

Synthesis of Fused Dibenzofuran Derivatives via Palladium-catalyzed Domino C-C Bond formation and Iron-catalyzed Cycloisomerization/Aromatization

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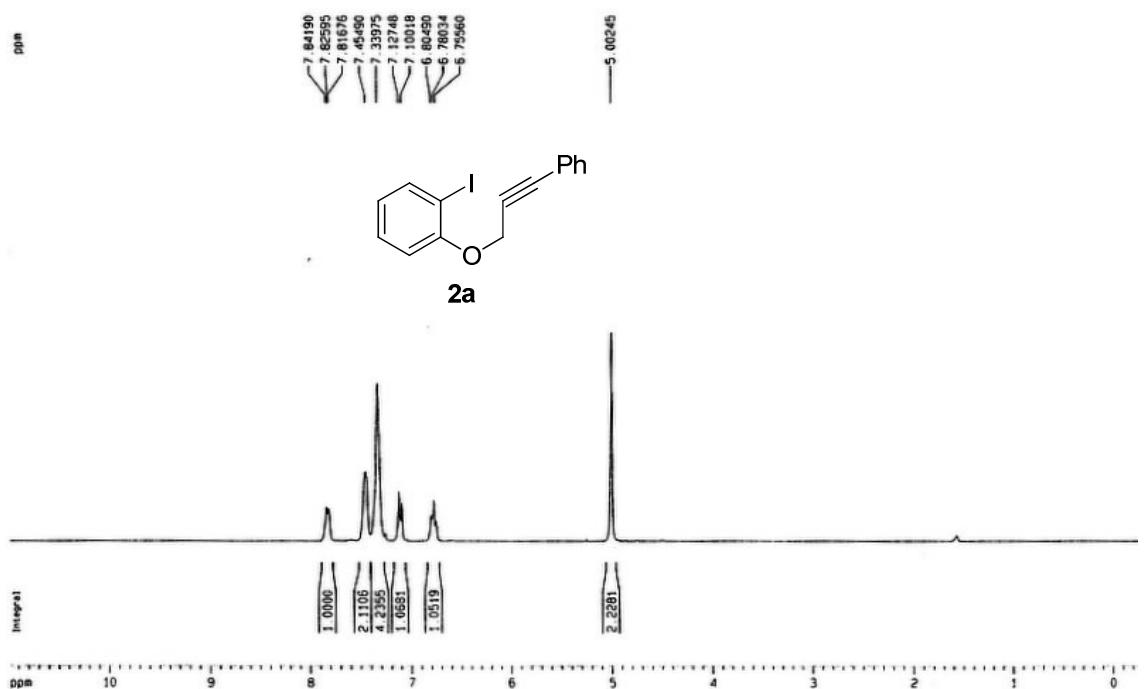
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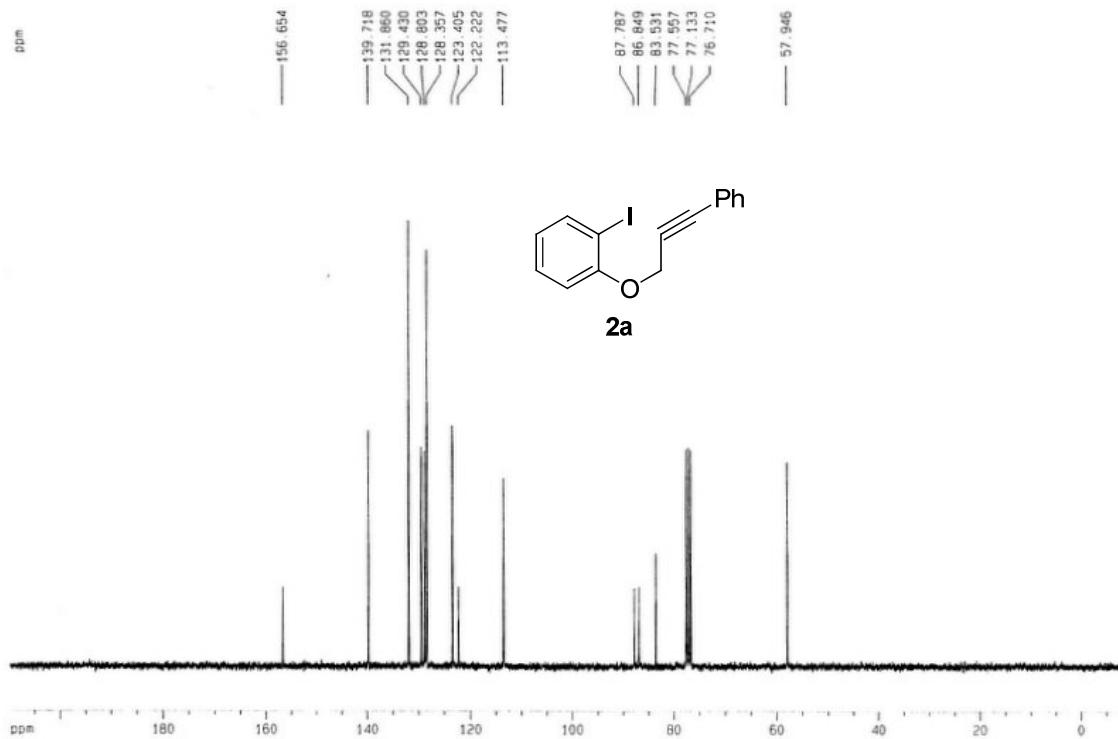
