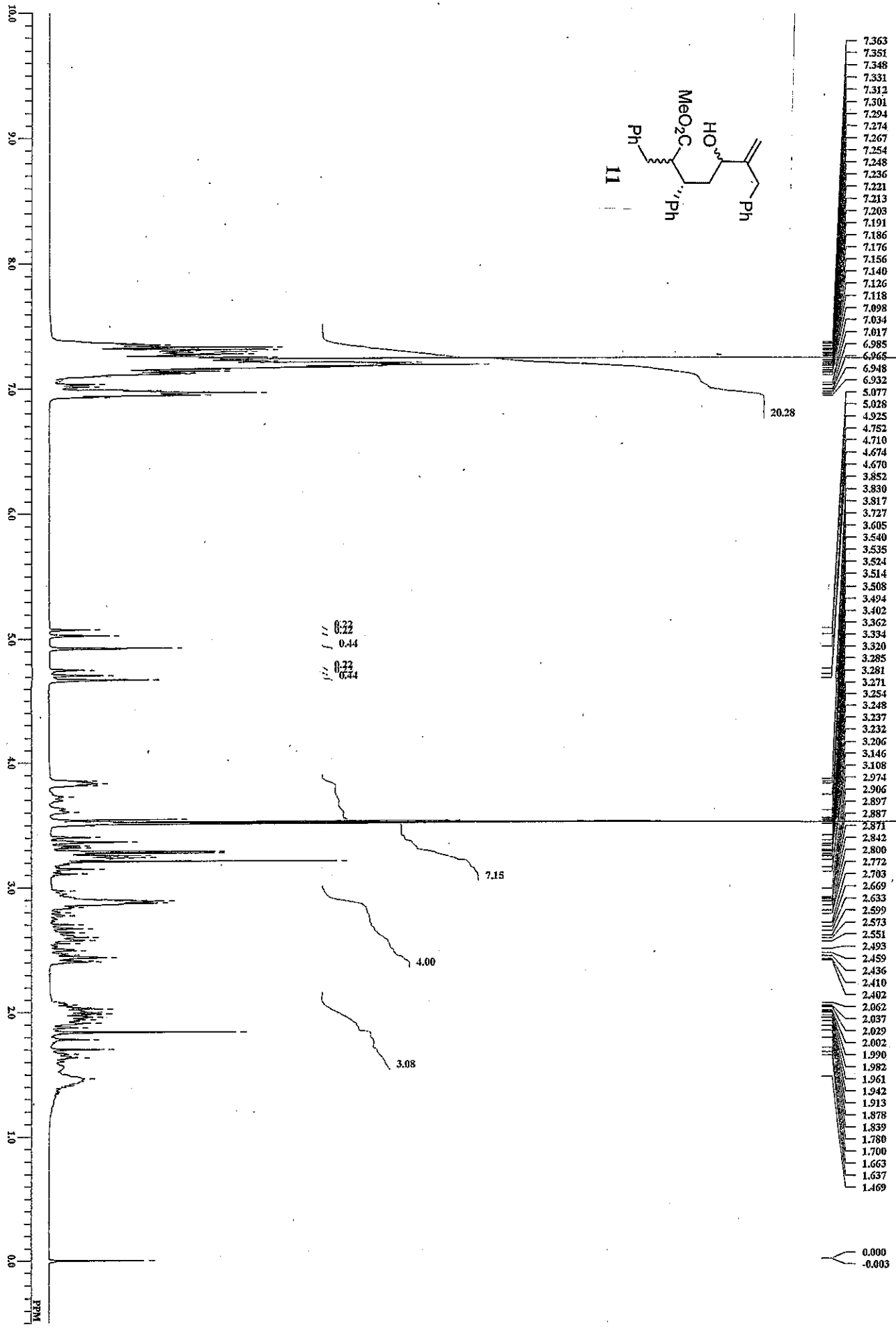
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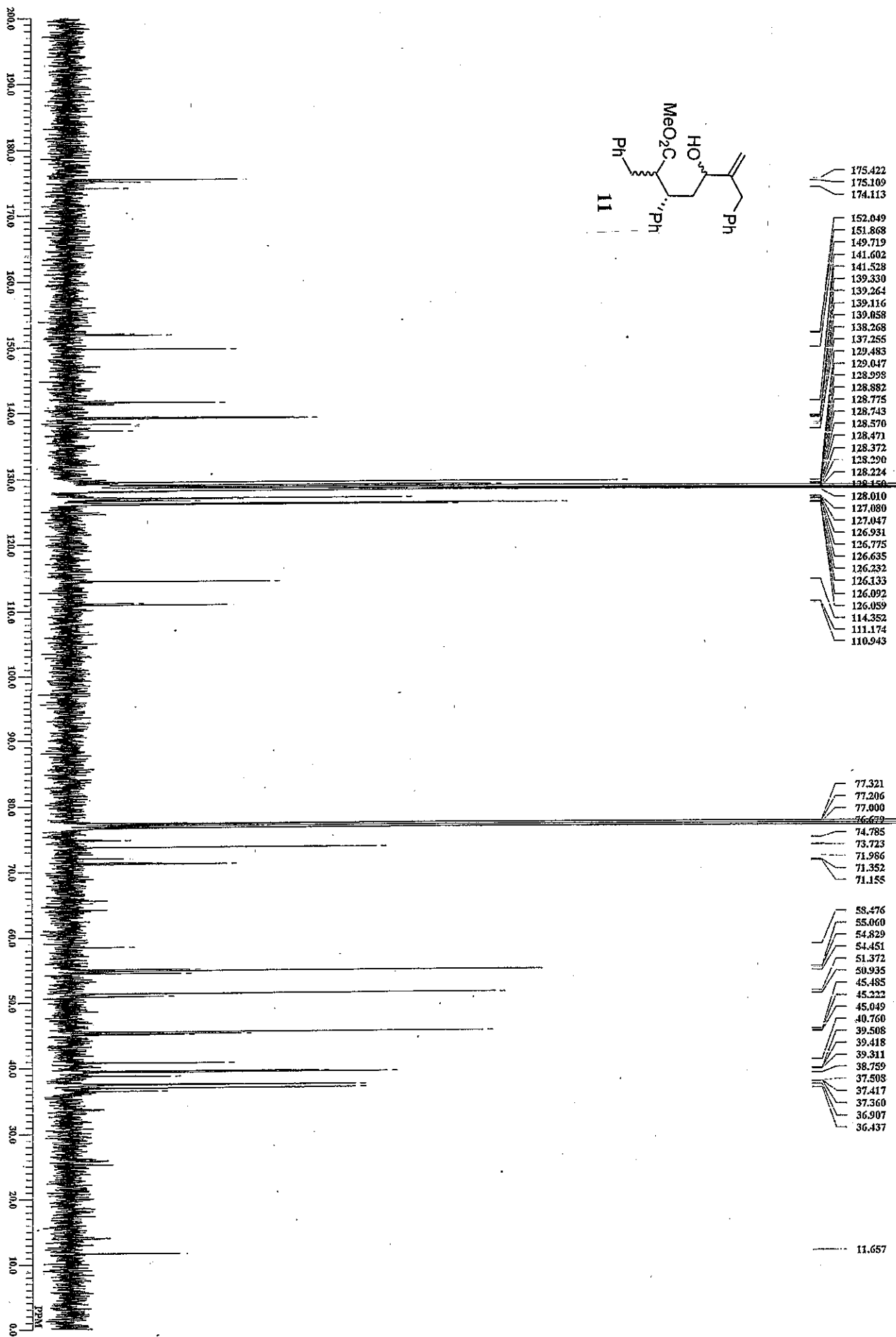


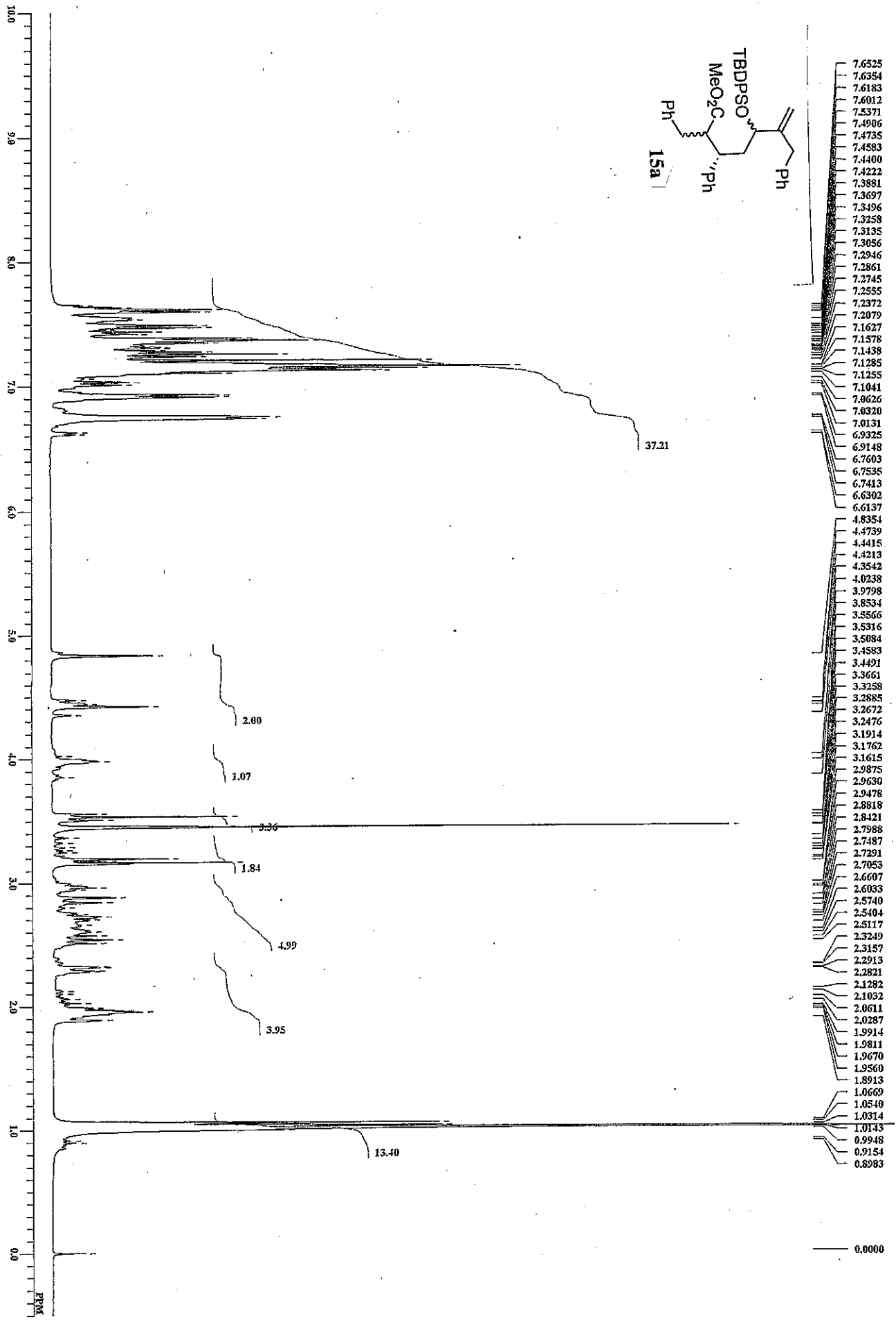
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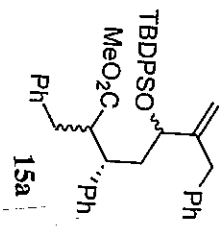
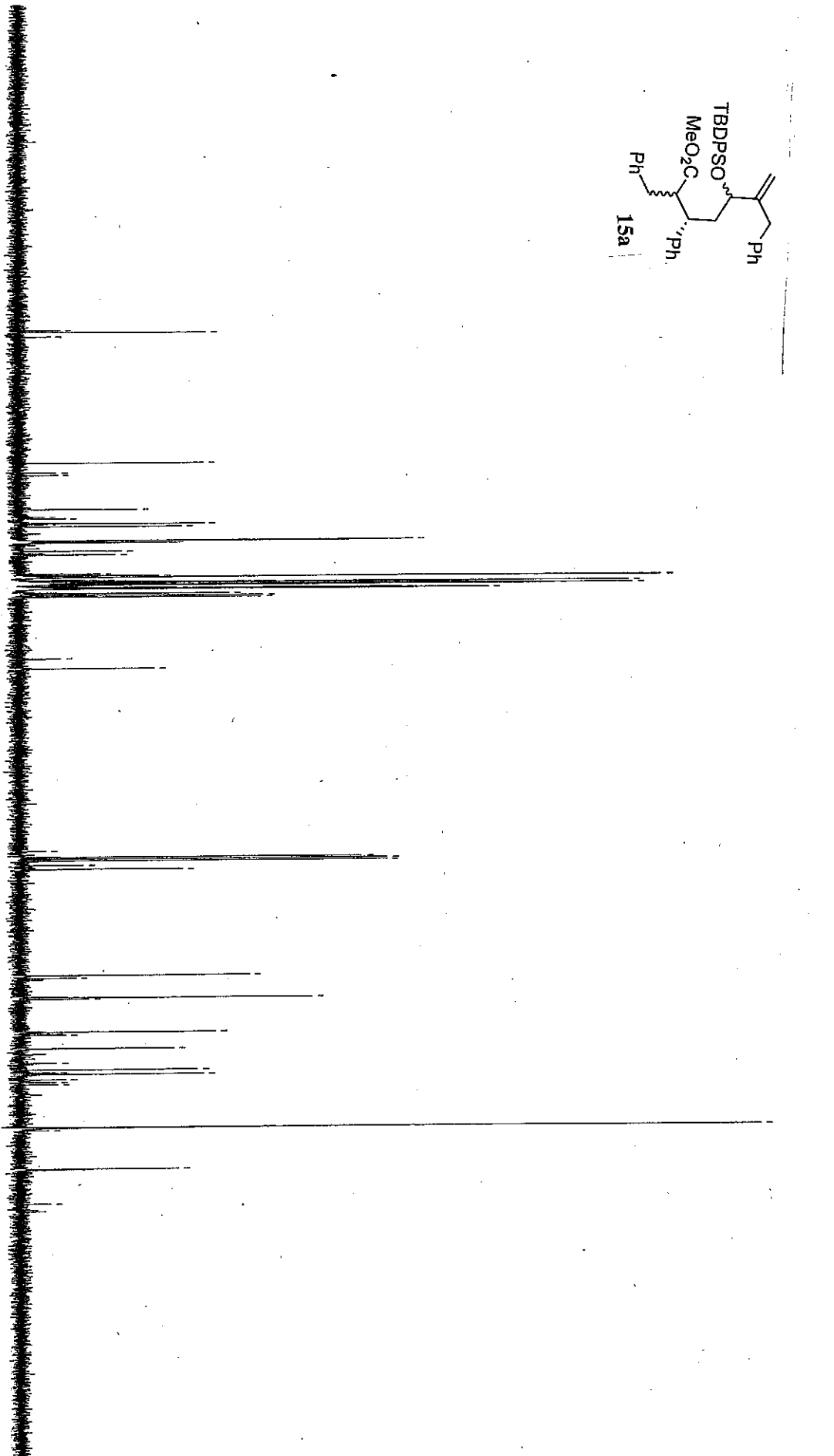




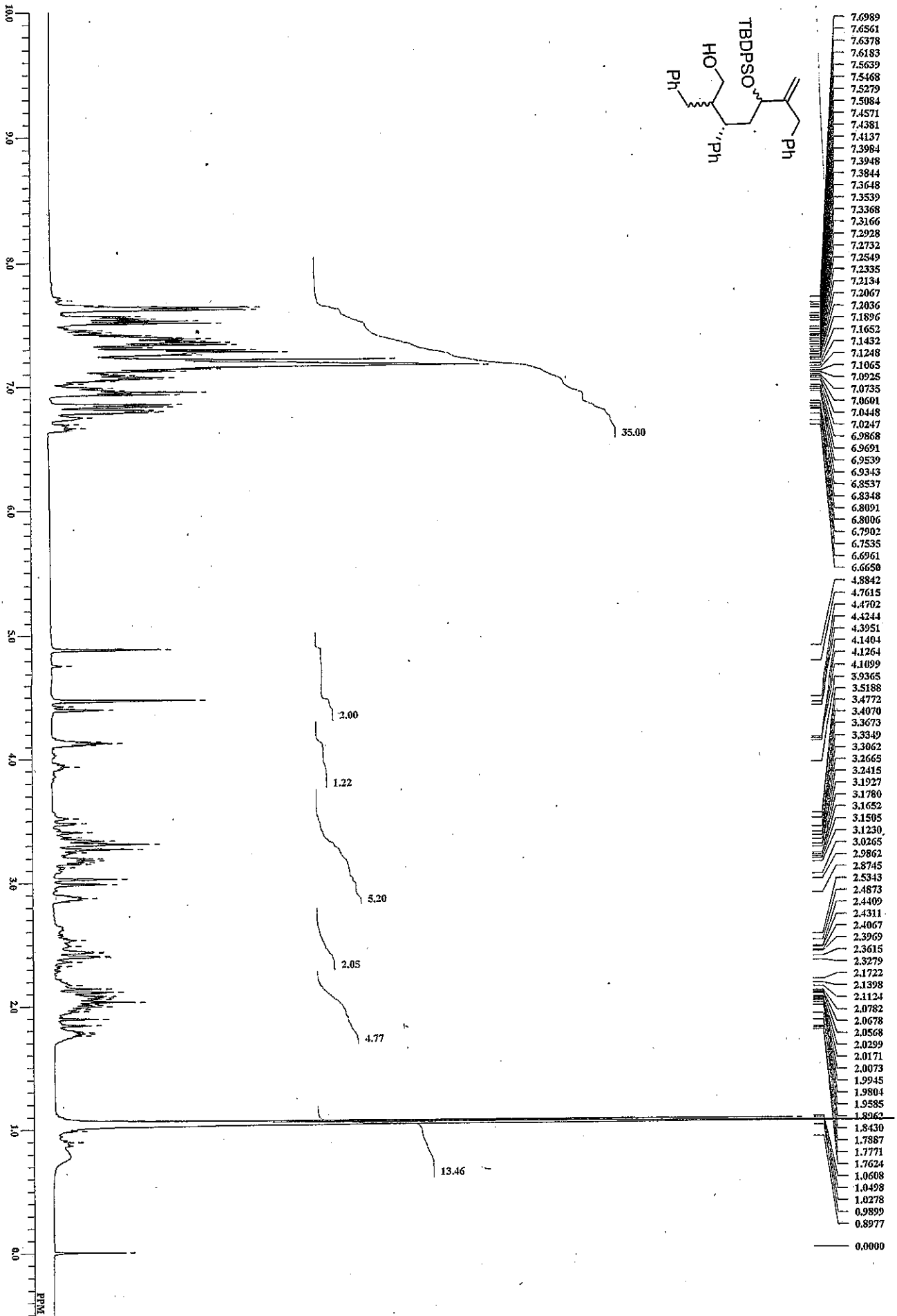




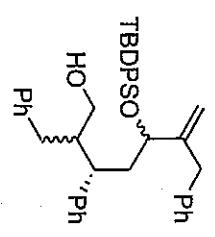
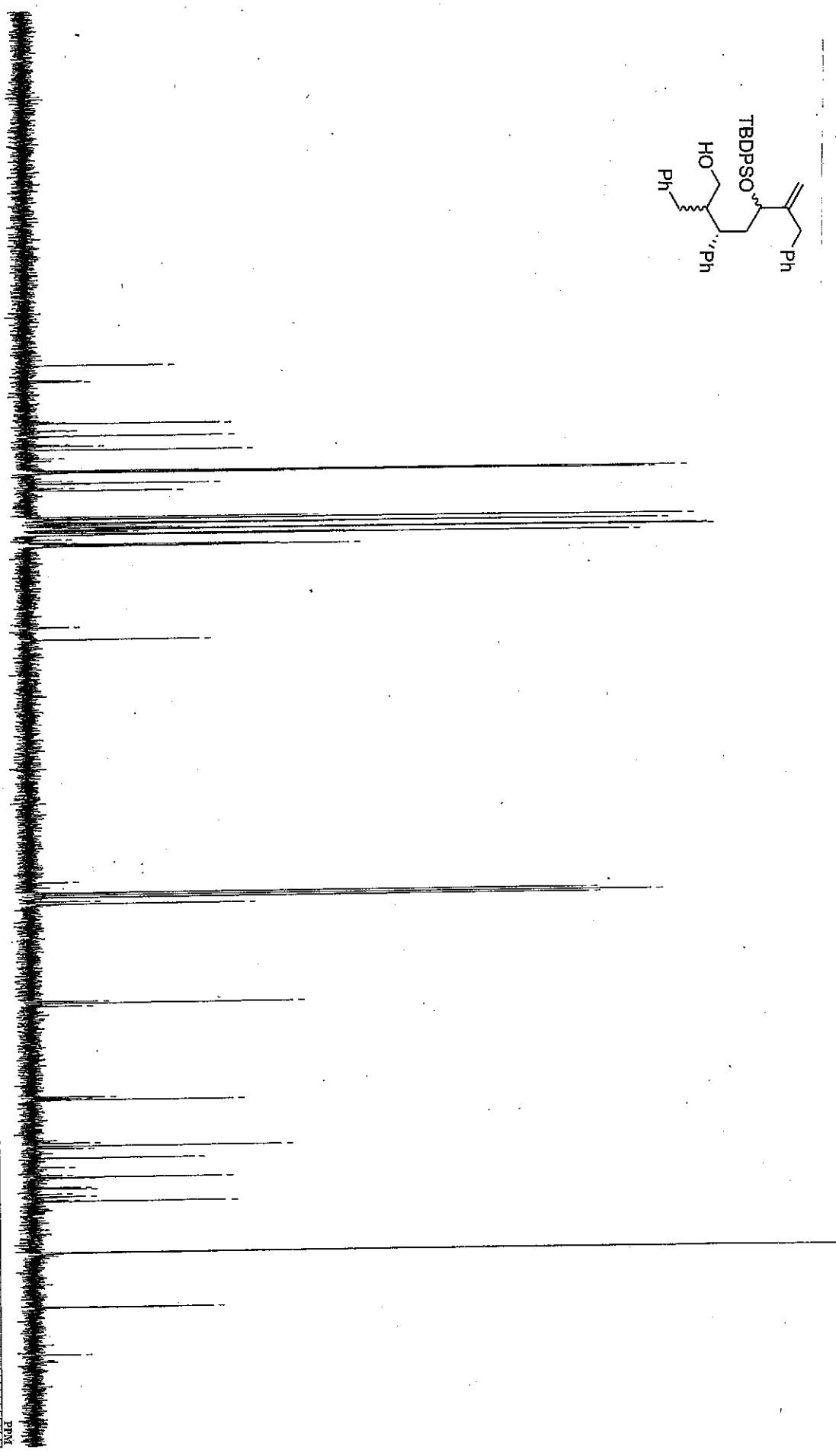
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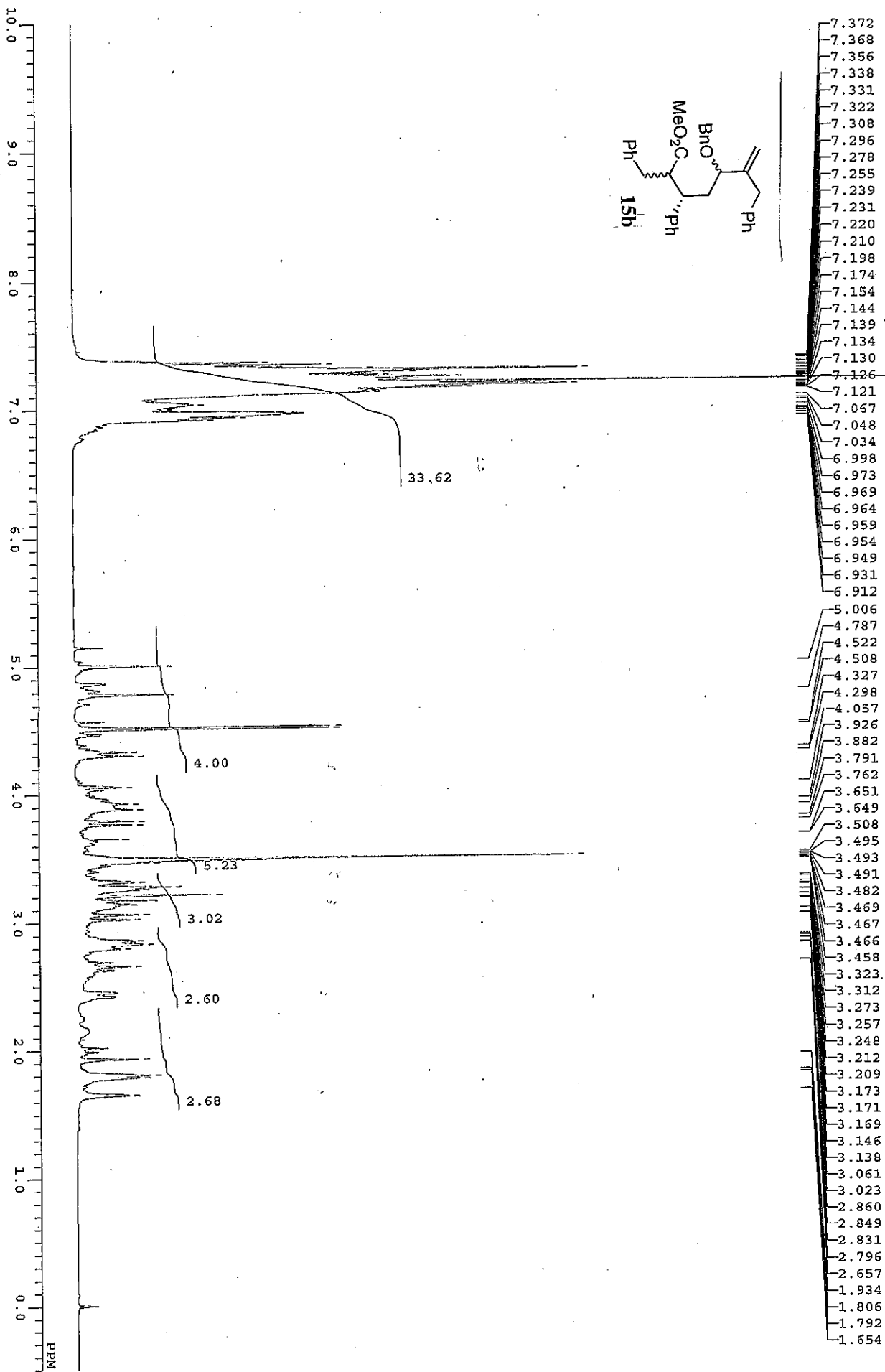
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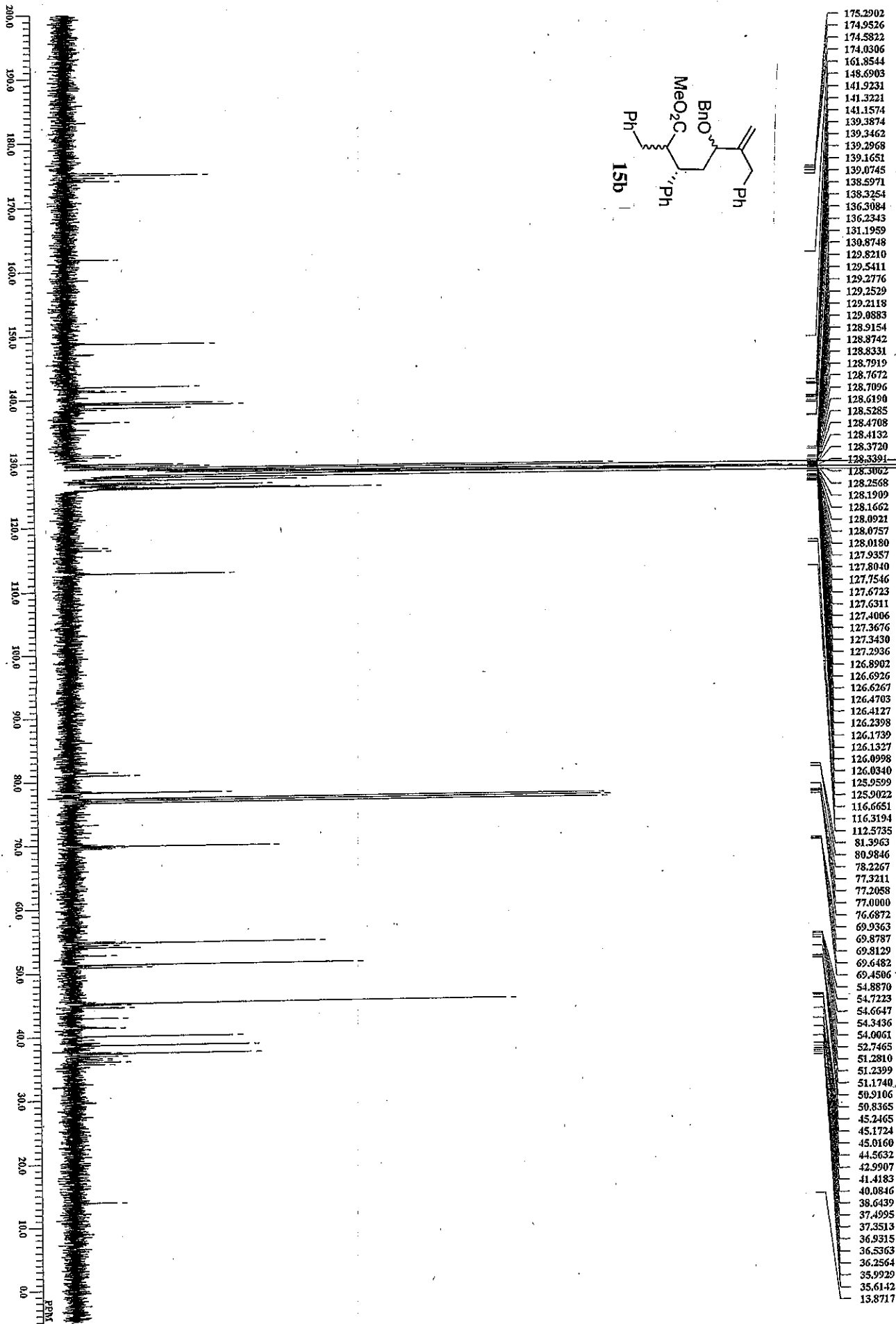


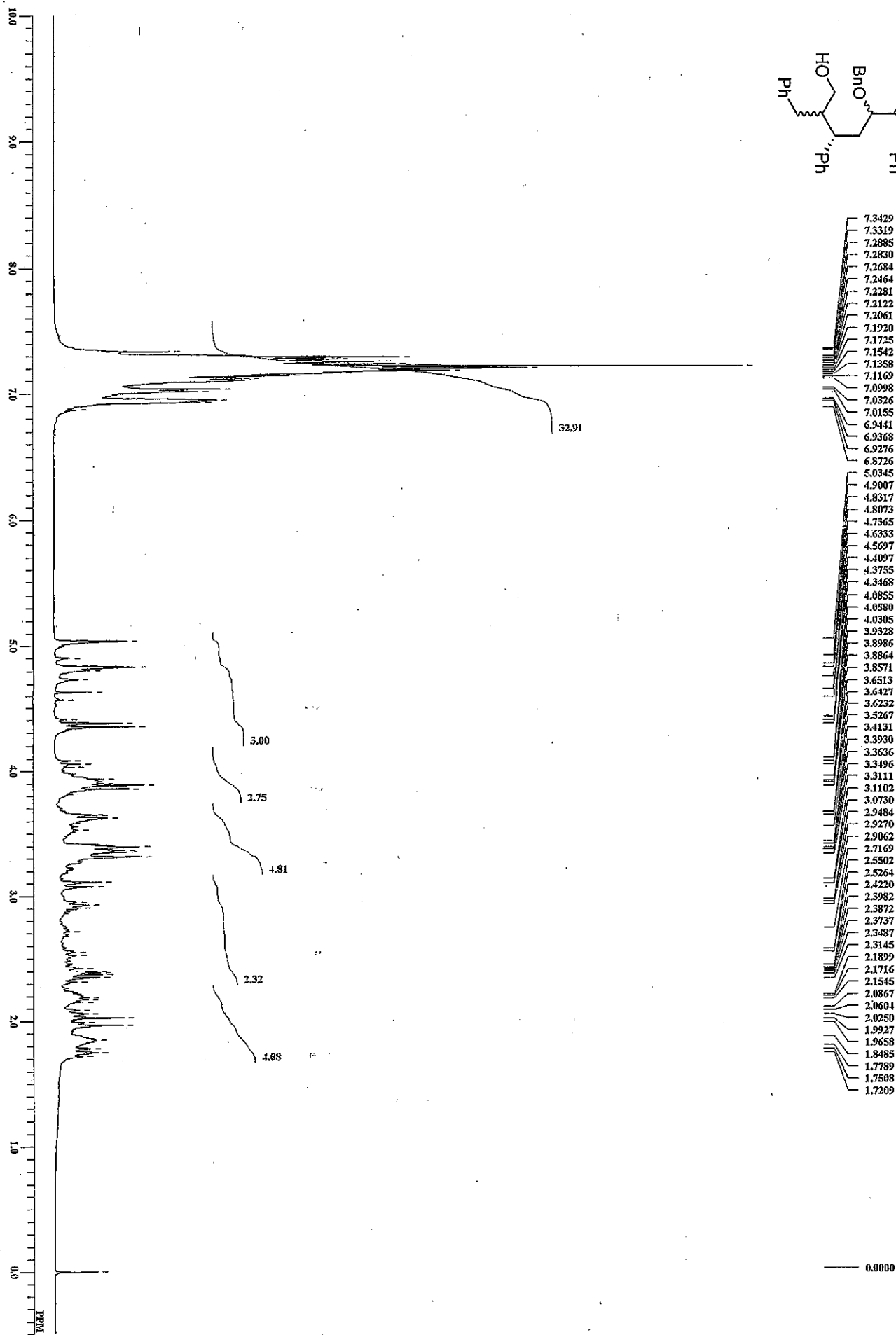
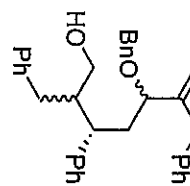
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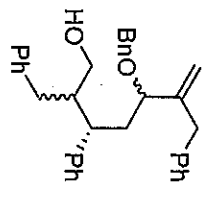
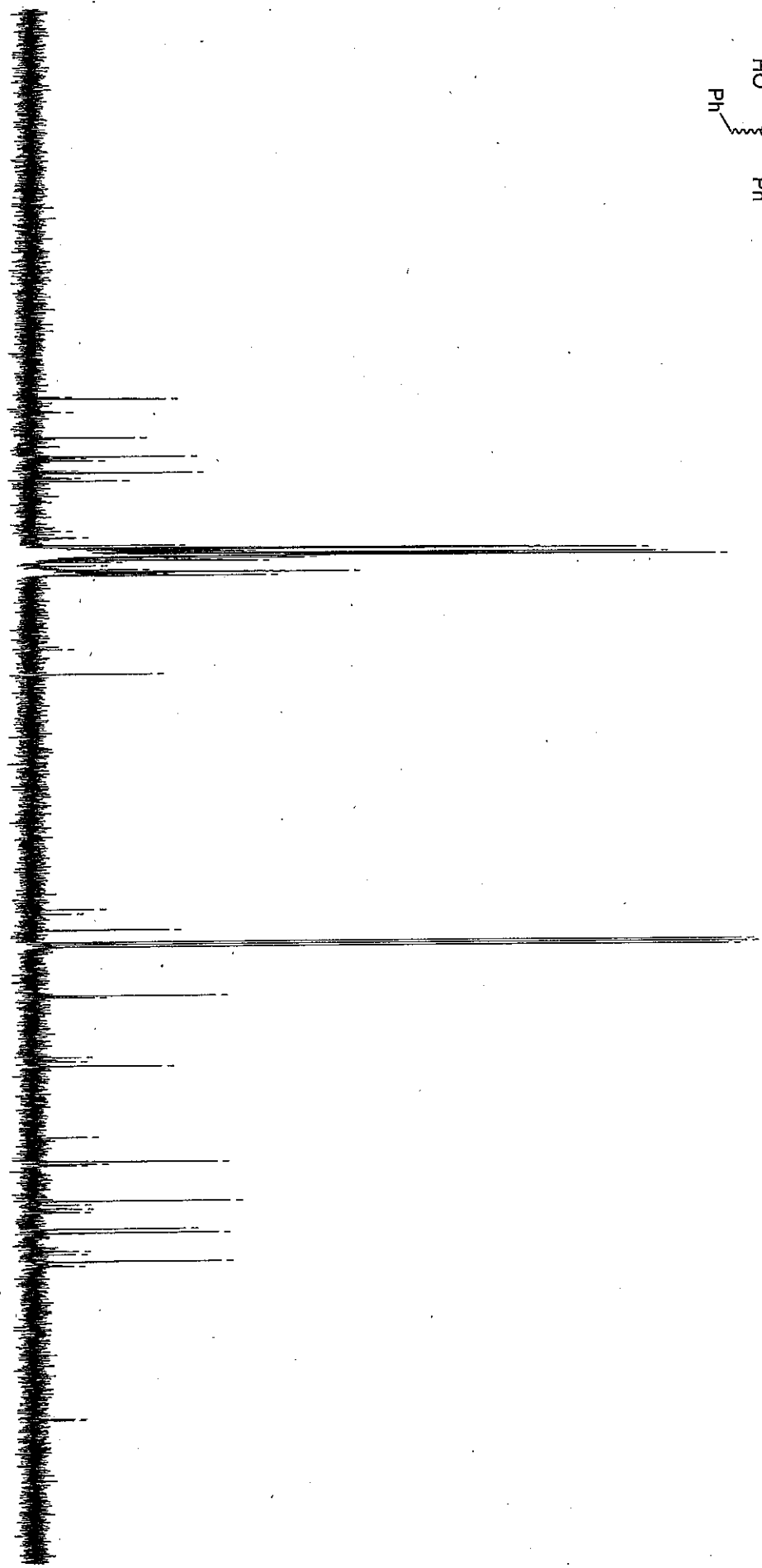




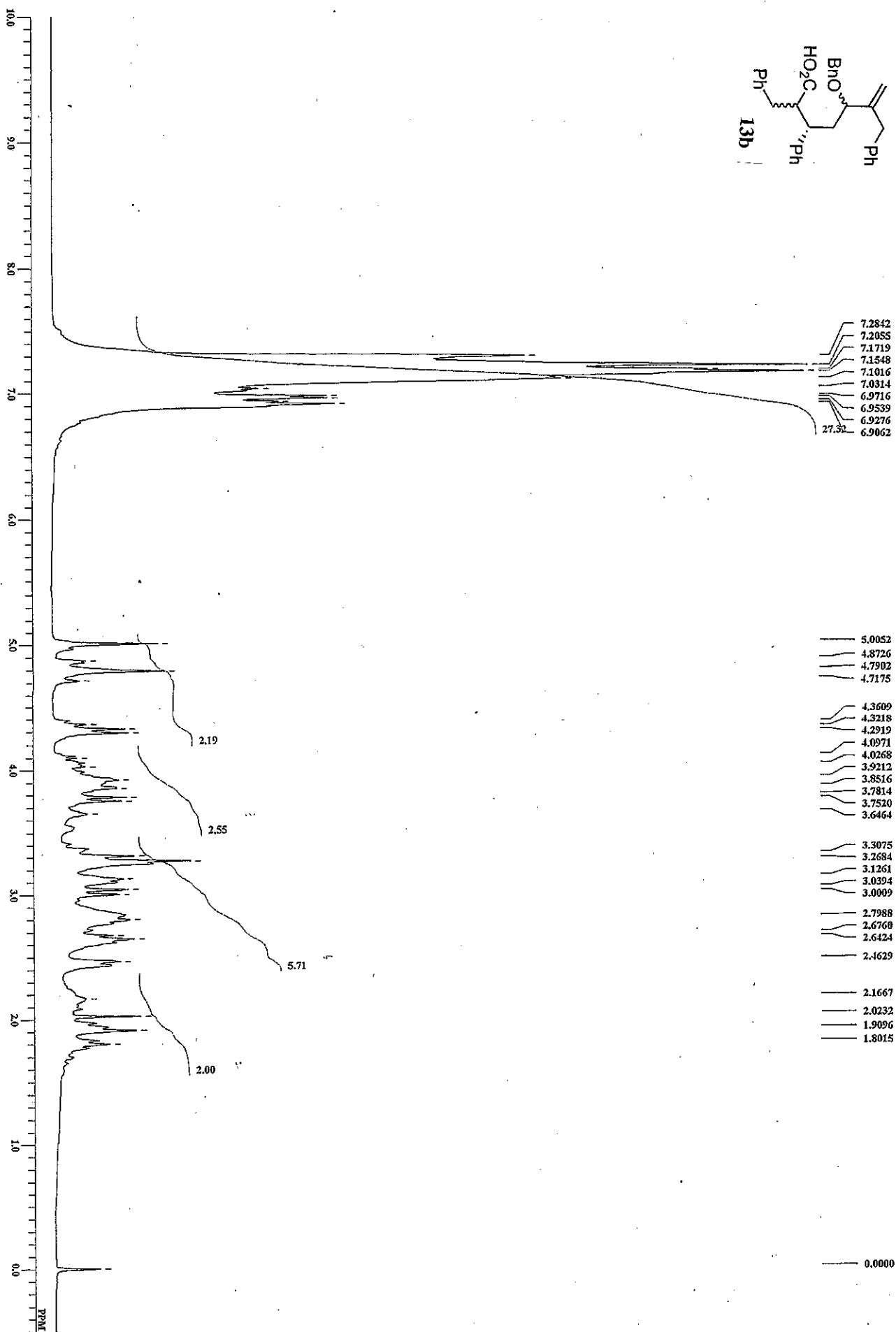
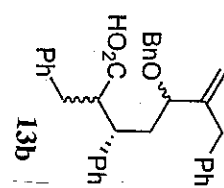