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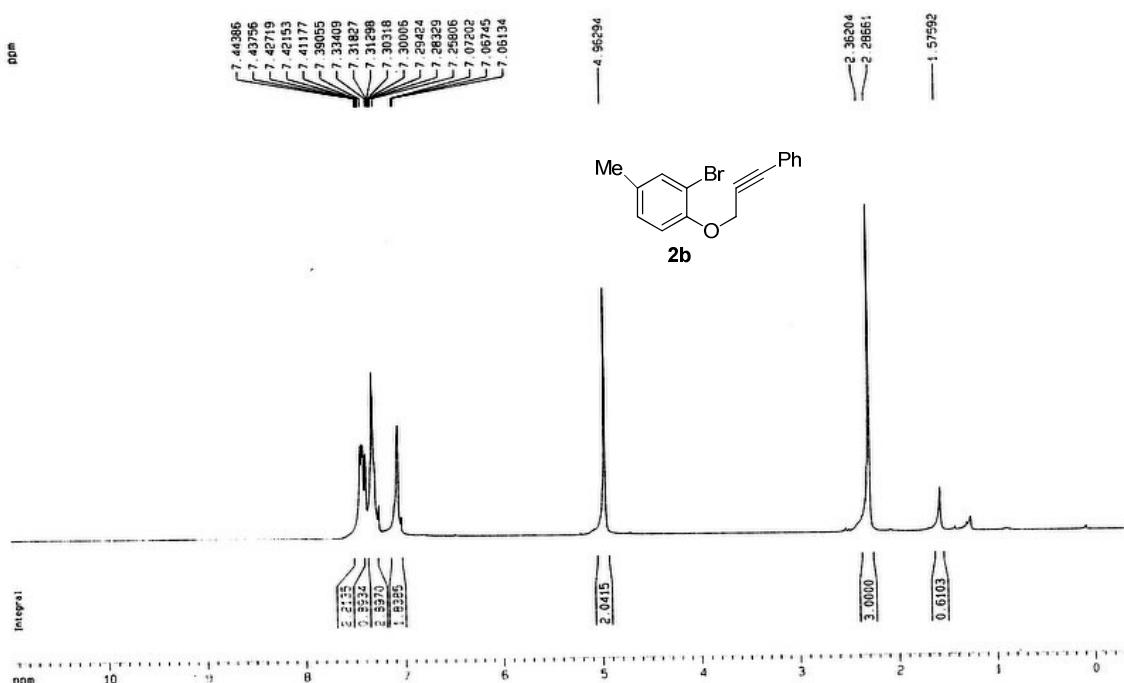
¹H NMR of **2a**, CDCl₃, 300 MHz



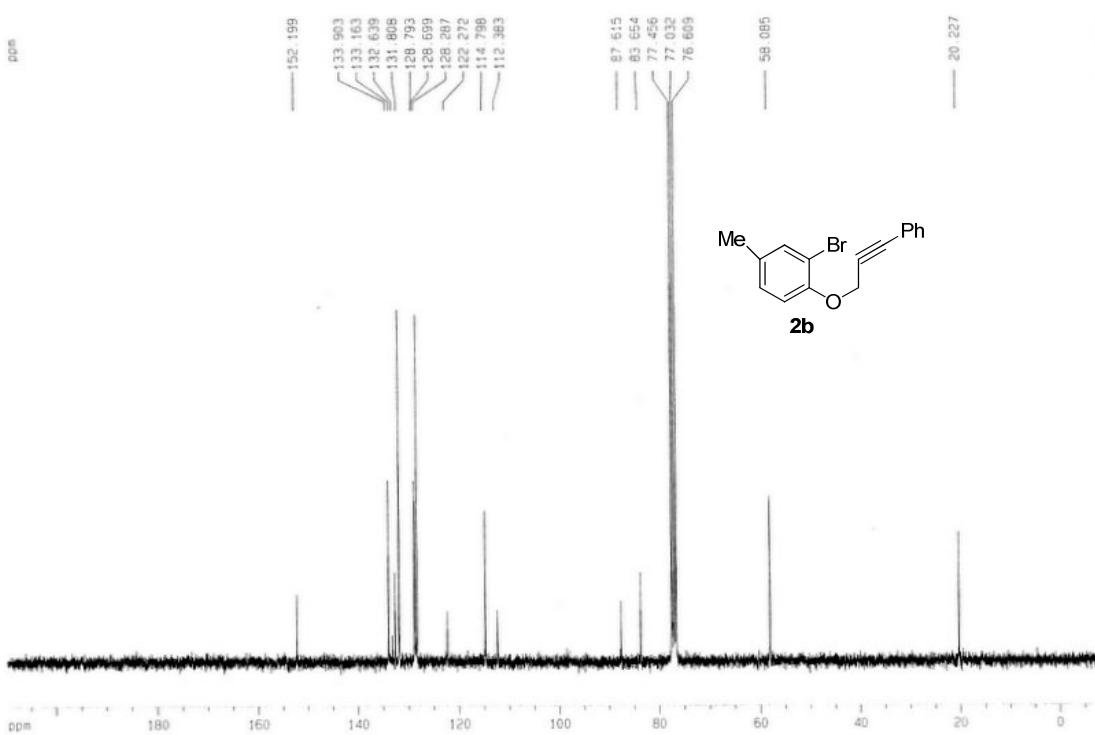
¹³C NMR of **2a**, CDCl₃, 75 MHz