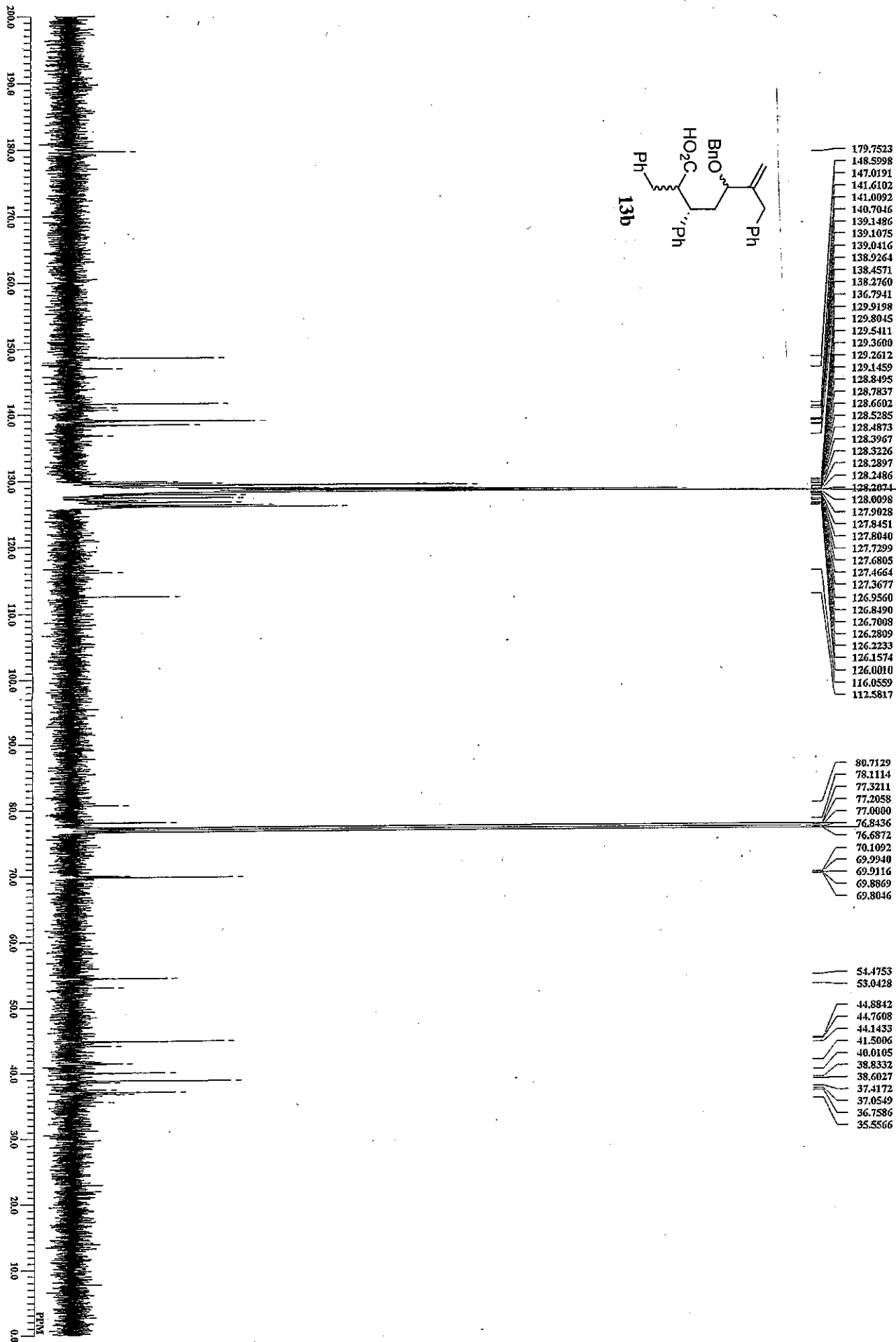


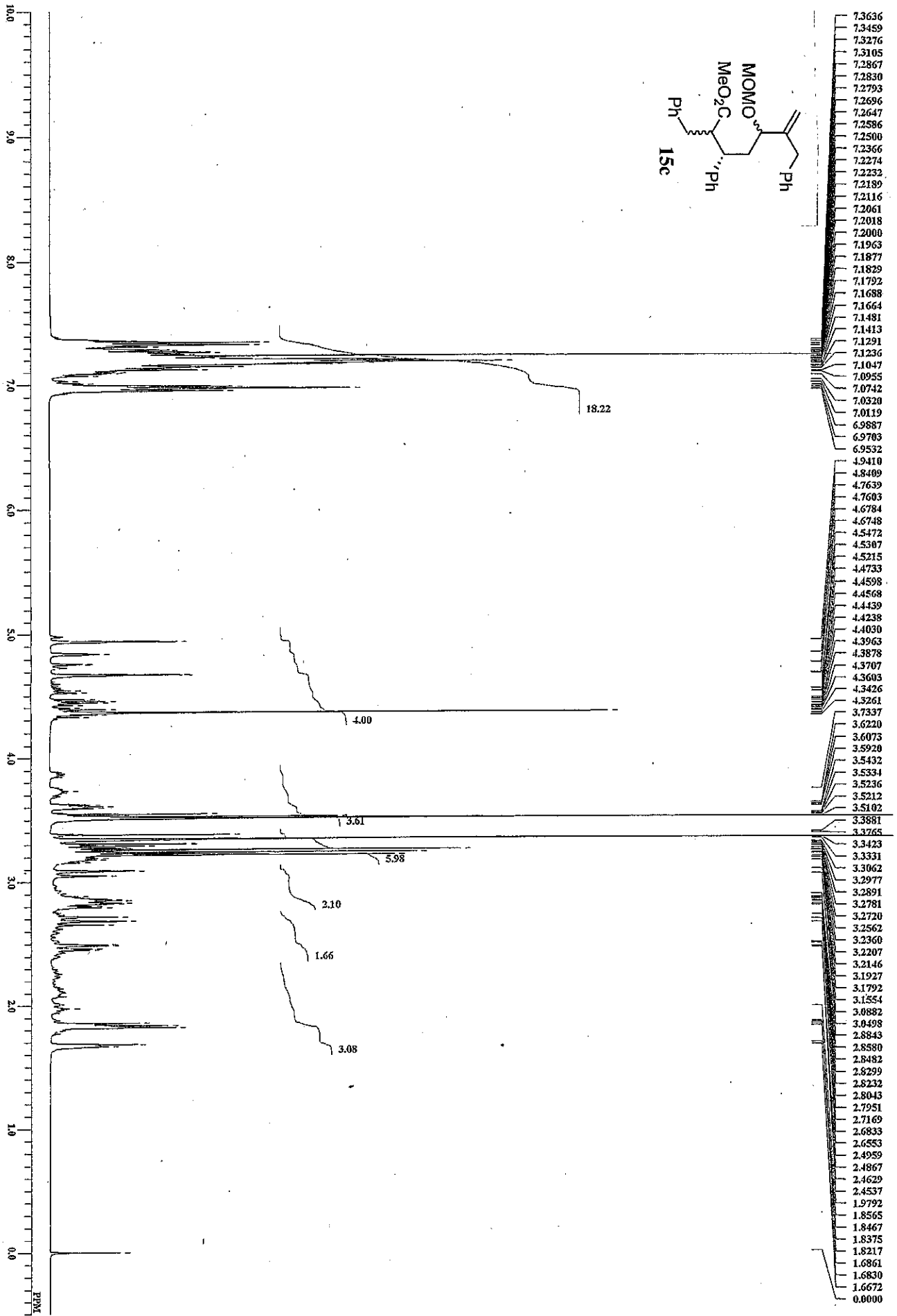
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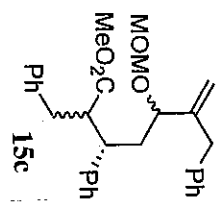
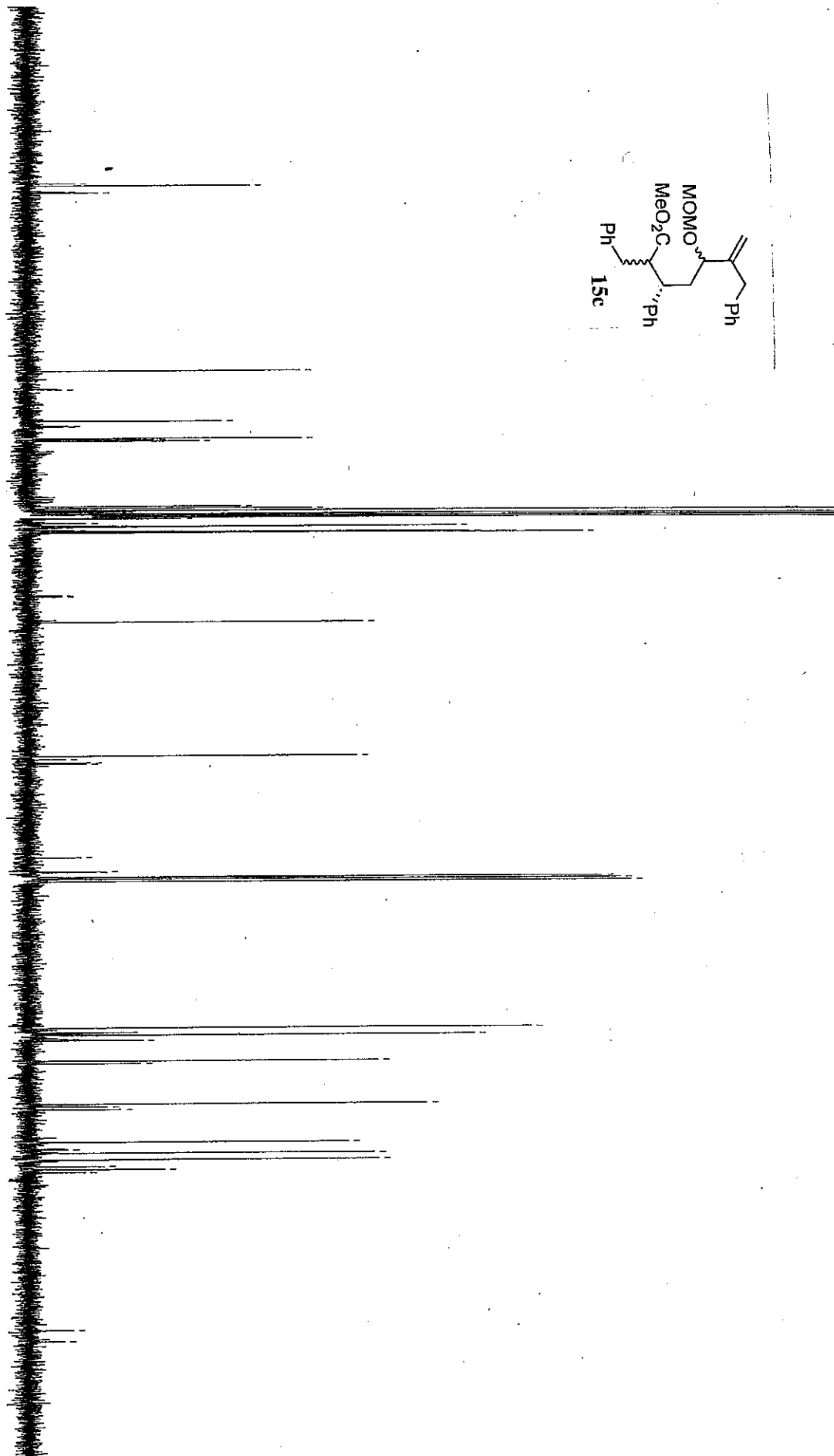
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- 14.0116



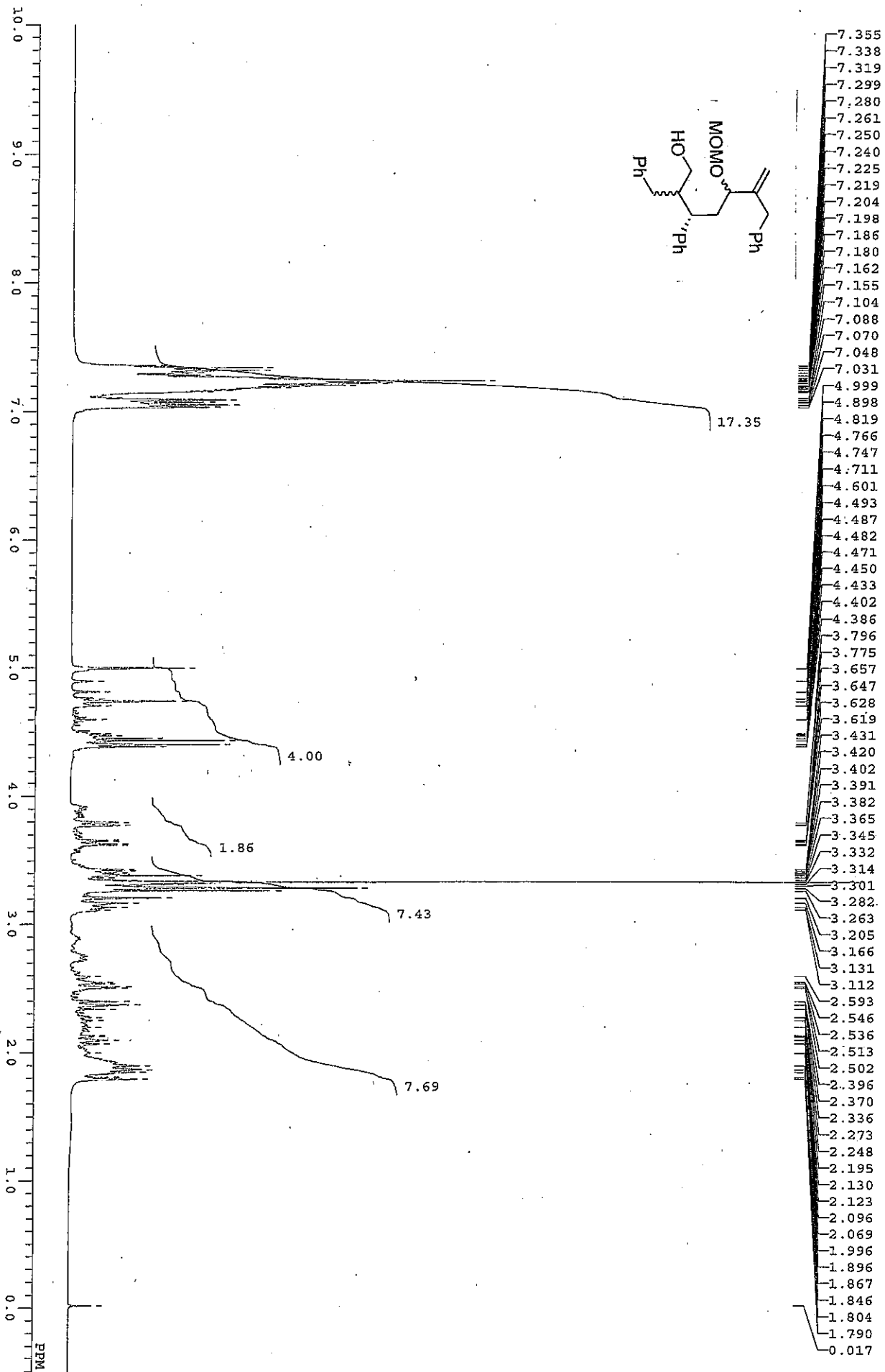




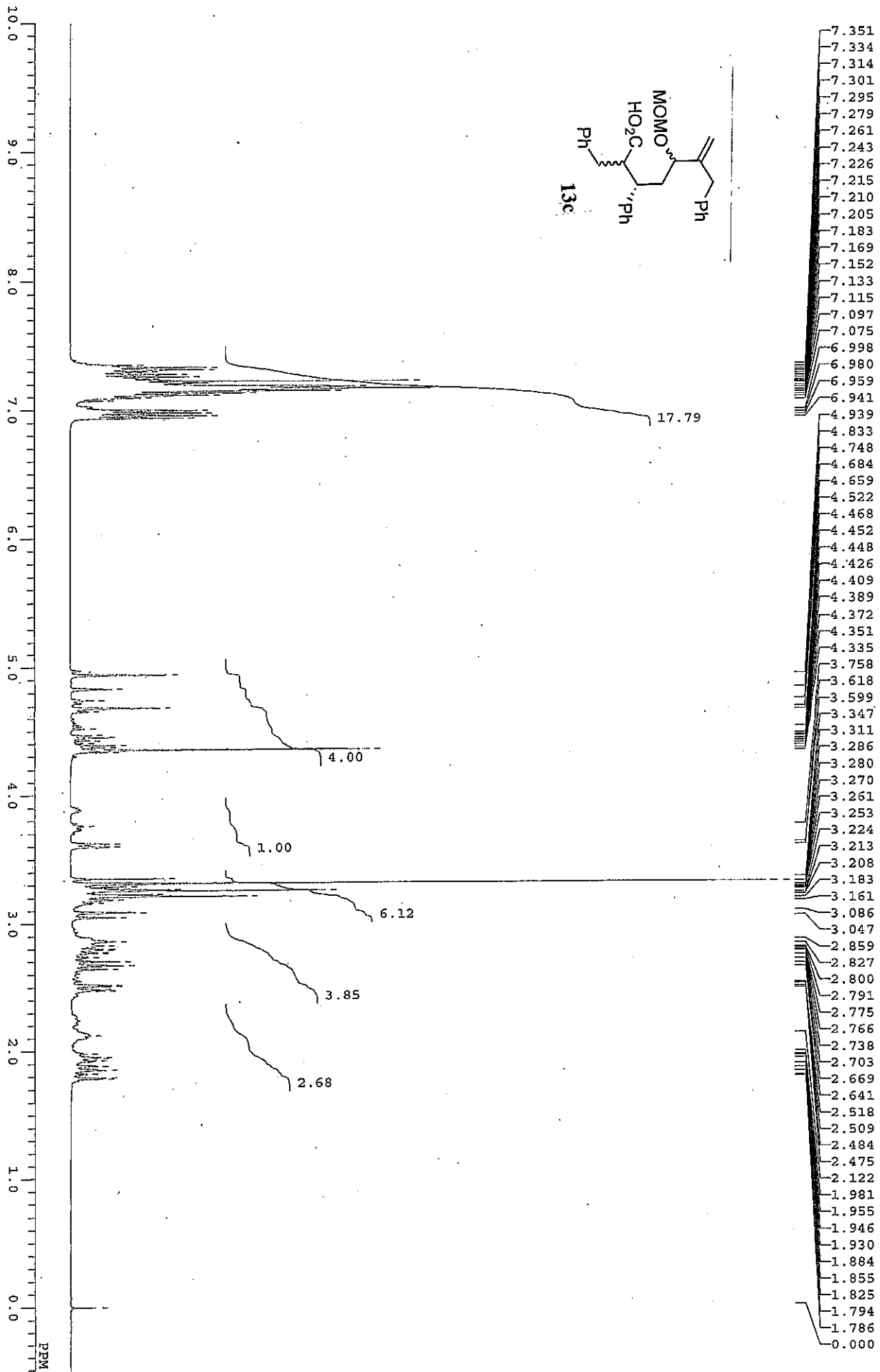
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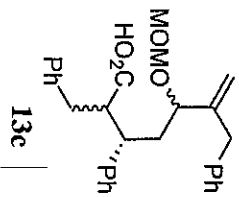
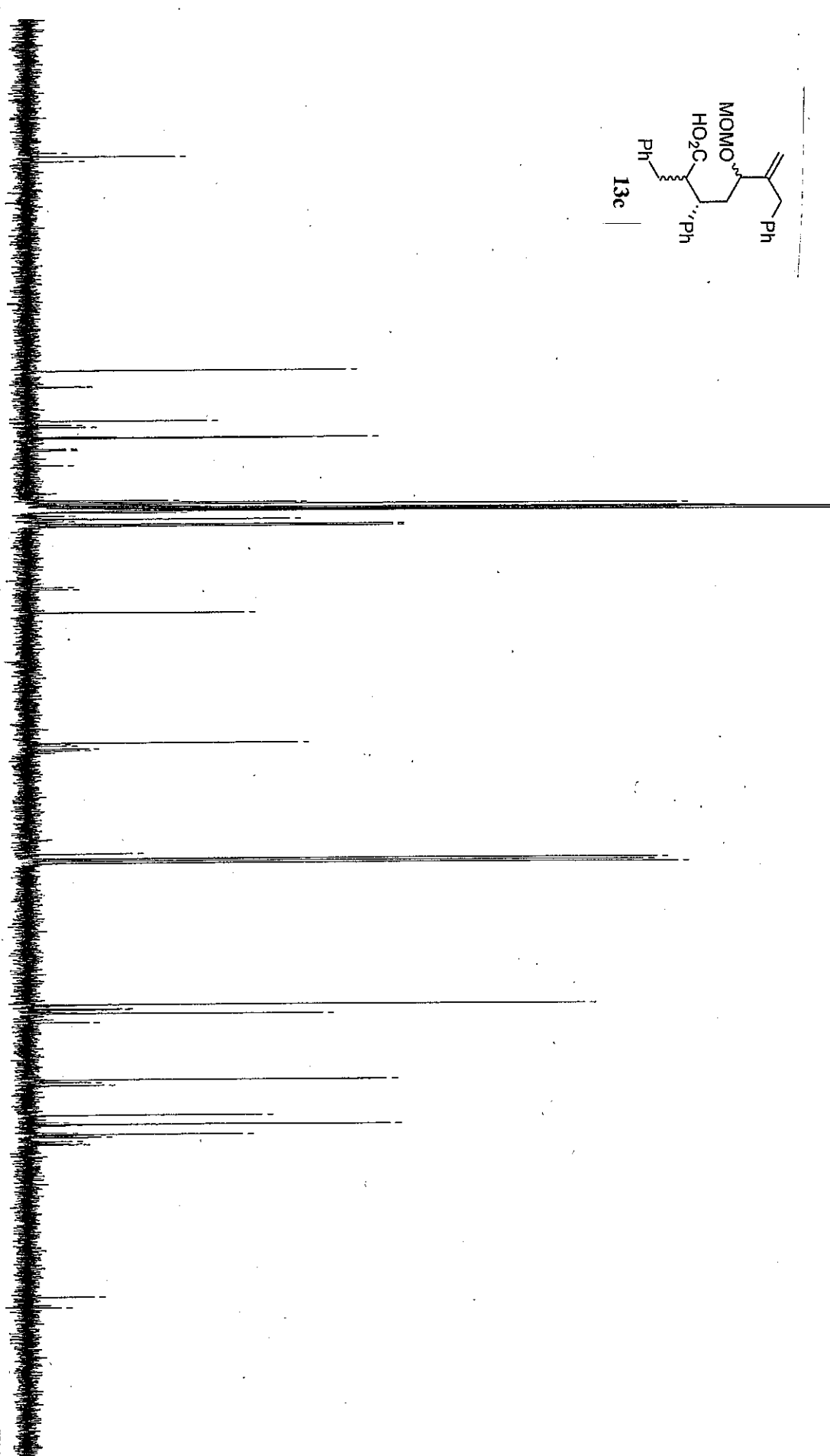
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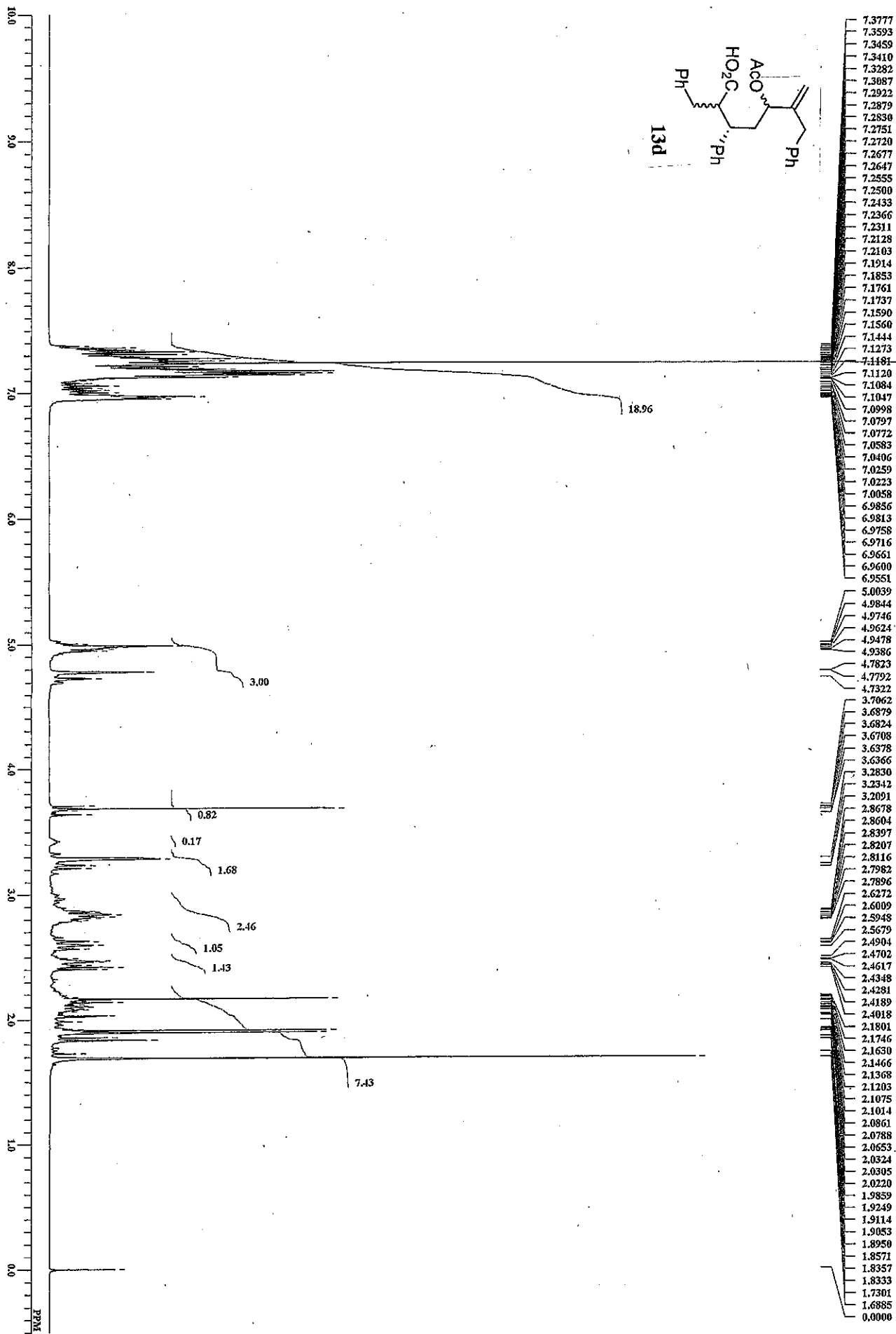


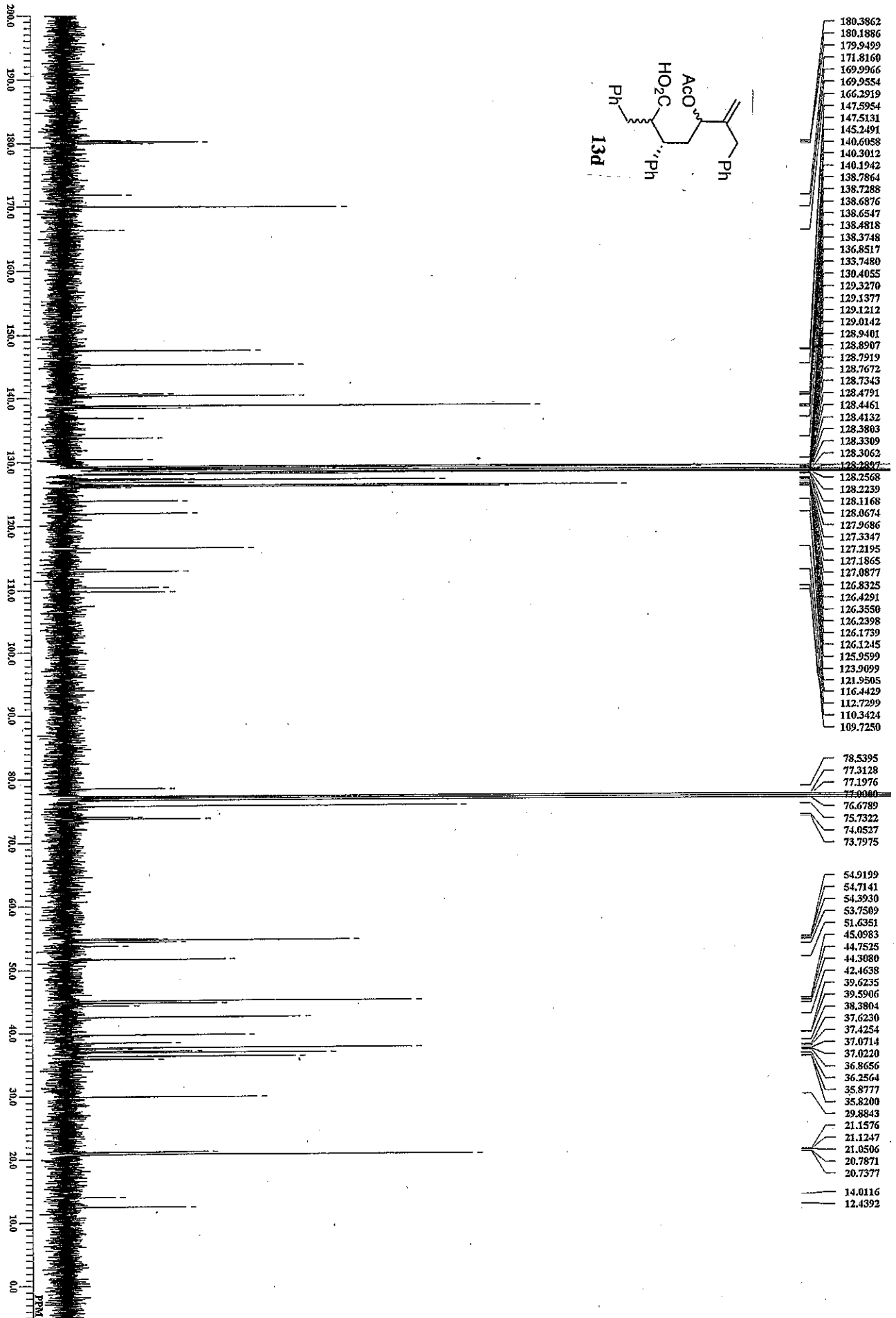


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- 179.1431
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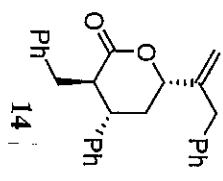






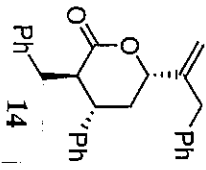
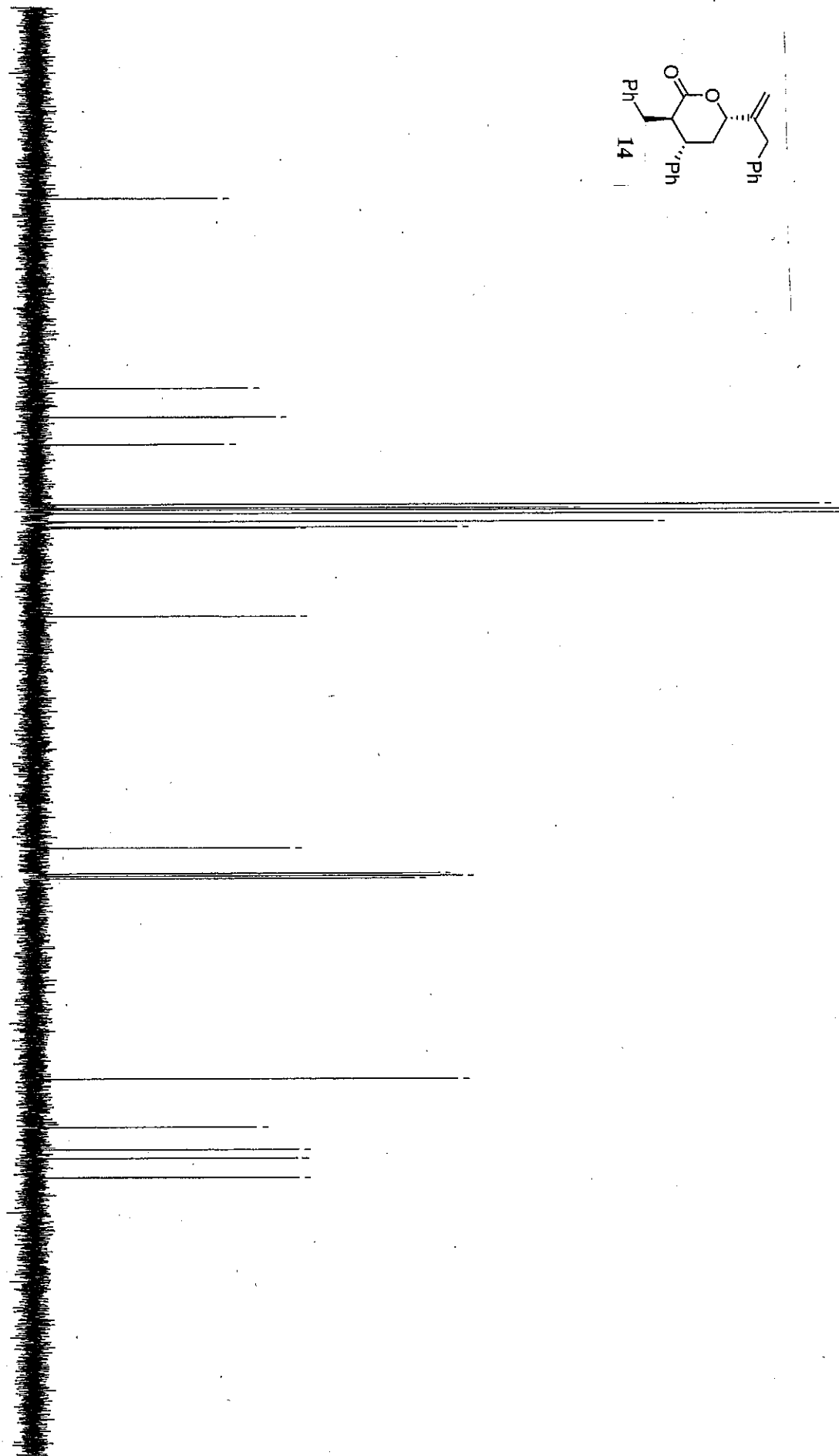
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- 1.00
- 3.07
- 1.00
- 1.01
- 1.01
- 0.96
- 0.97

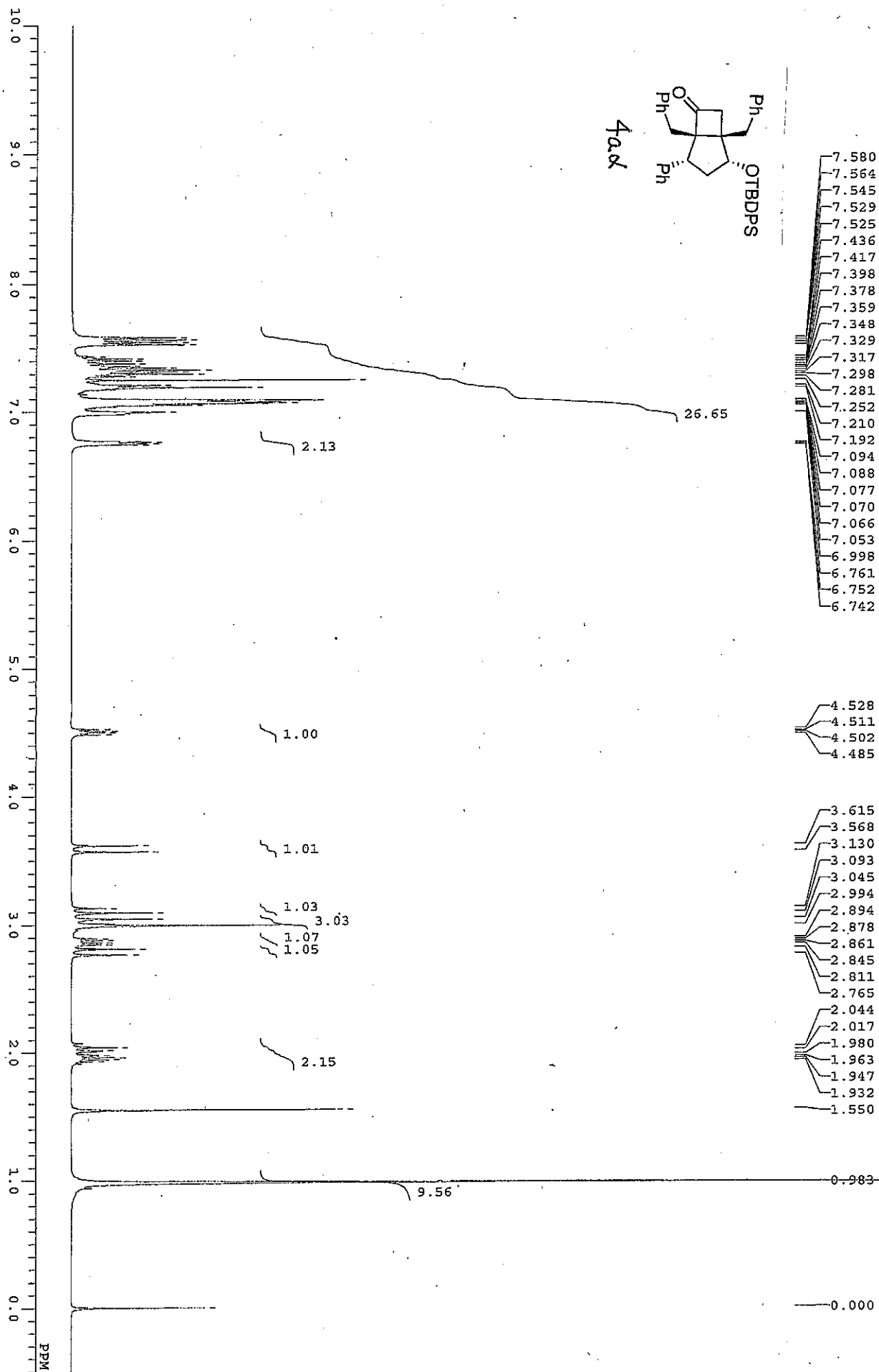


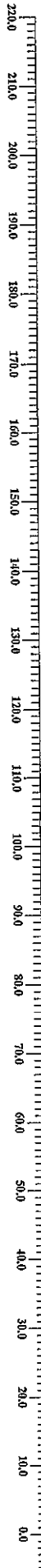
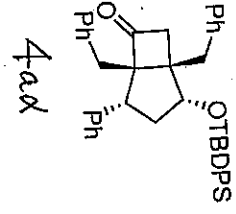
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- 7.2318
- 7.2184
- 7.2050
- 7.2001
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- 4.5036
- 4.4804
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- 1.8144
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- 76.6872
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- 41.5665
- 38.4216
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- 34.4699





- 138.1278
- 138.0208
- 136.9011
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- 135.8144
- 134.1020
- 133.6080
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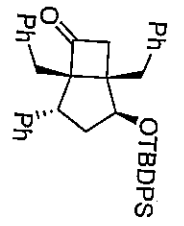
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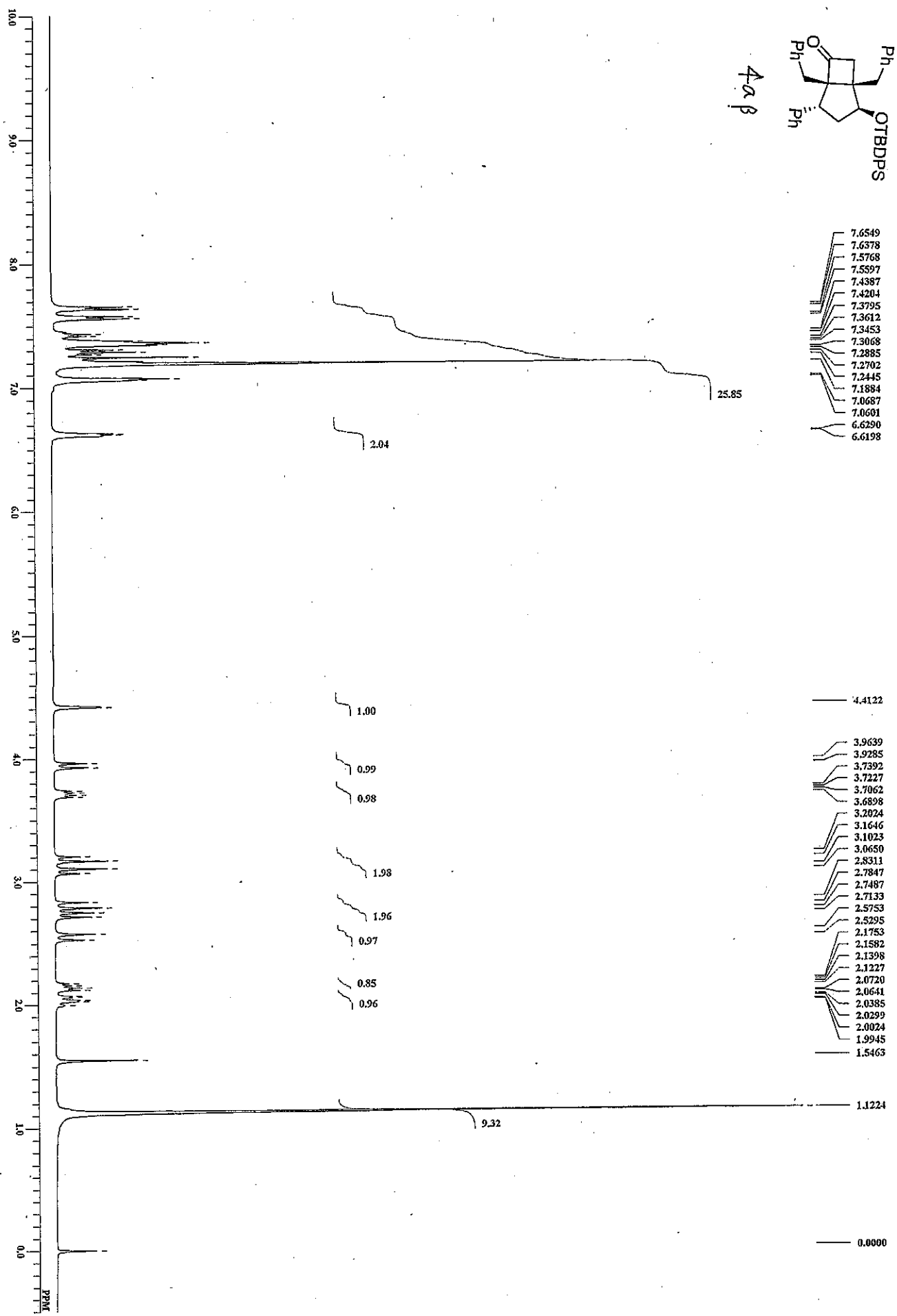
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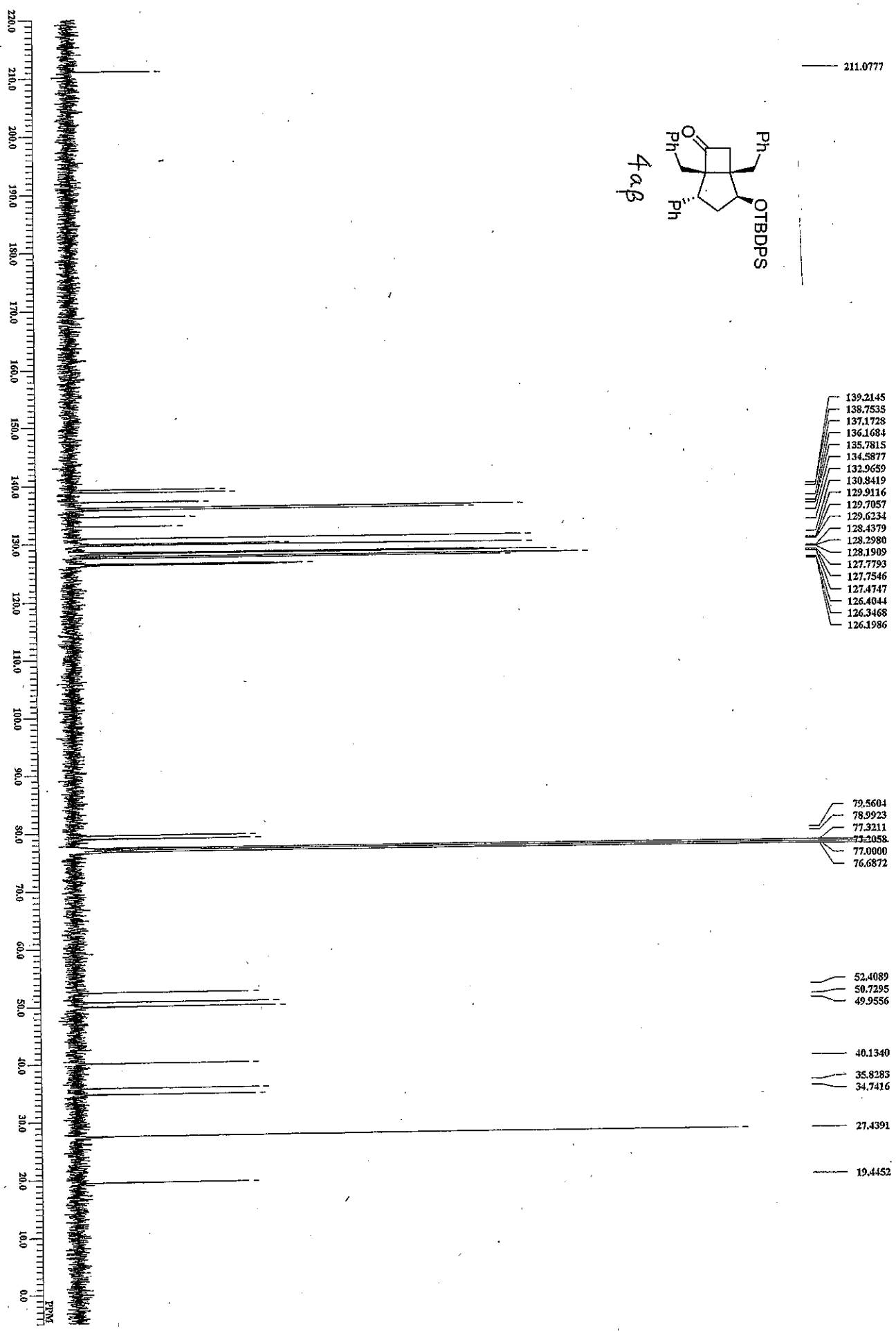
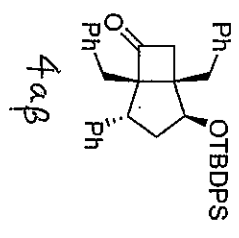
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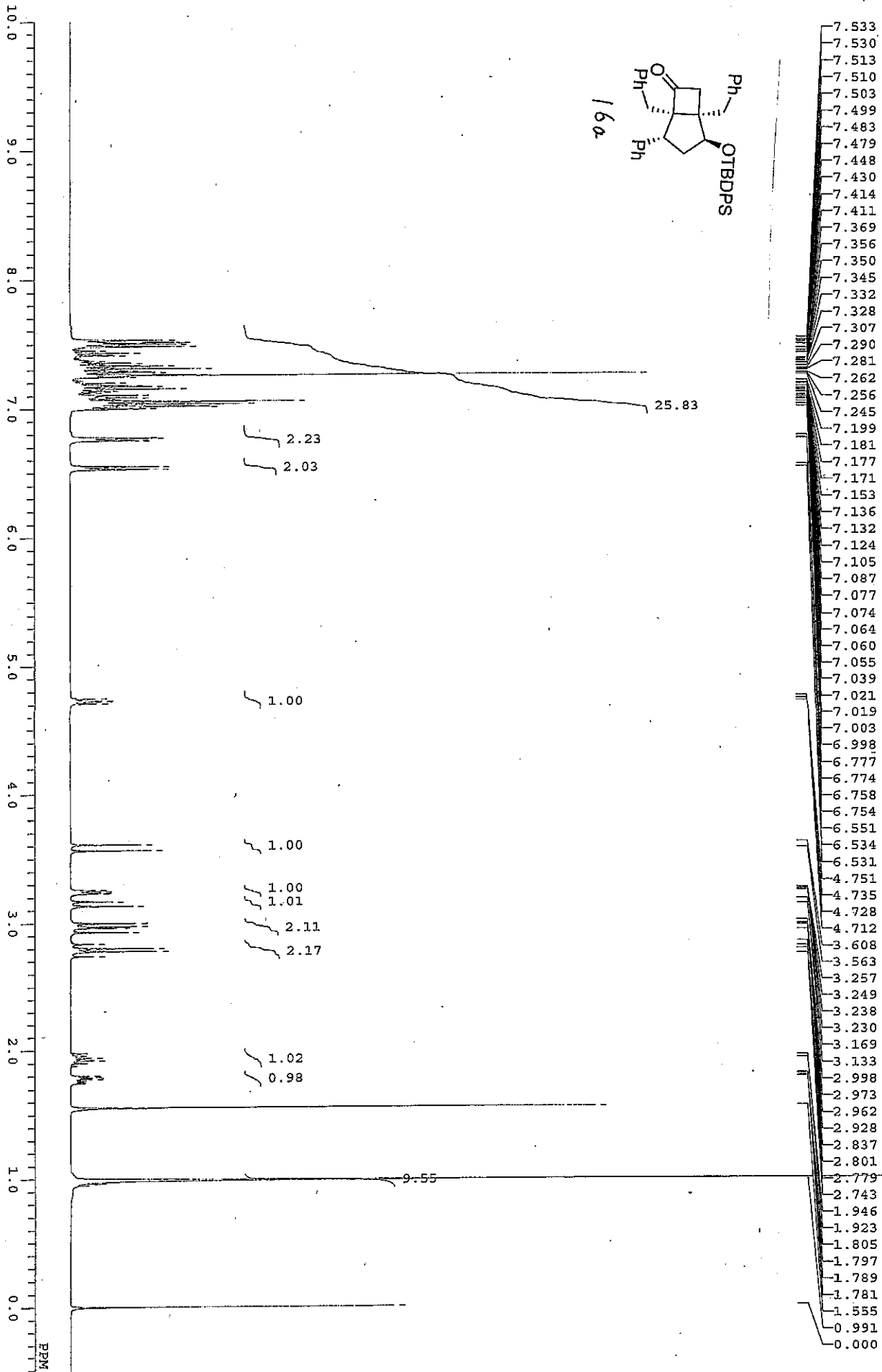


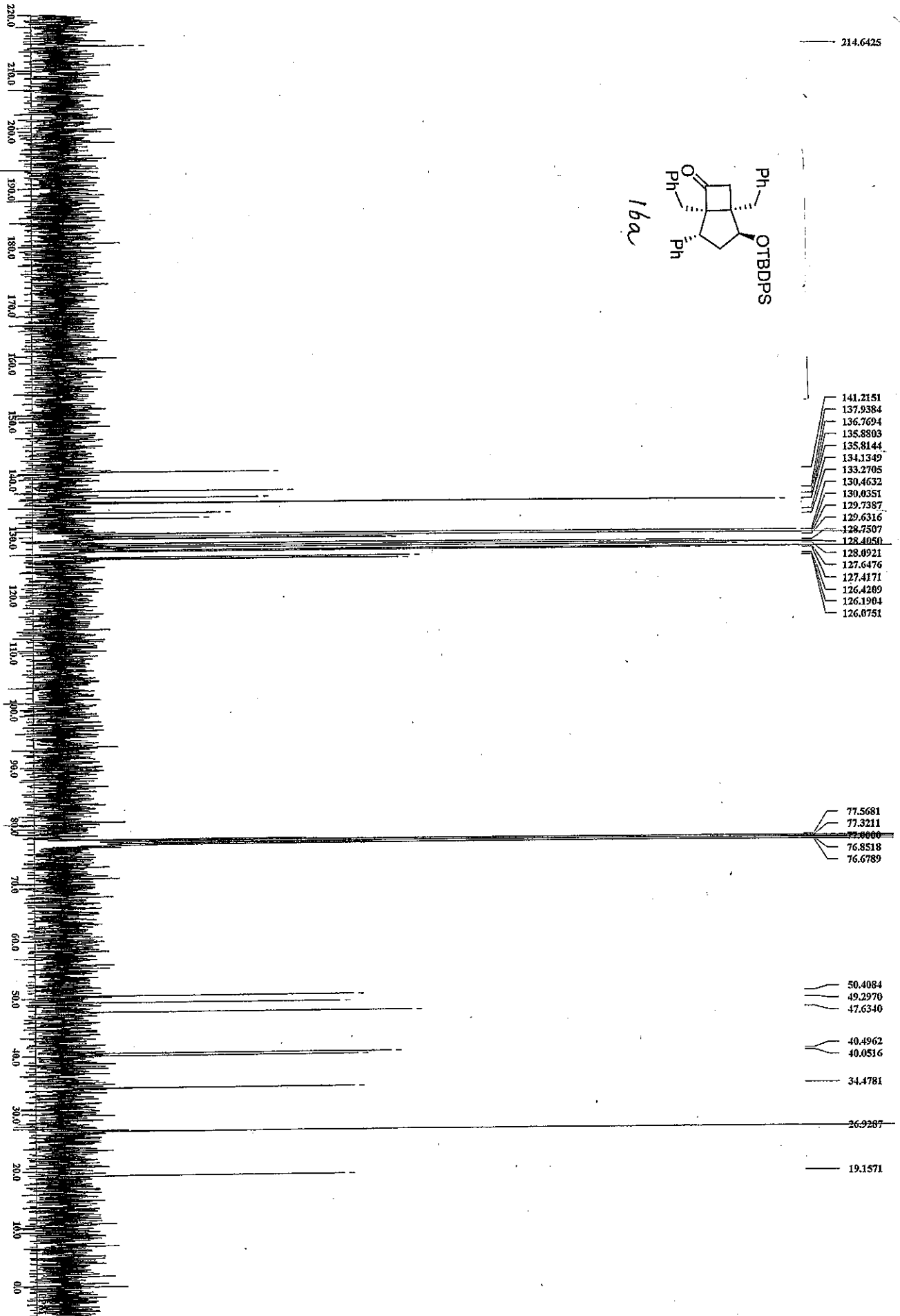
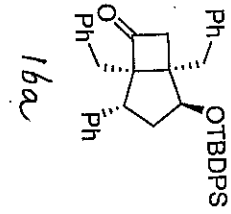
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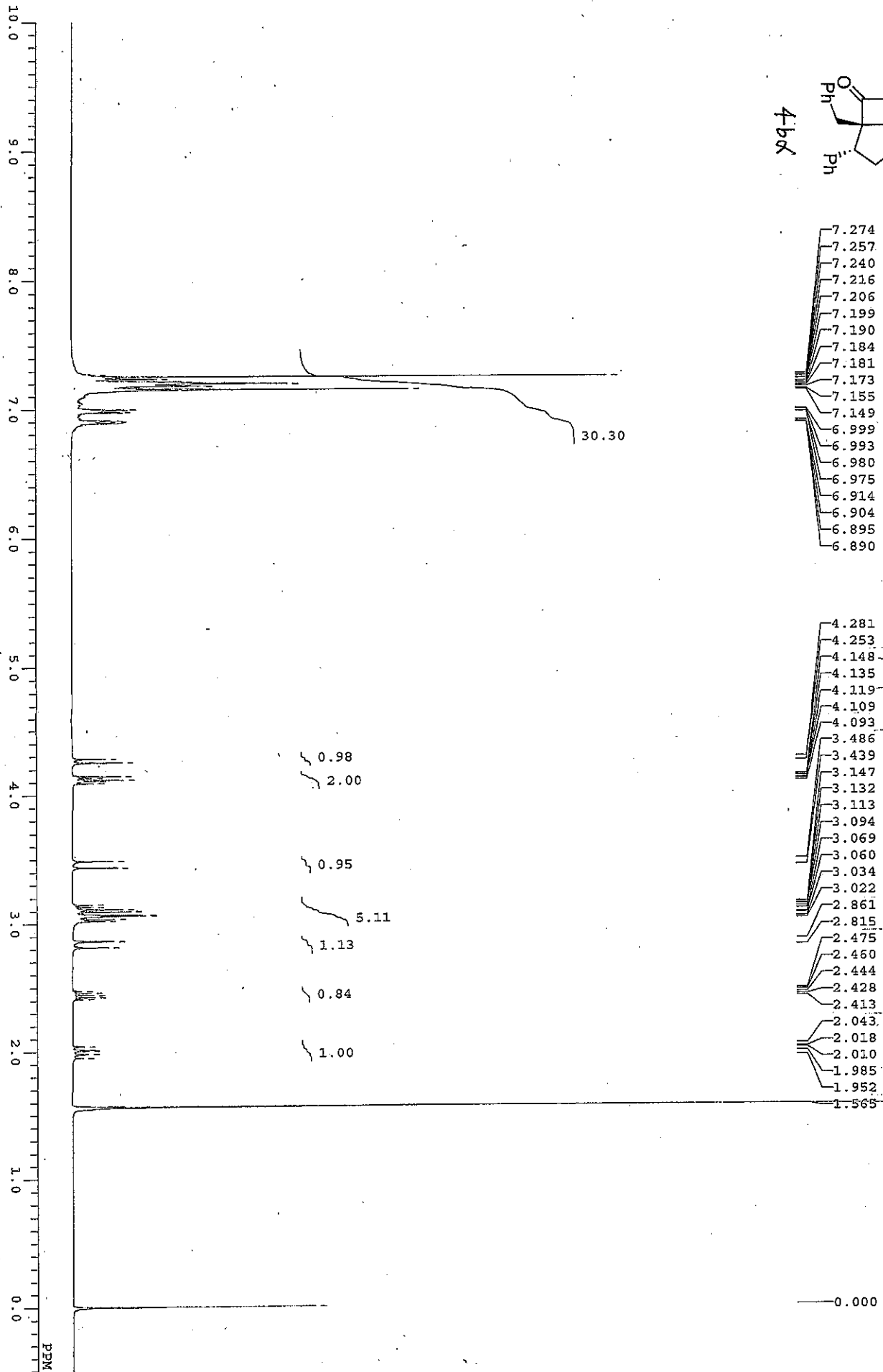
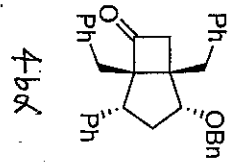


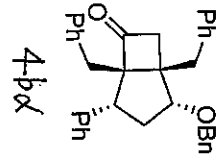
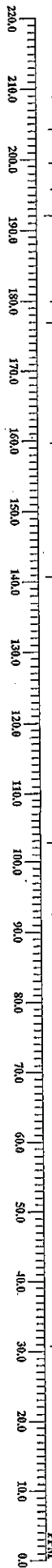
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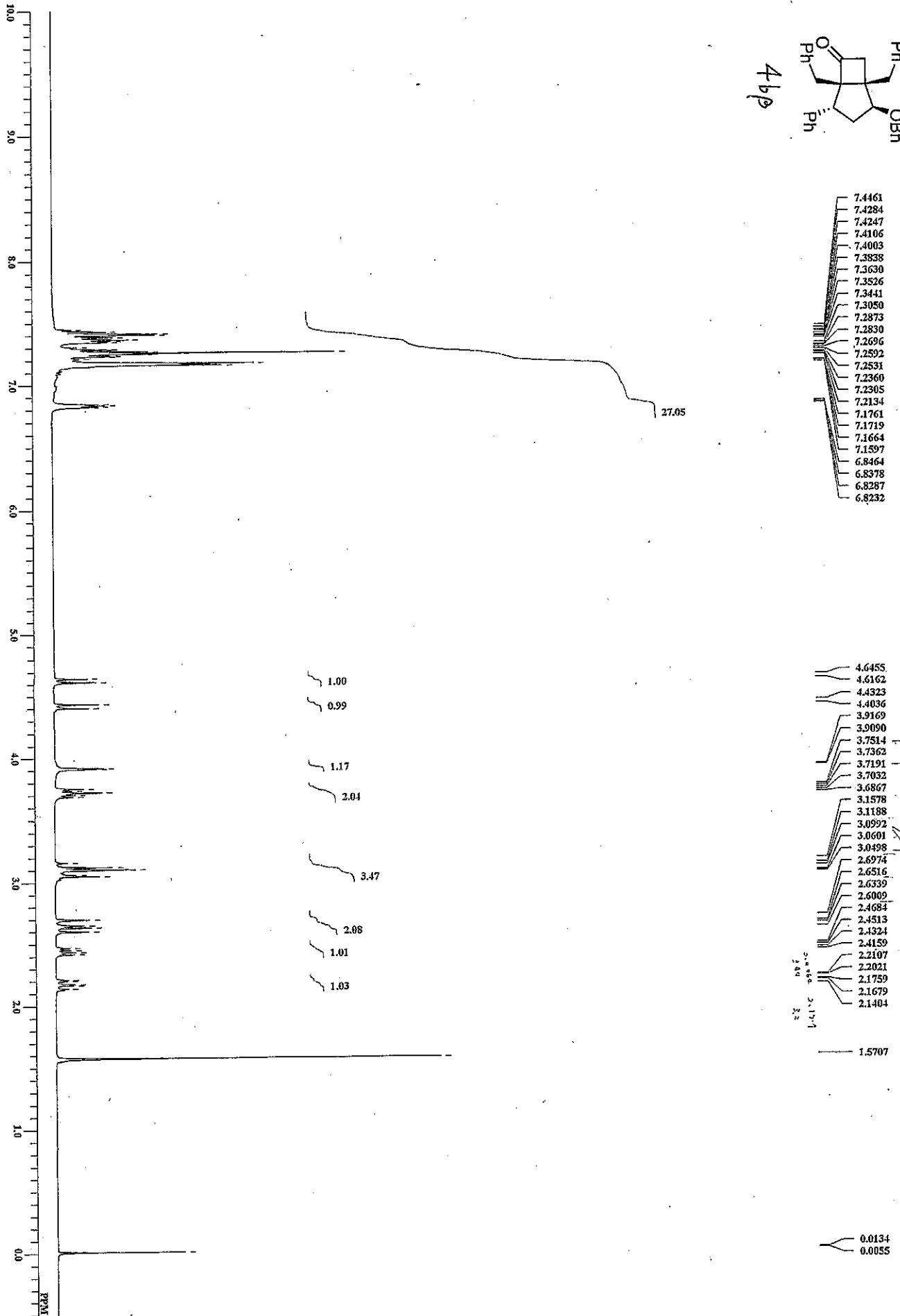
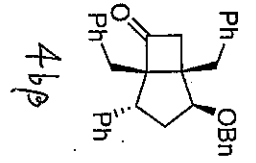


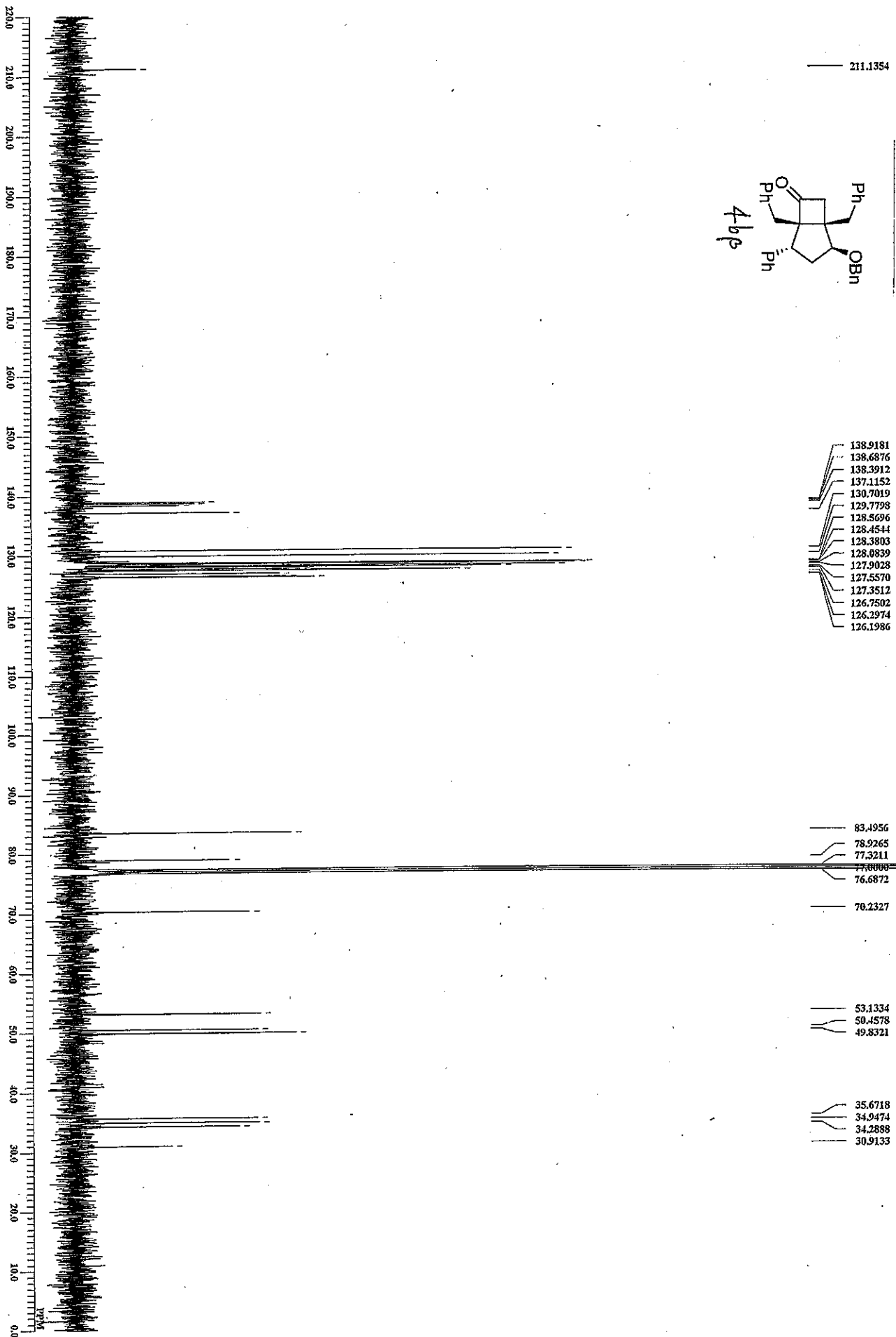
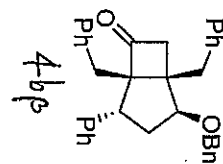


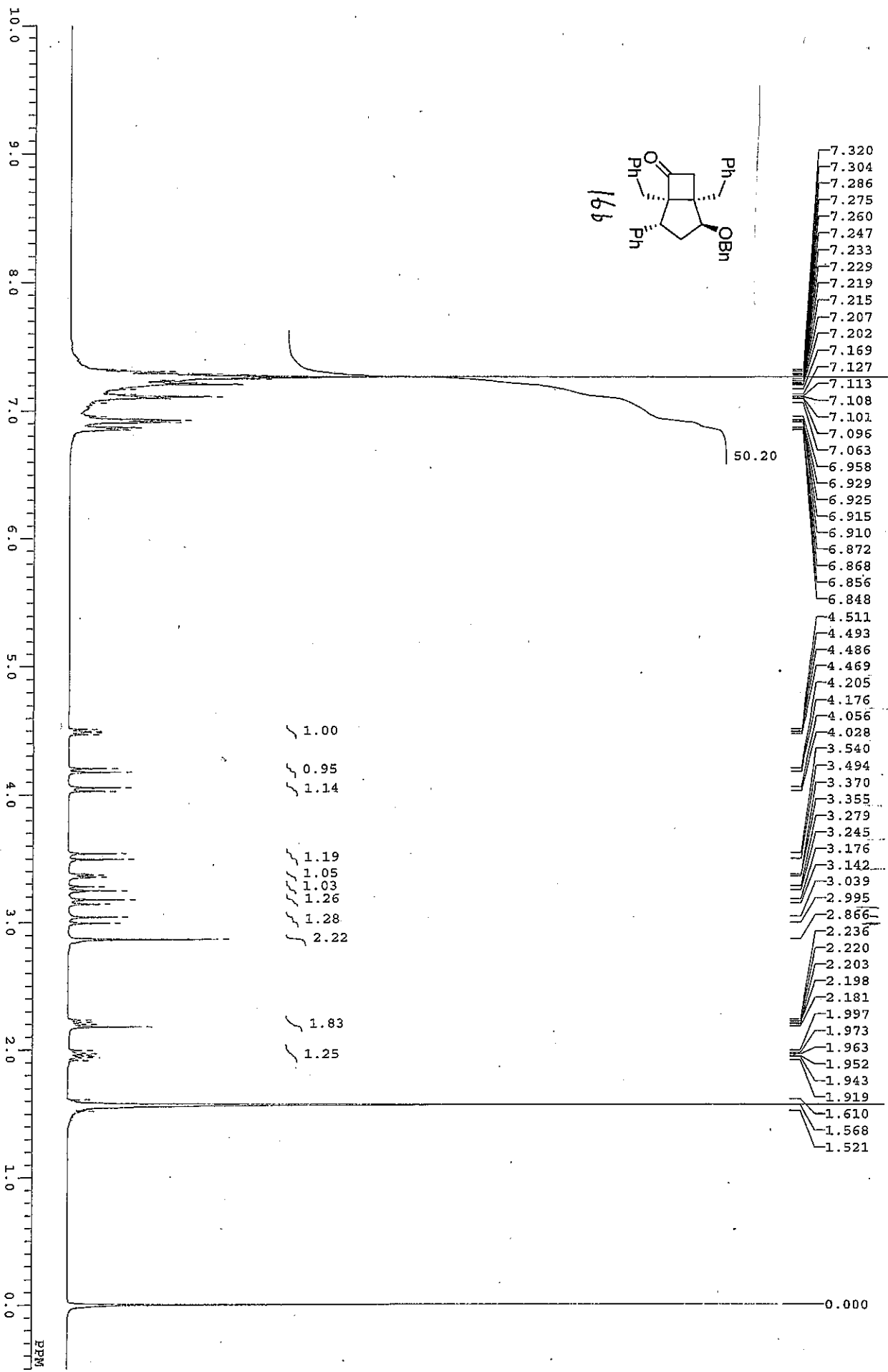
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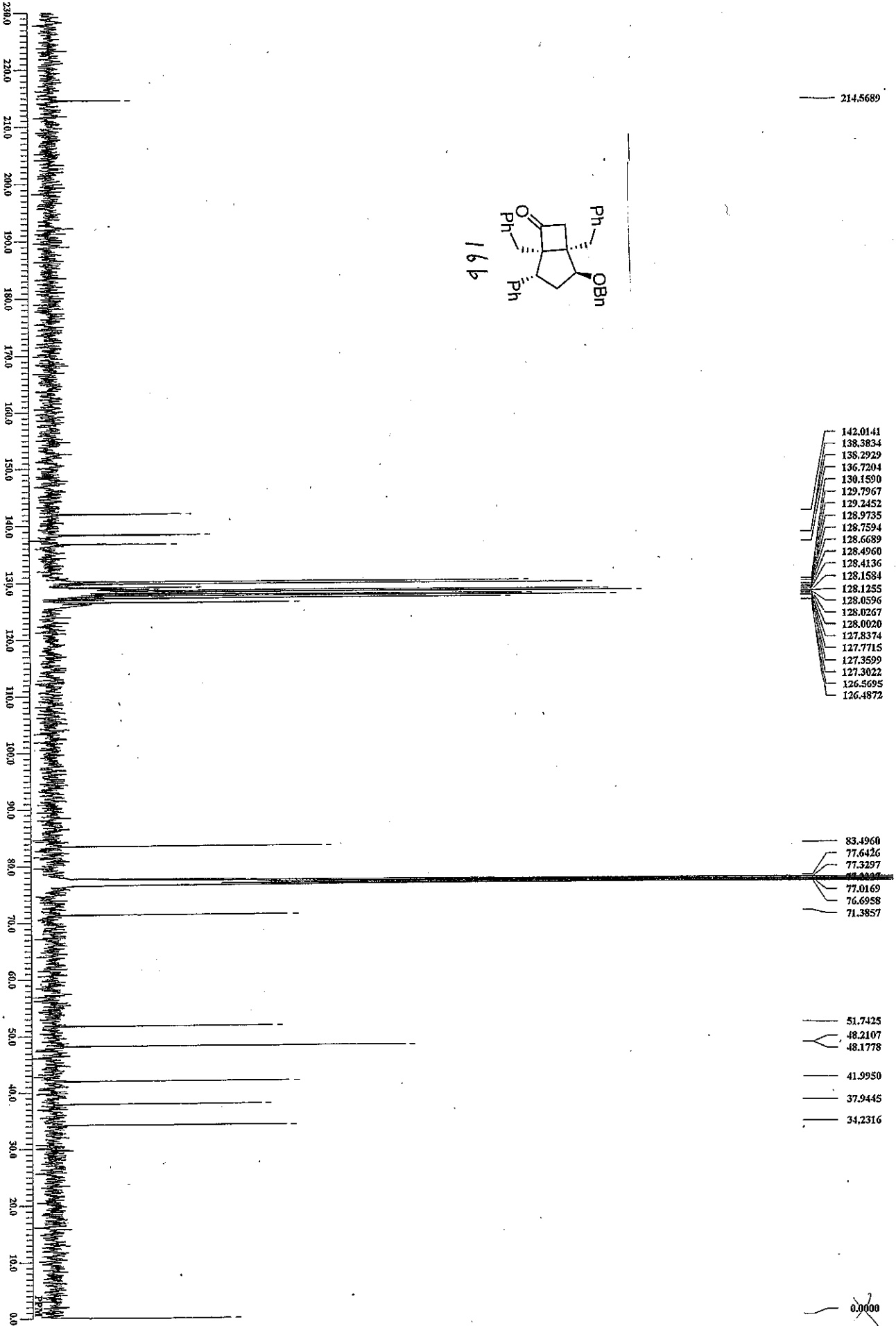
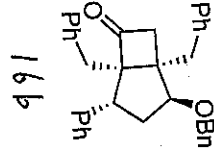
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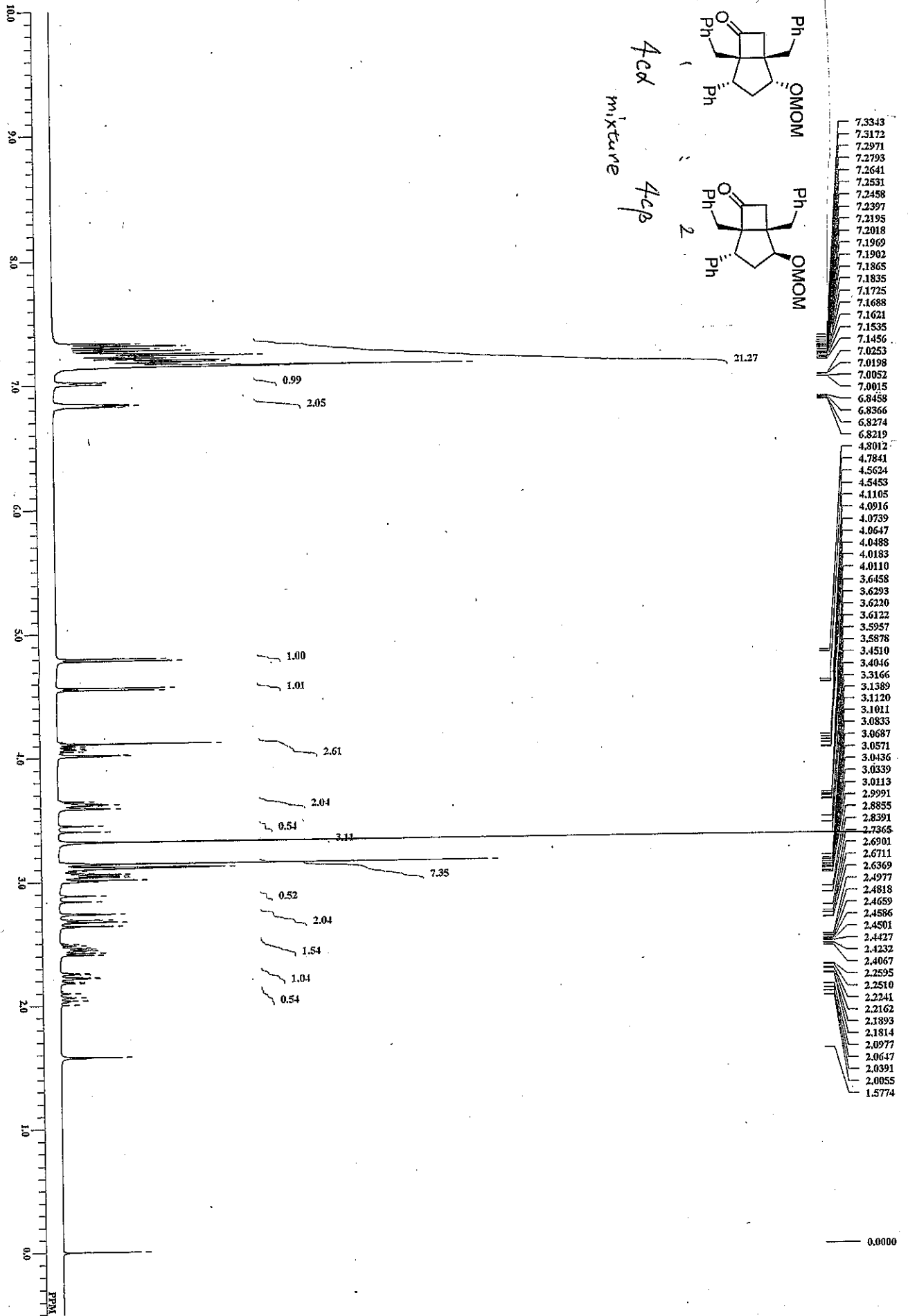
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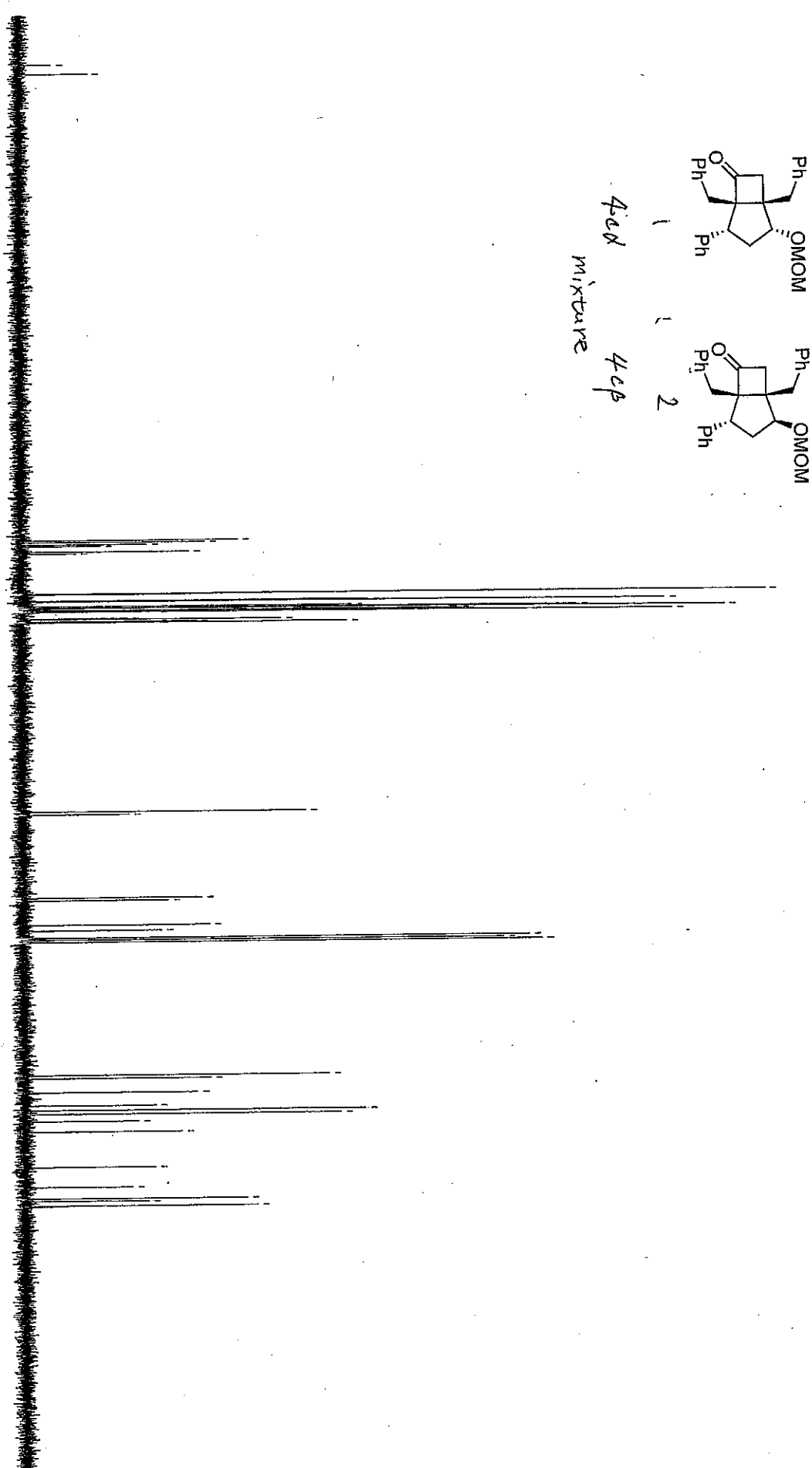






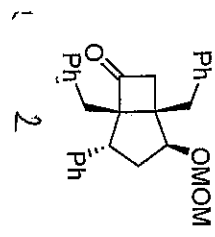
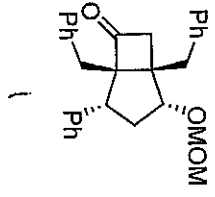


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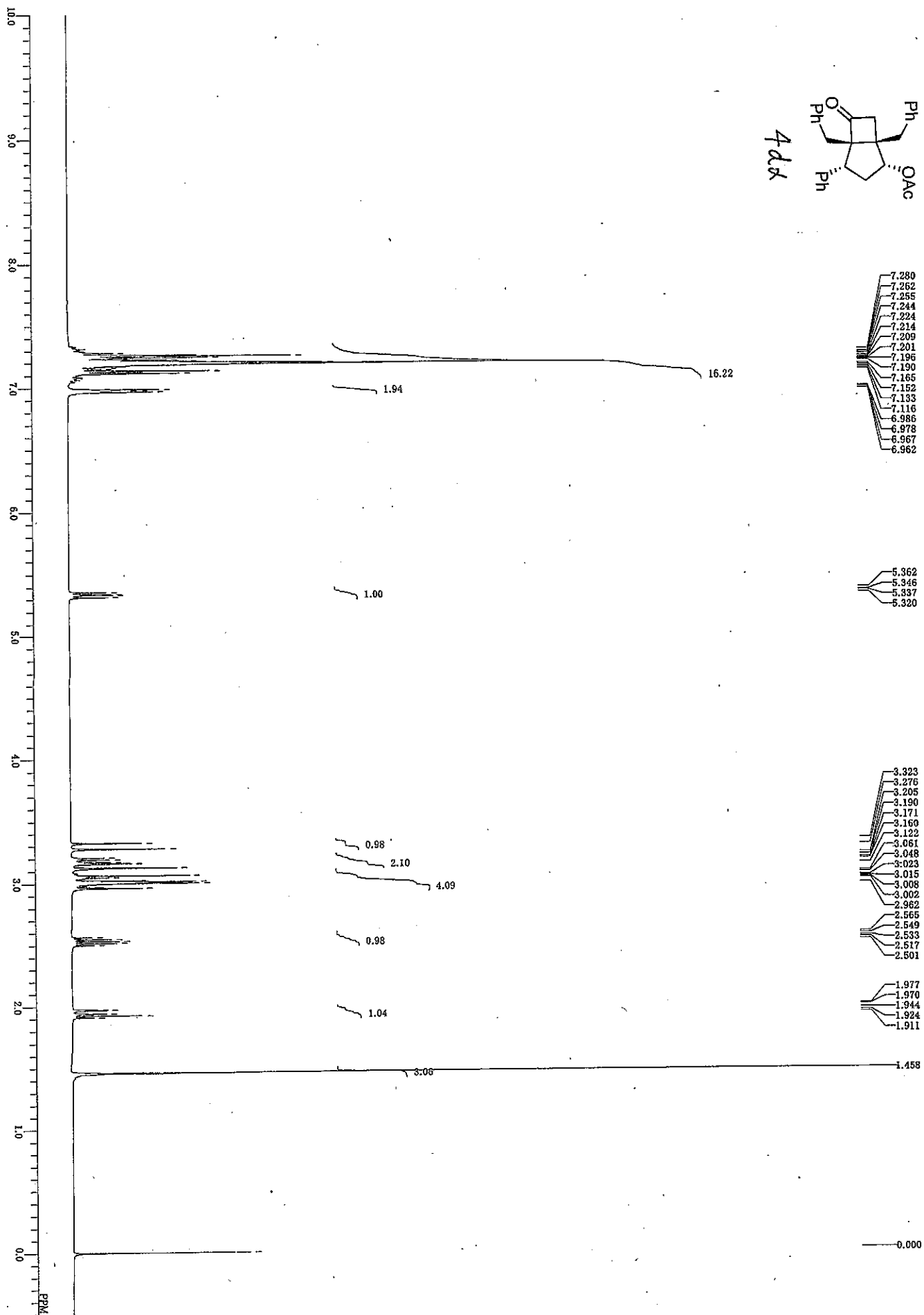
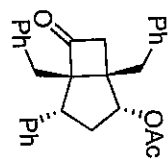


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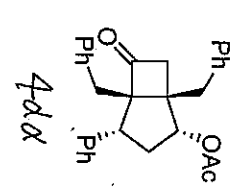
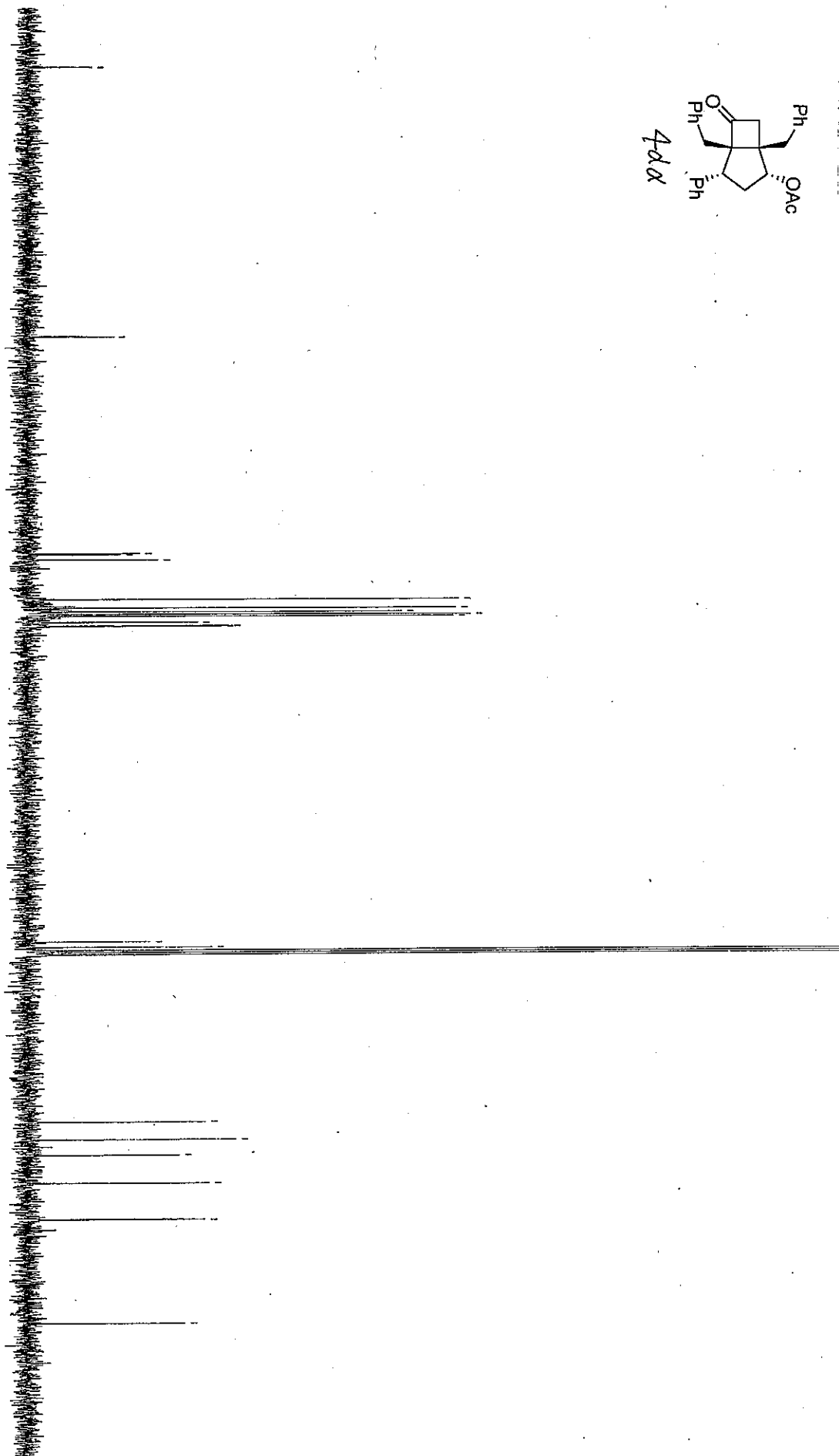
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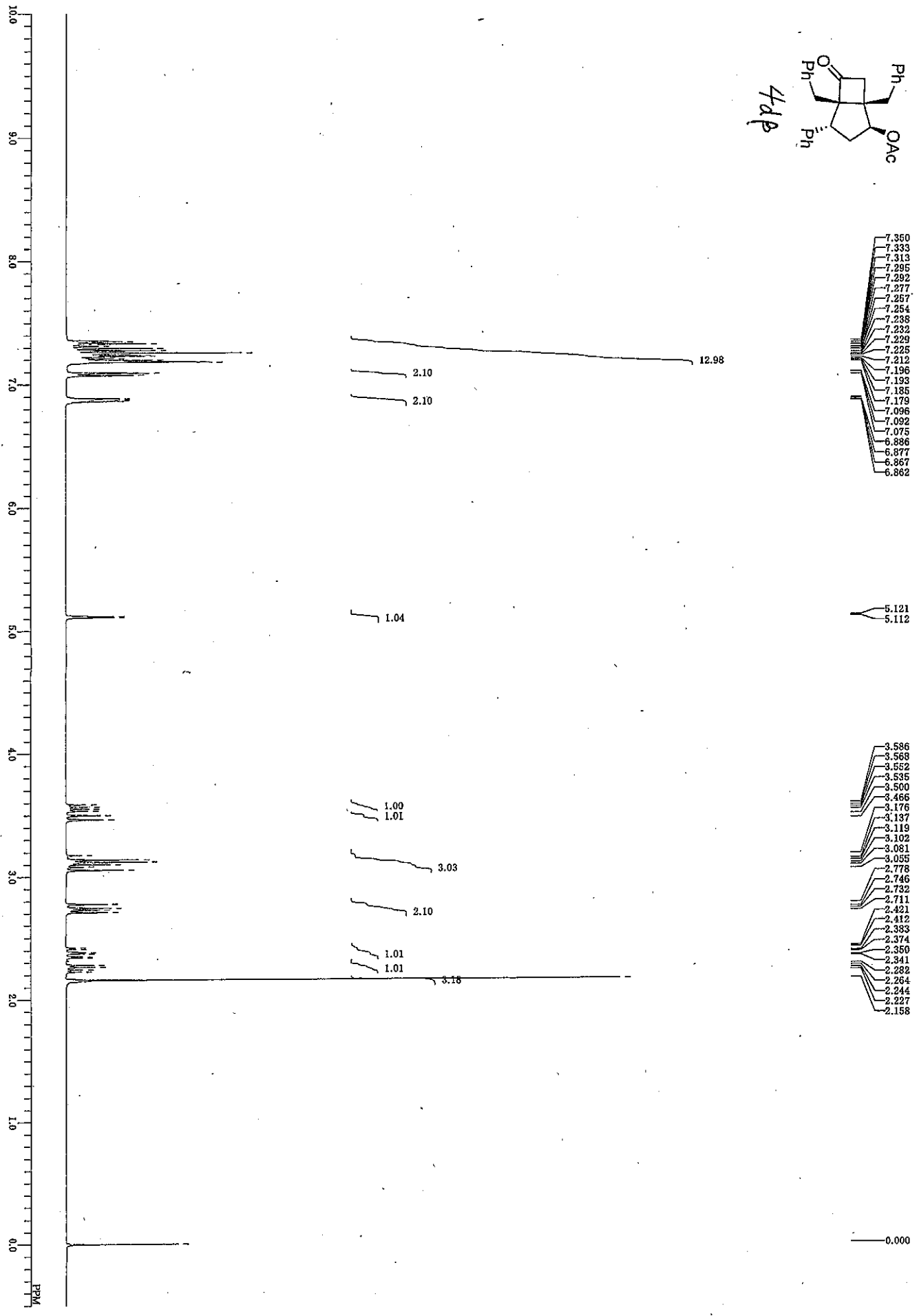
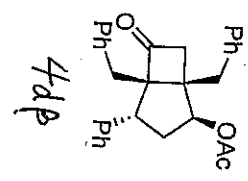
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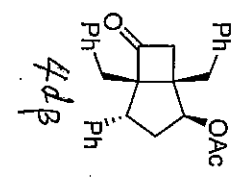
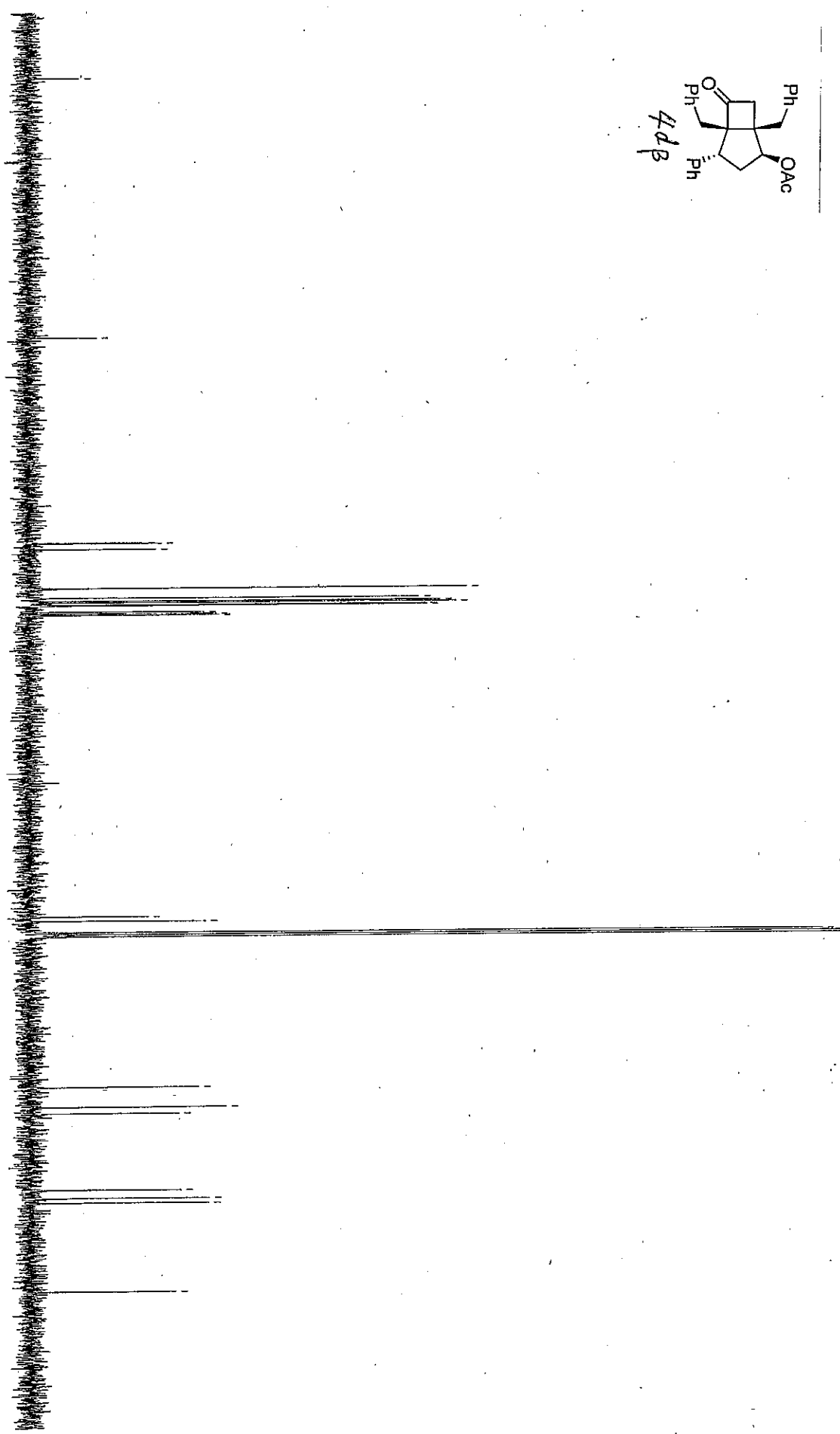
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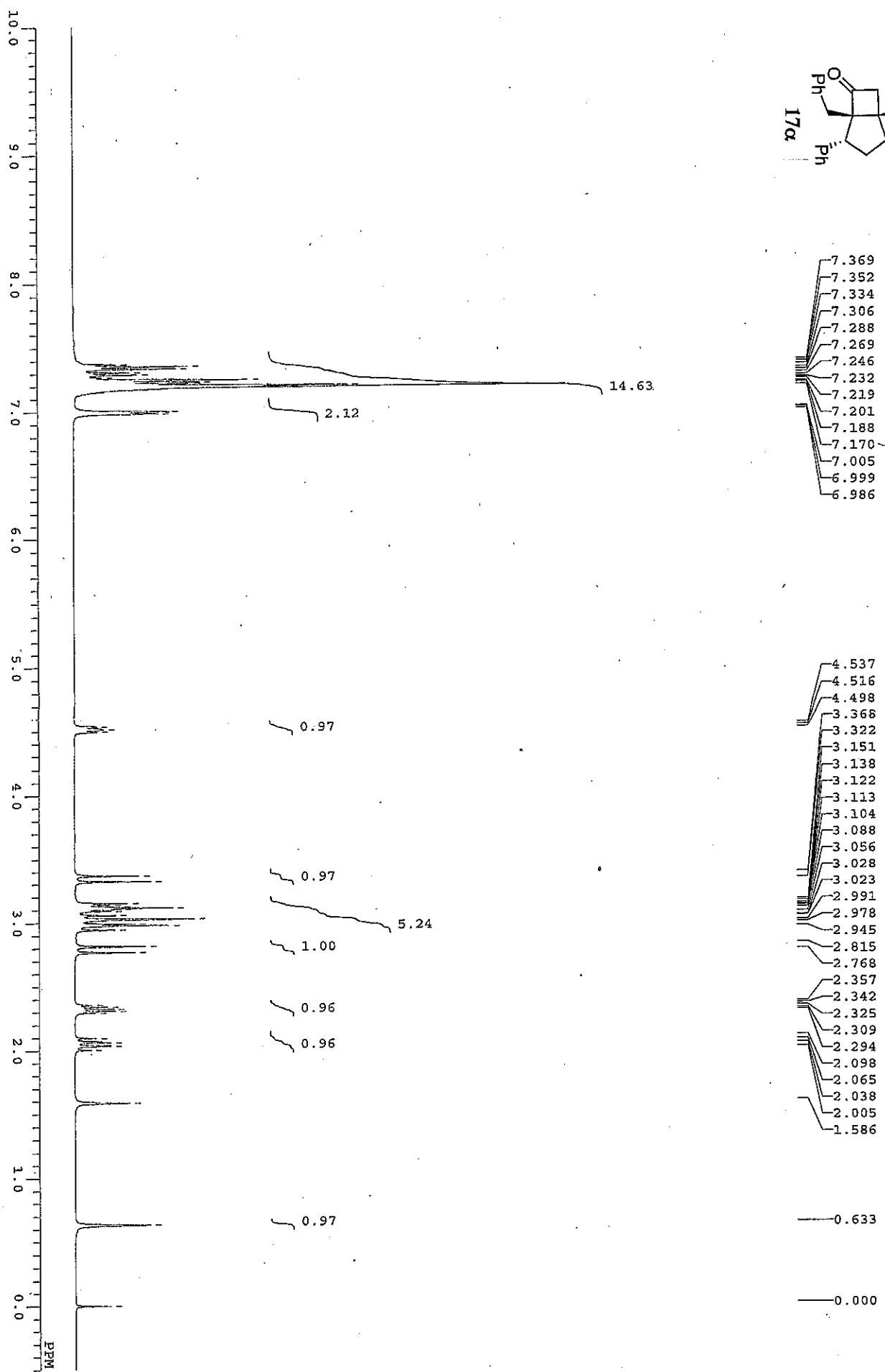
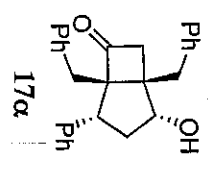
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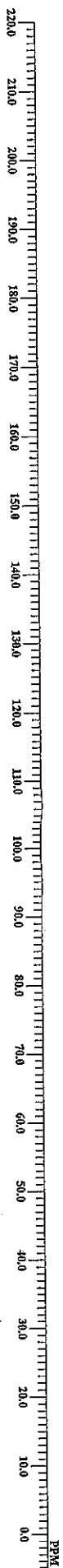
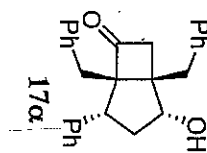
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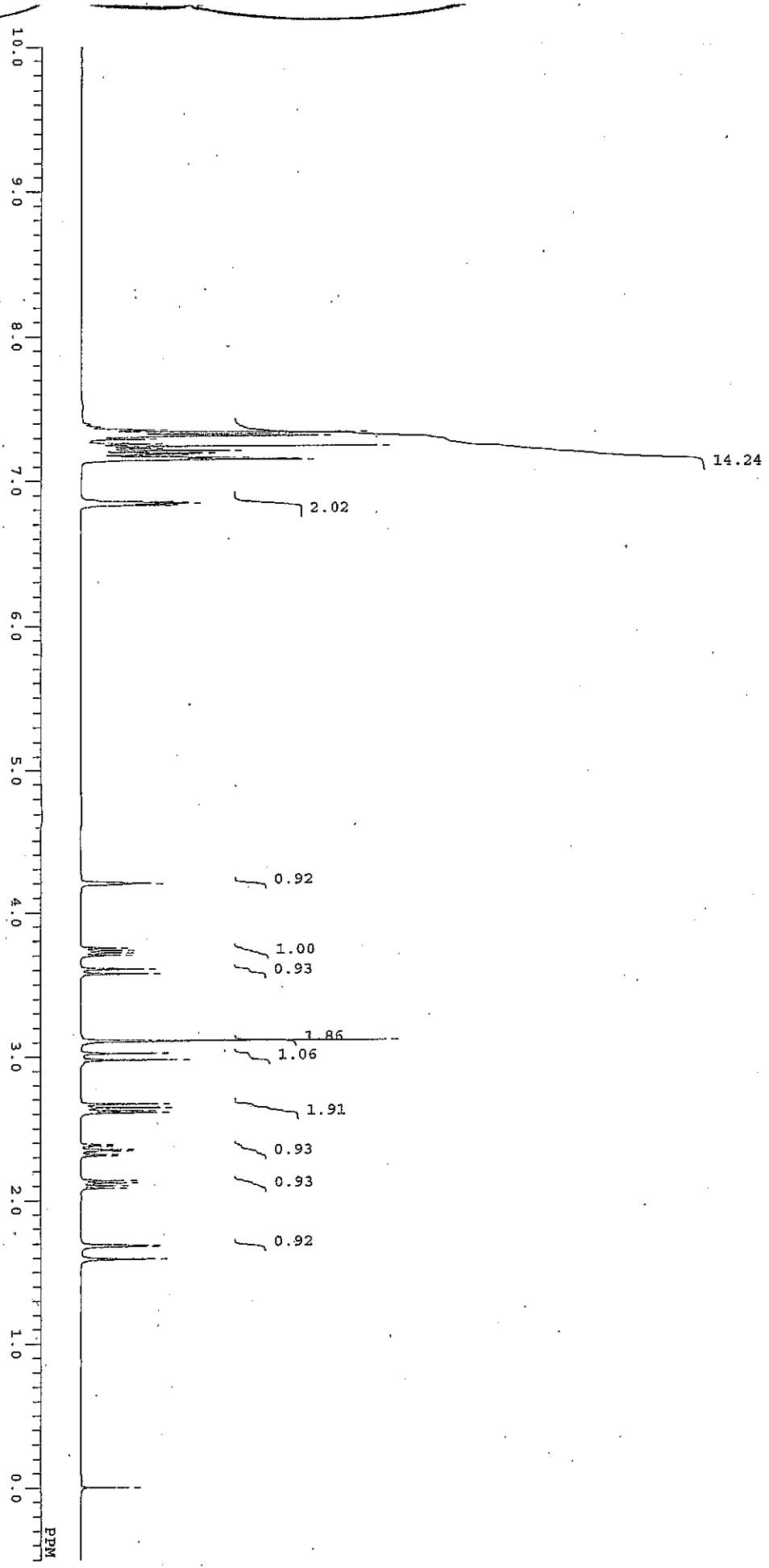
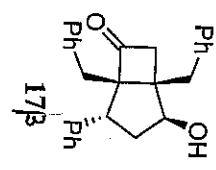


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- 126.4456

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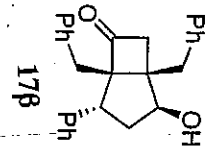
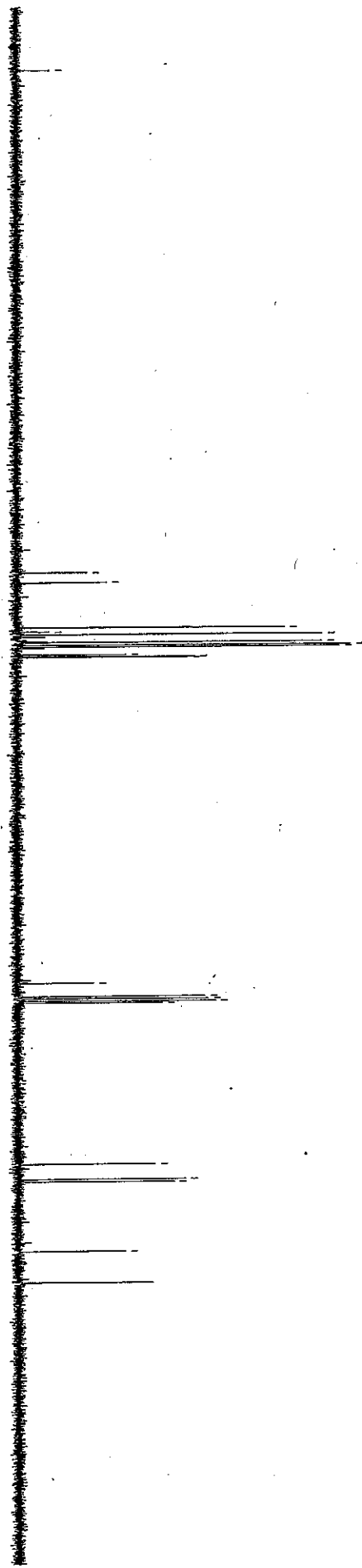


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- 6.830

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- 3.571
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- 3.020
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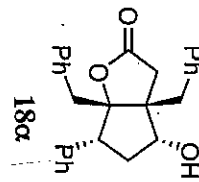
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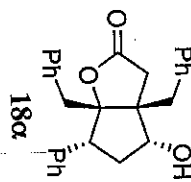
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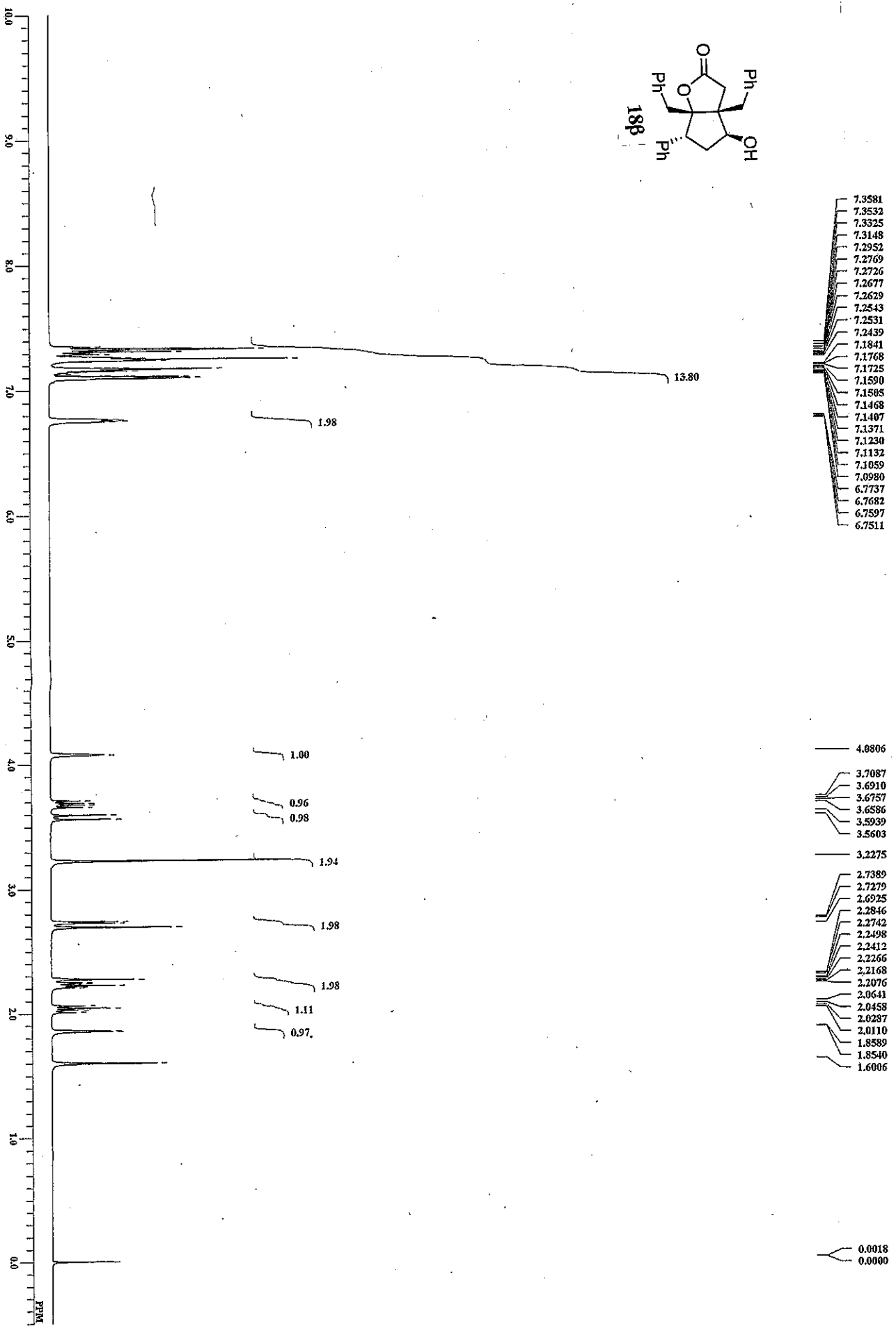
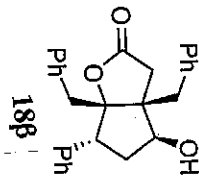
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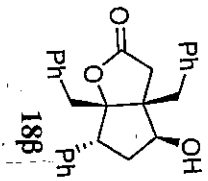
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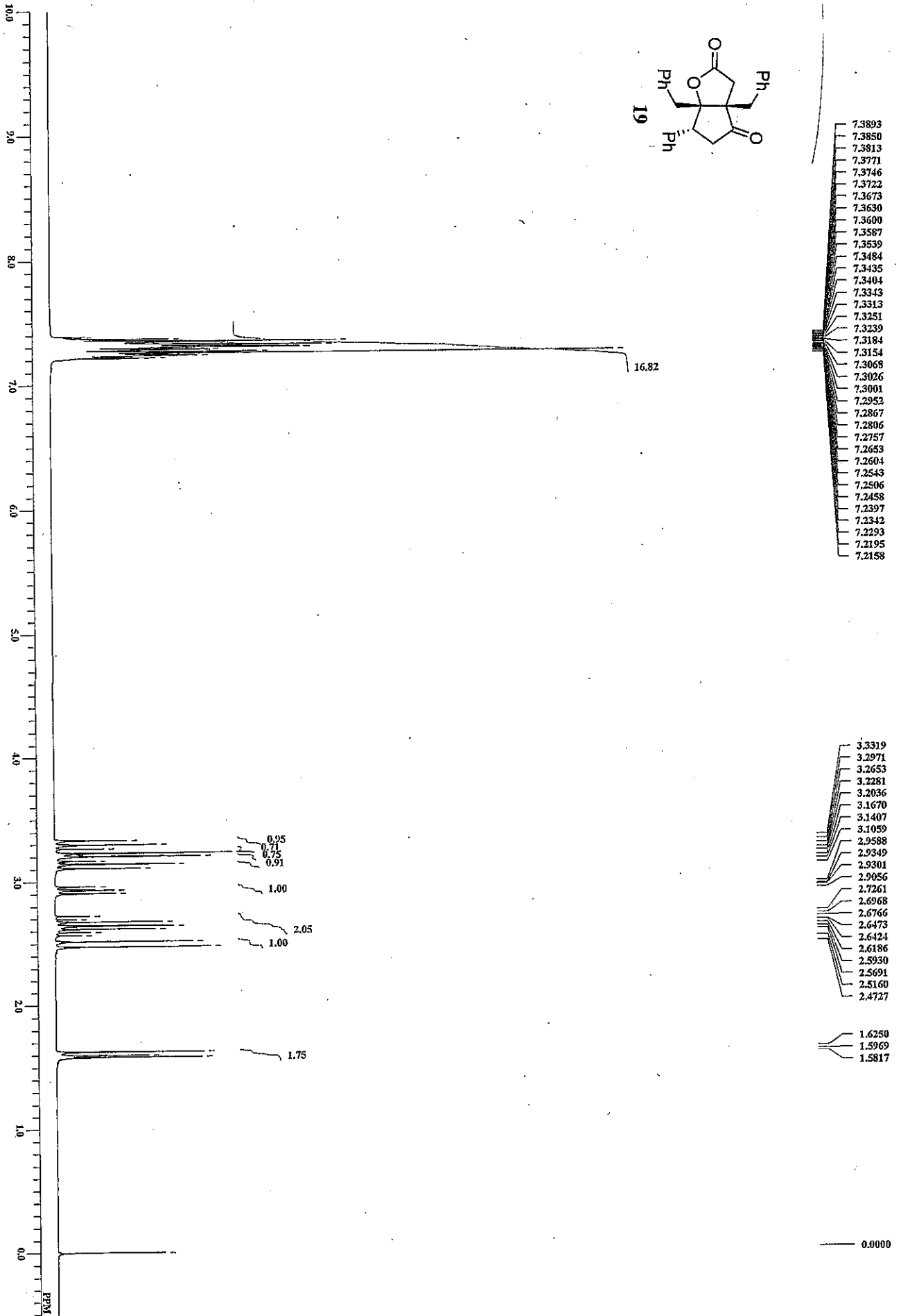
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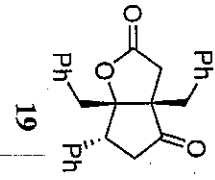
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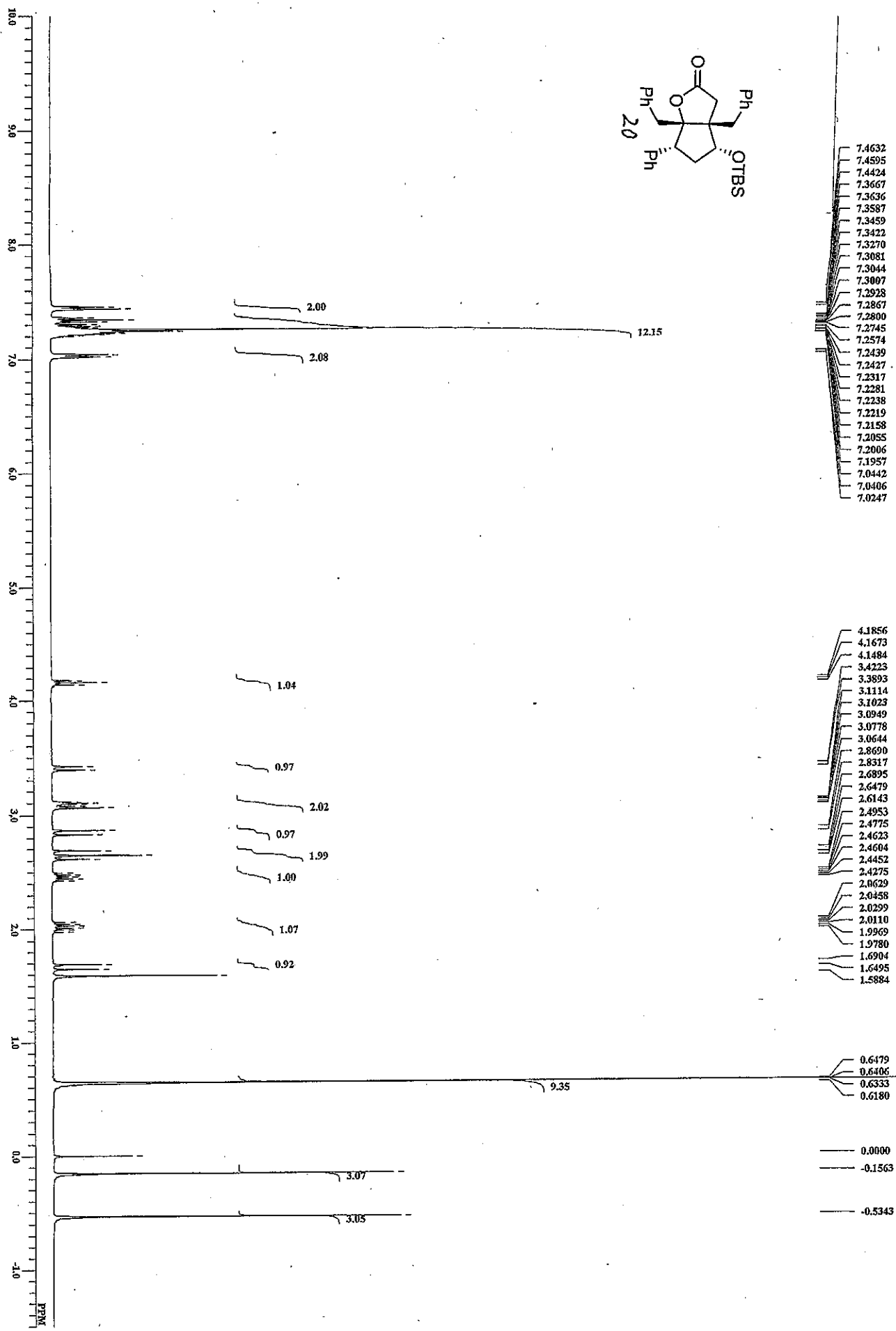
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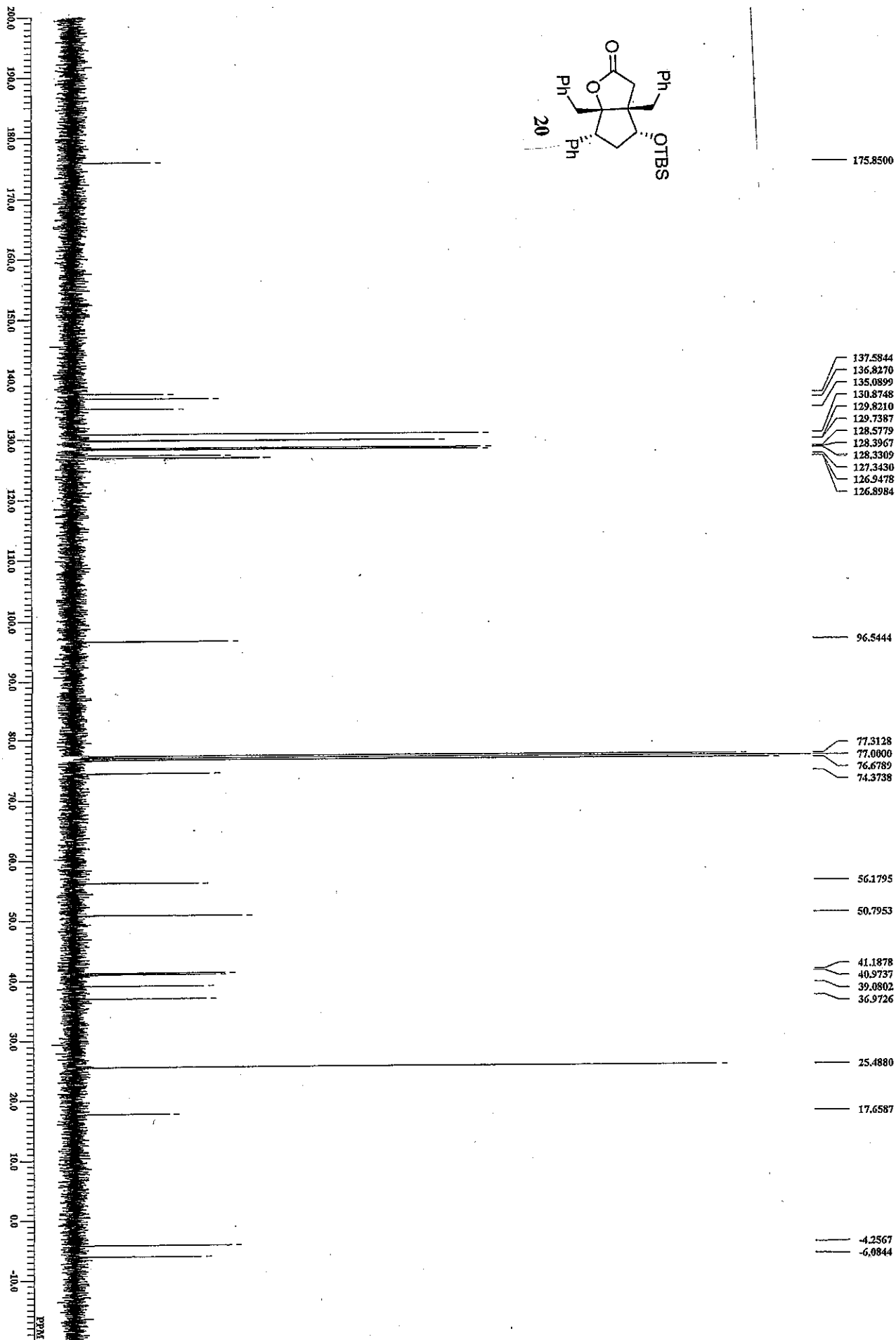
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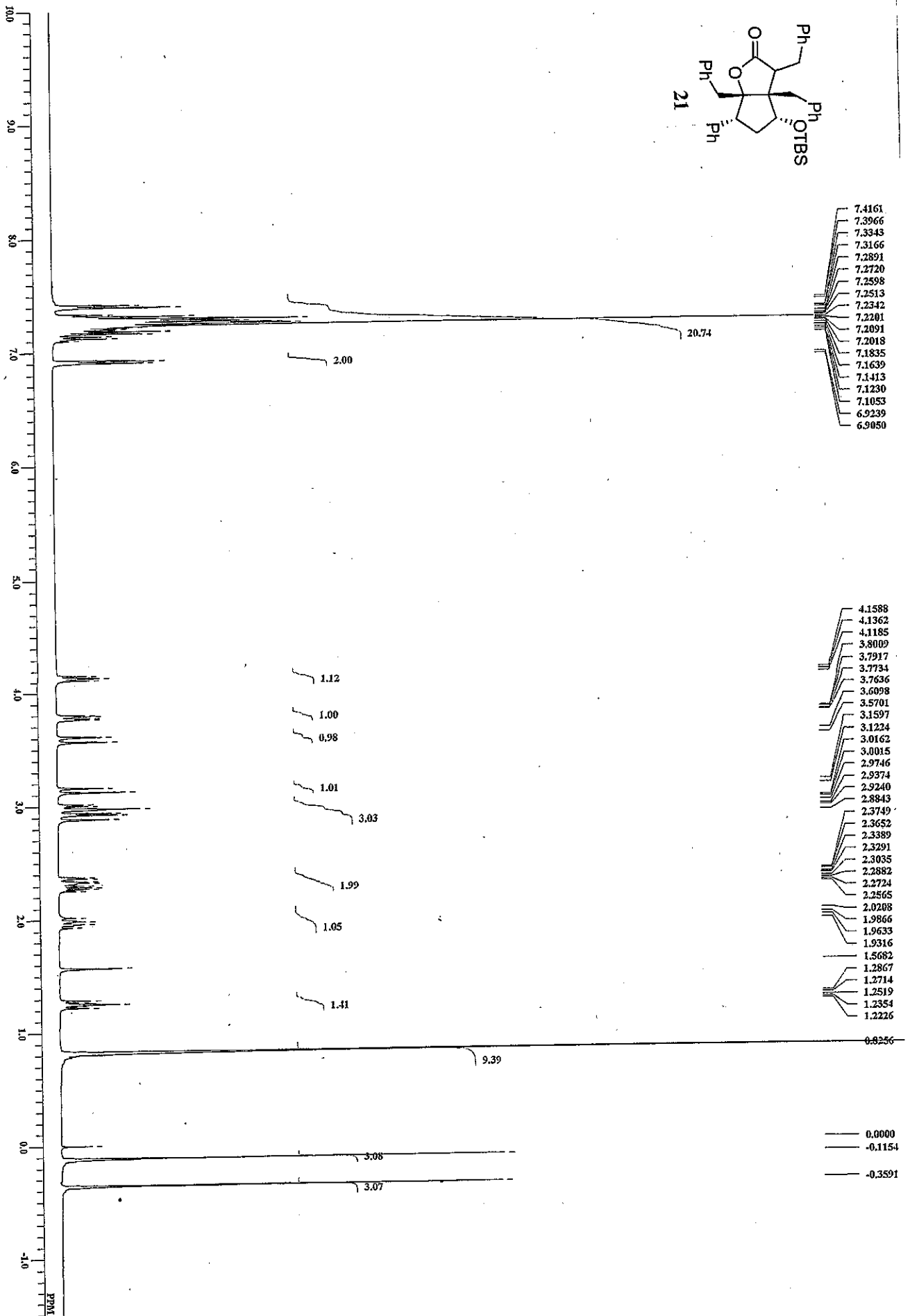
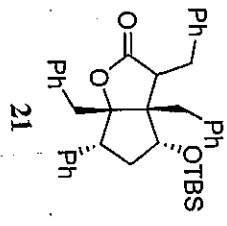
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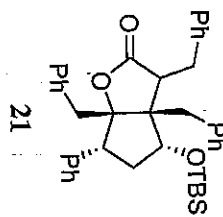
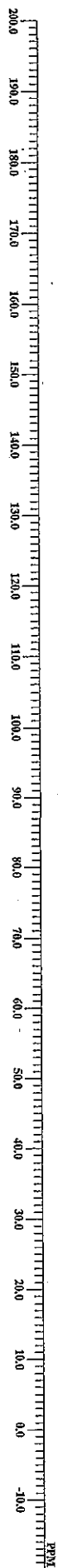
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- 128.8495
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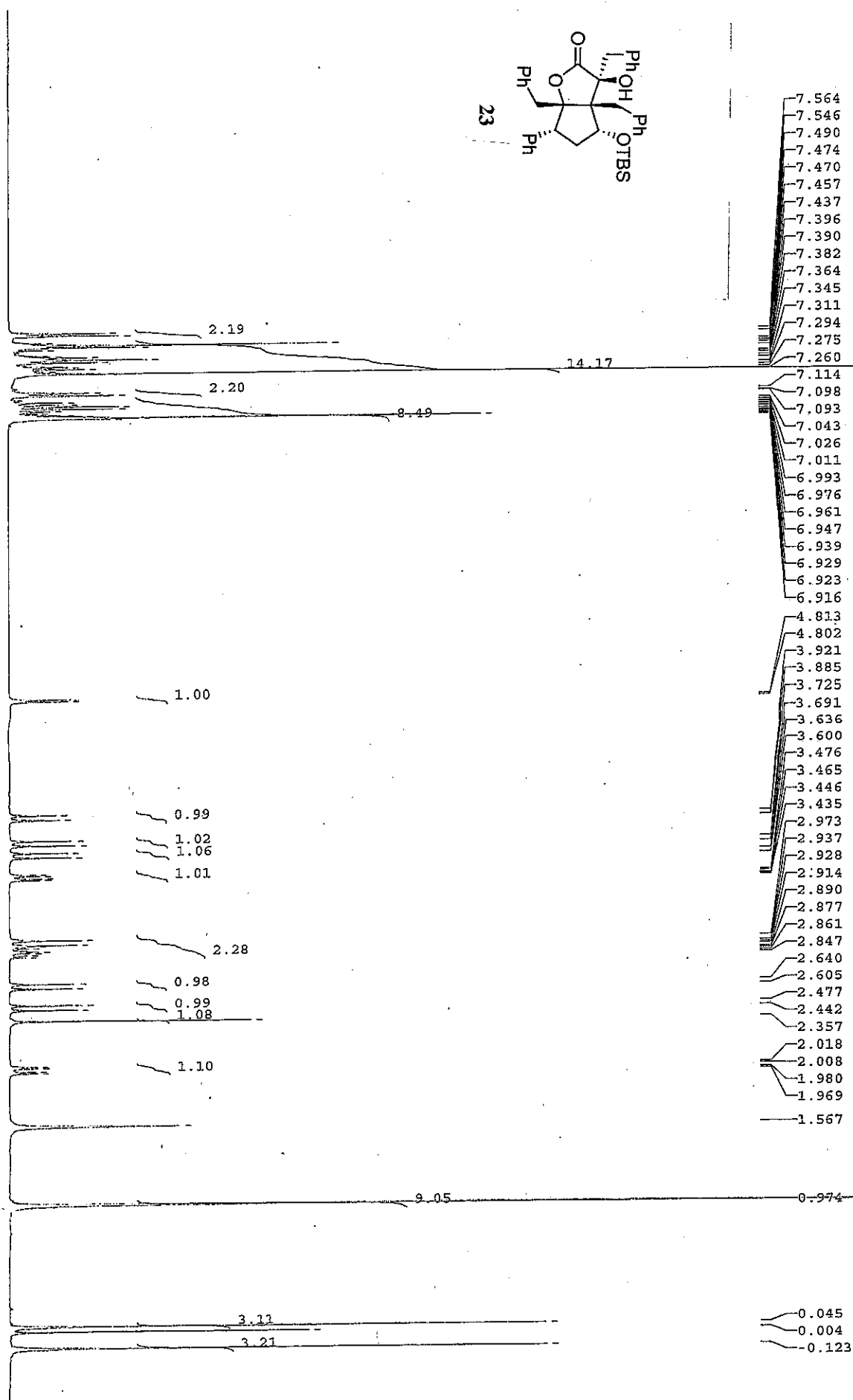
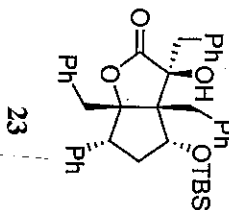
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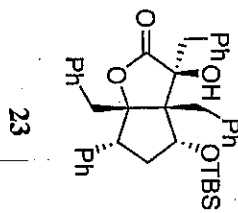
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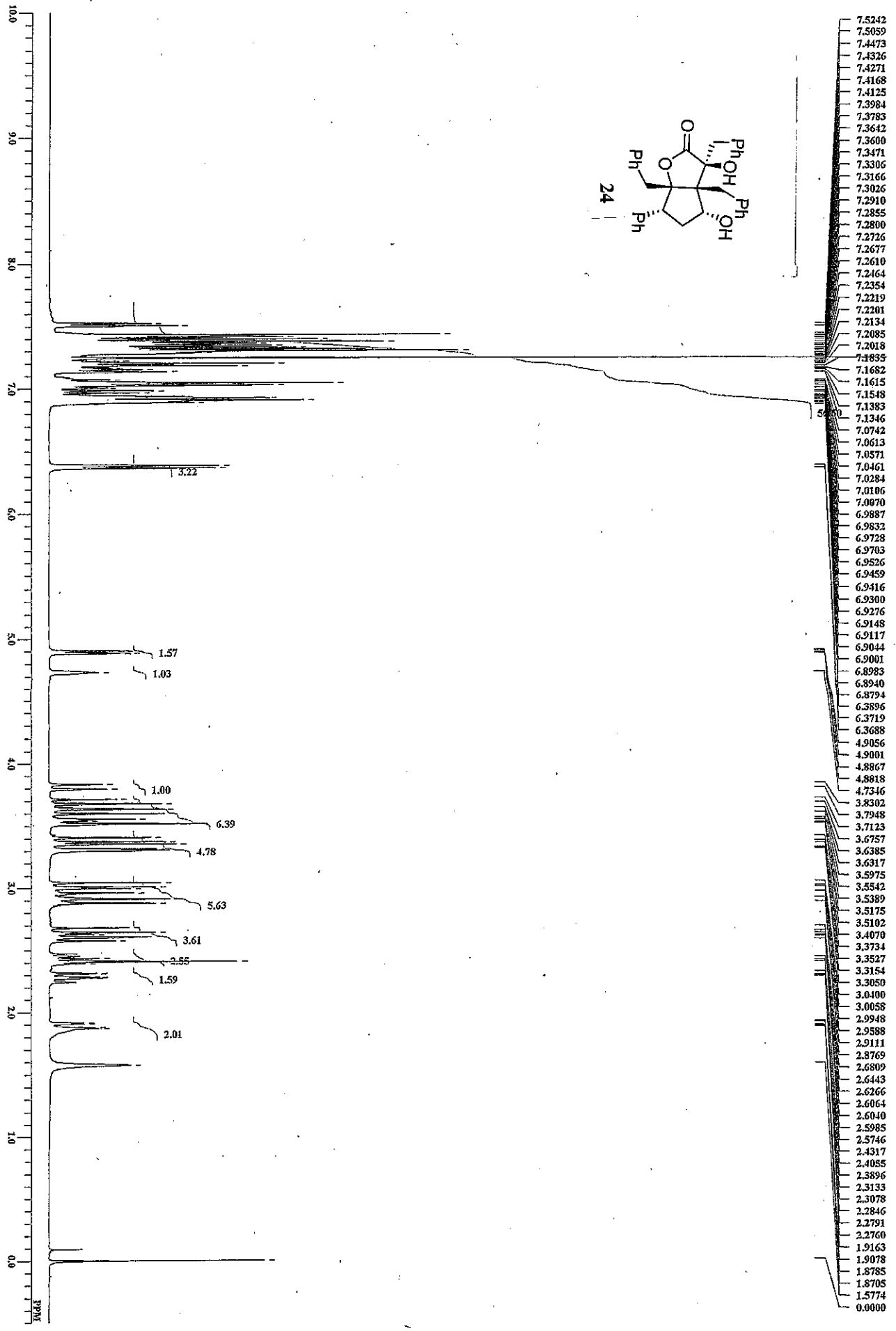
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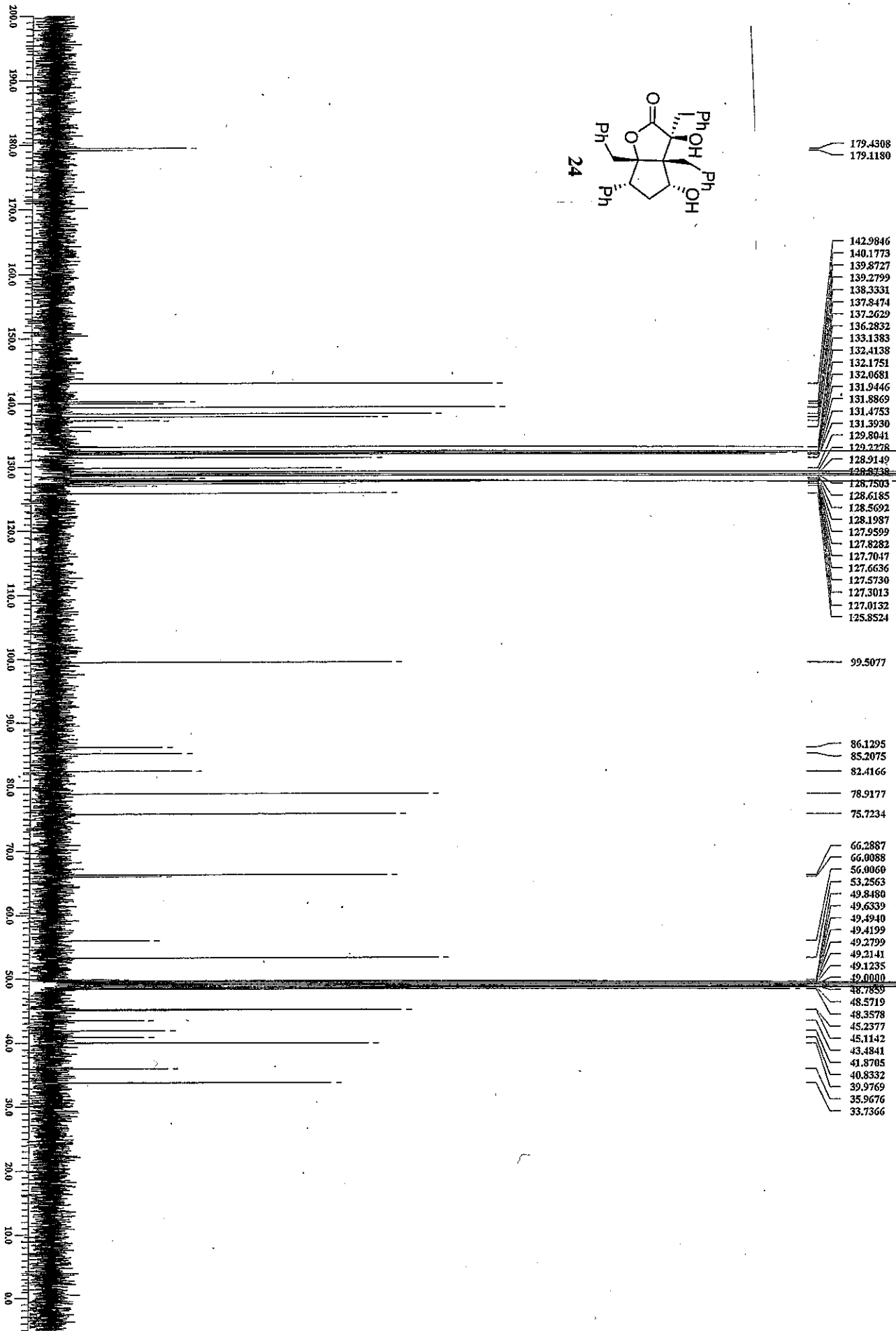


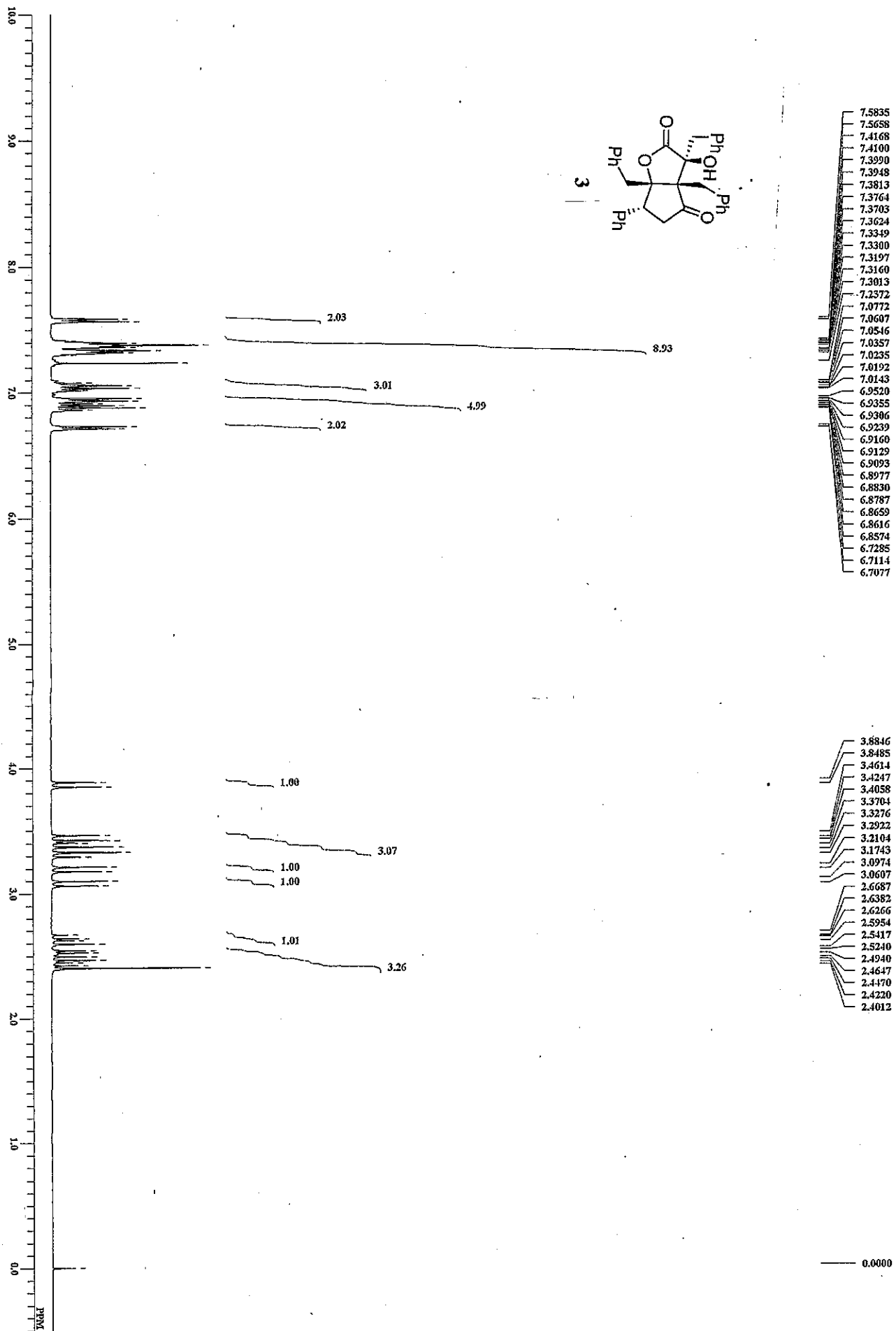
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- 7.311
- 7.294
- 7.275
- 7.260
- 7.114
- 7.098
- 7.093
- 7.043
- 7.026
- 7.011
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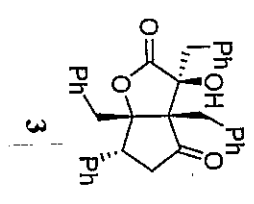
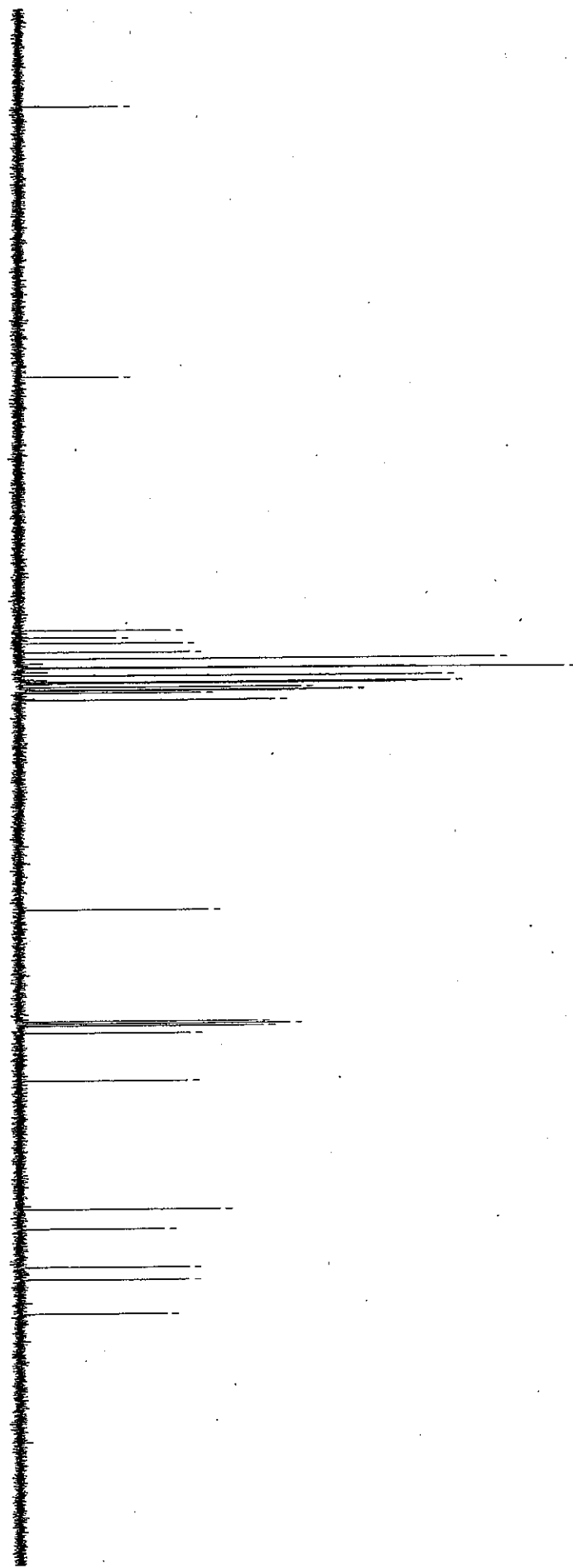
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- 128.5614
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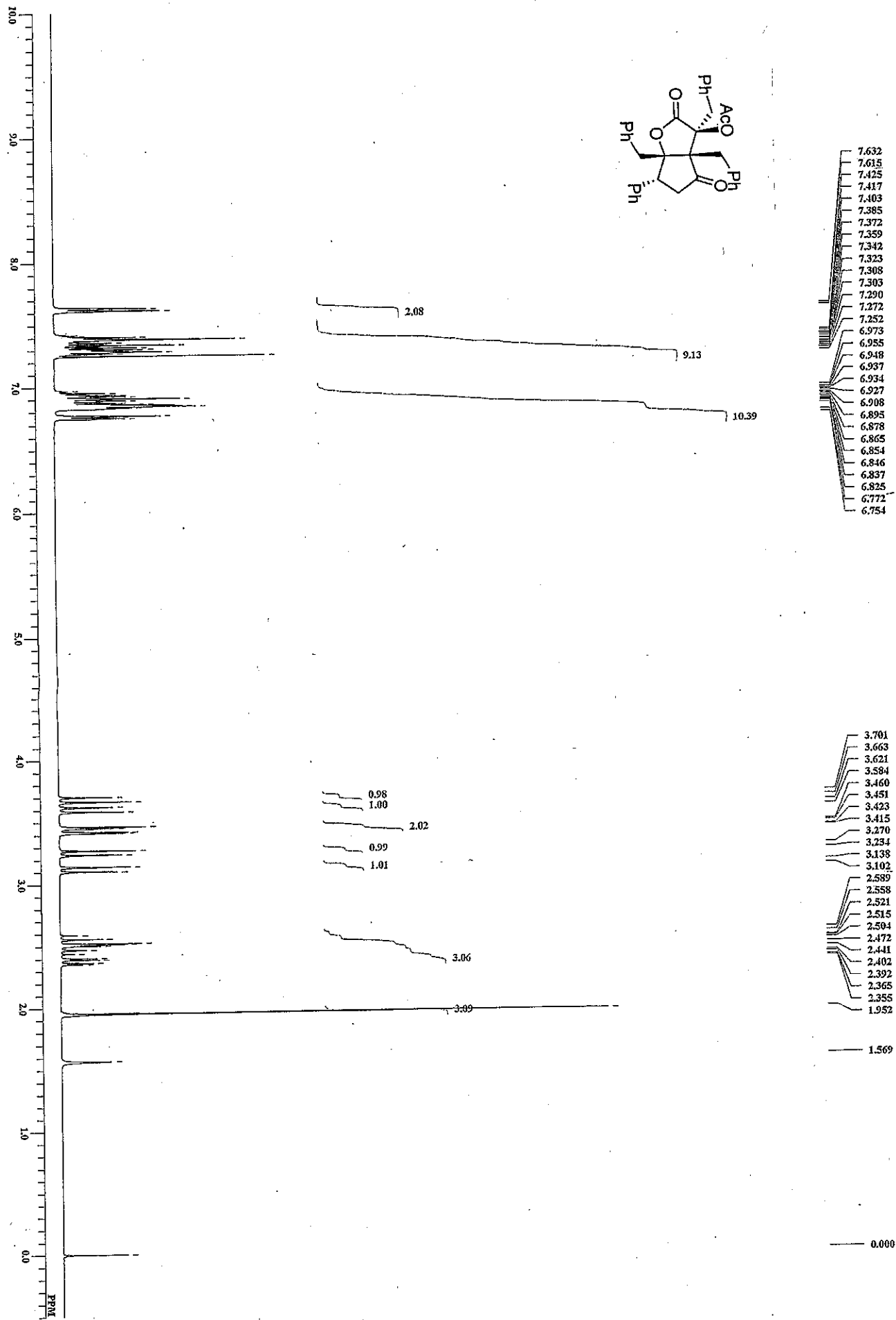




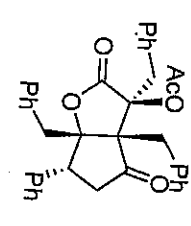
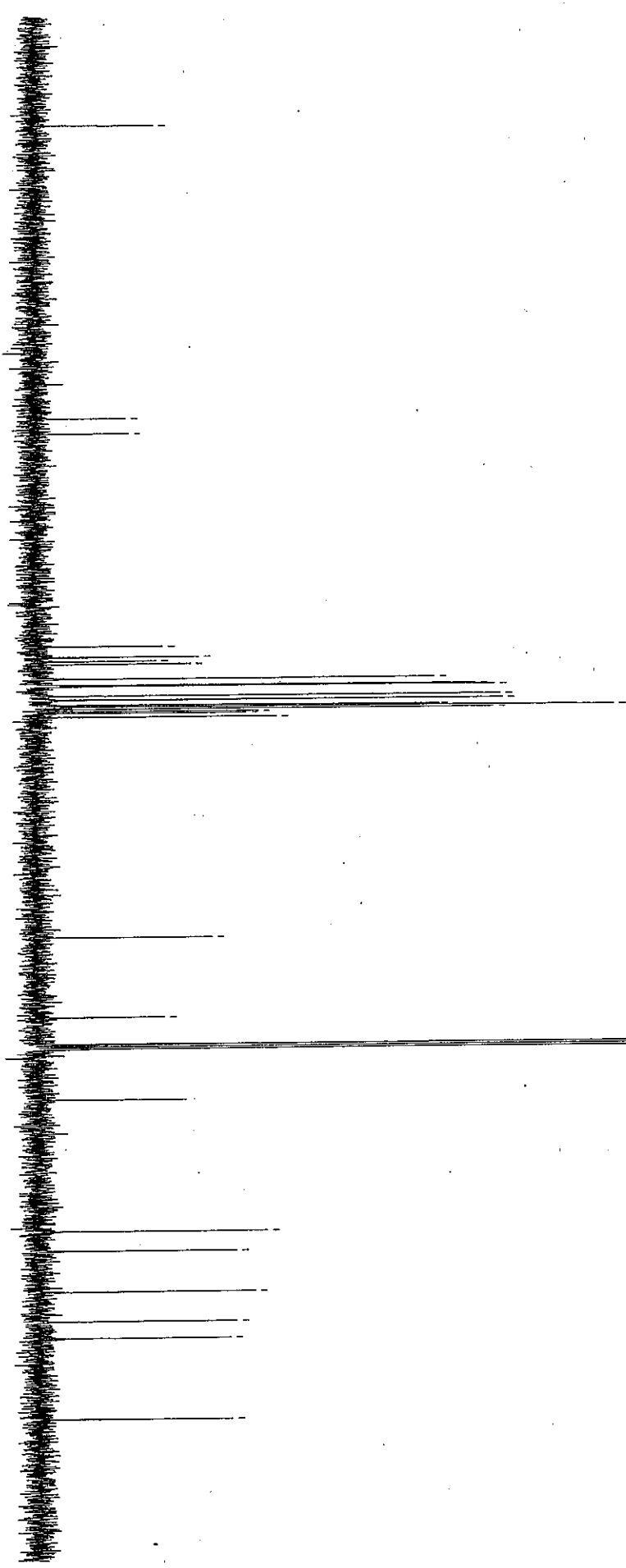
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