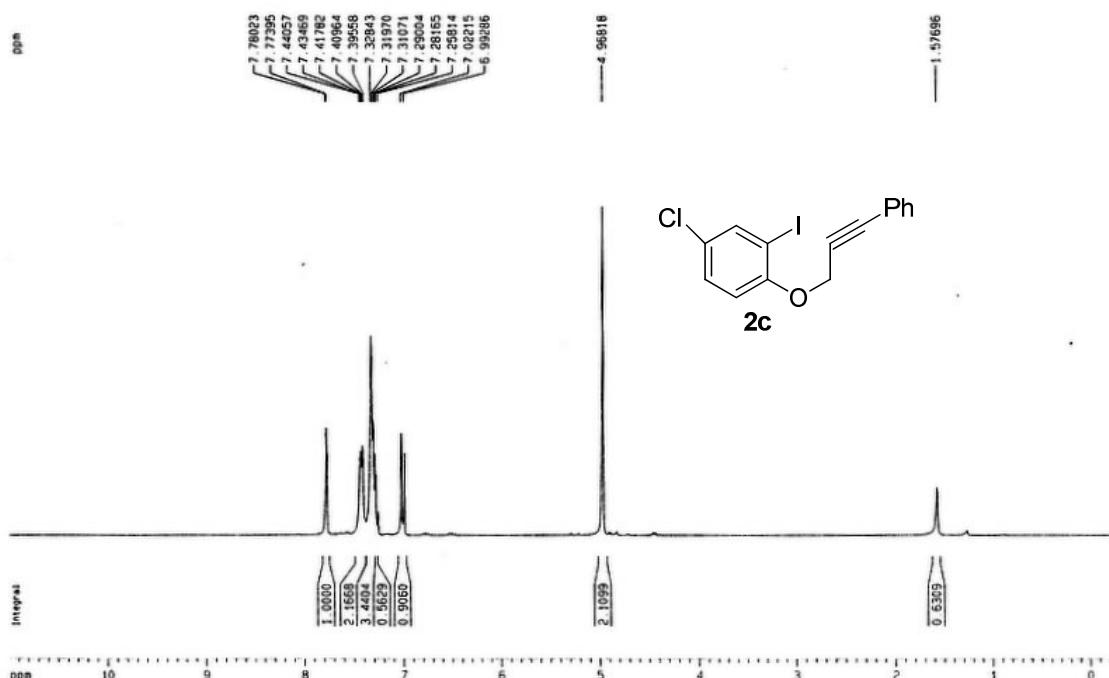
¹H NMR of **2b**, CDCl₃, 300 MHz



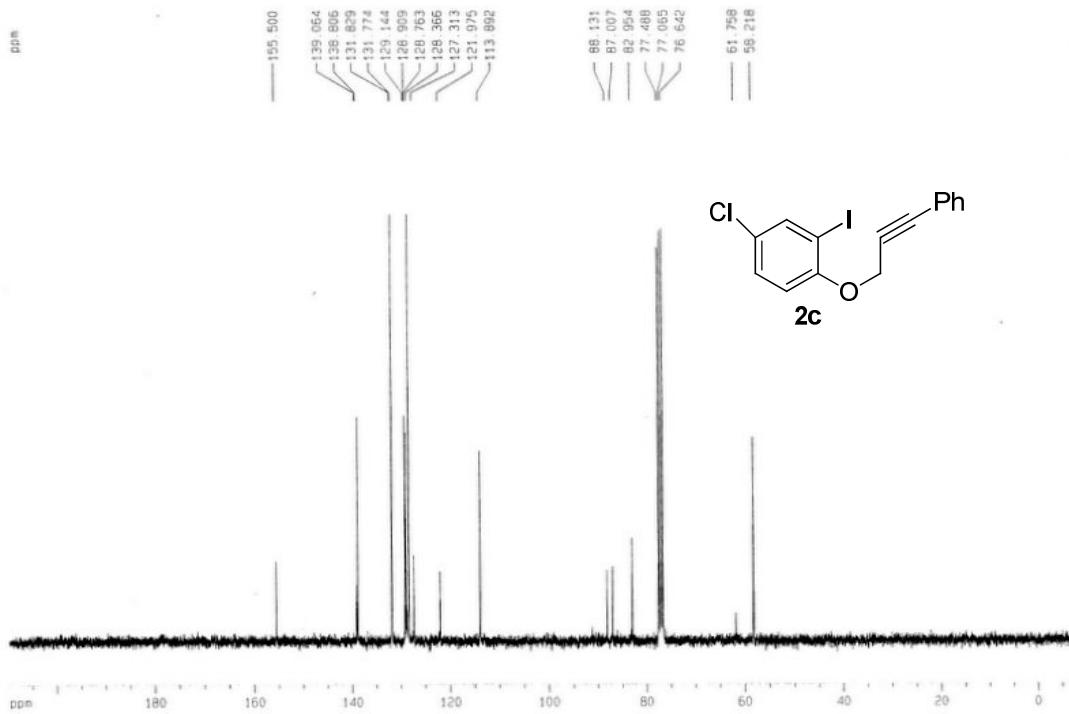
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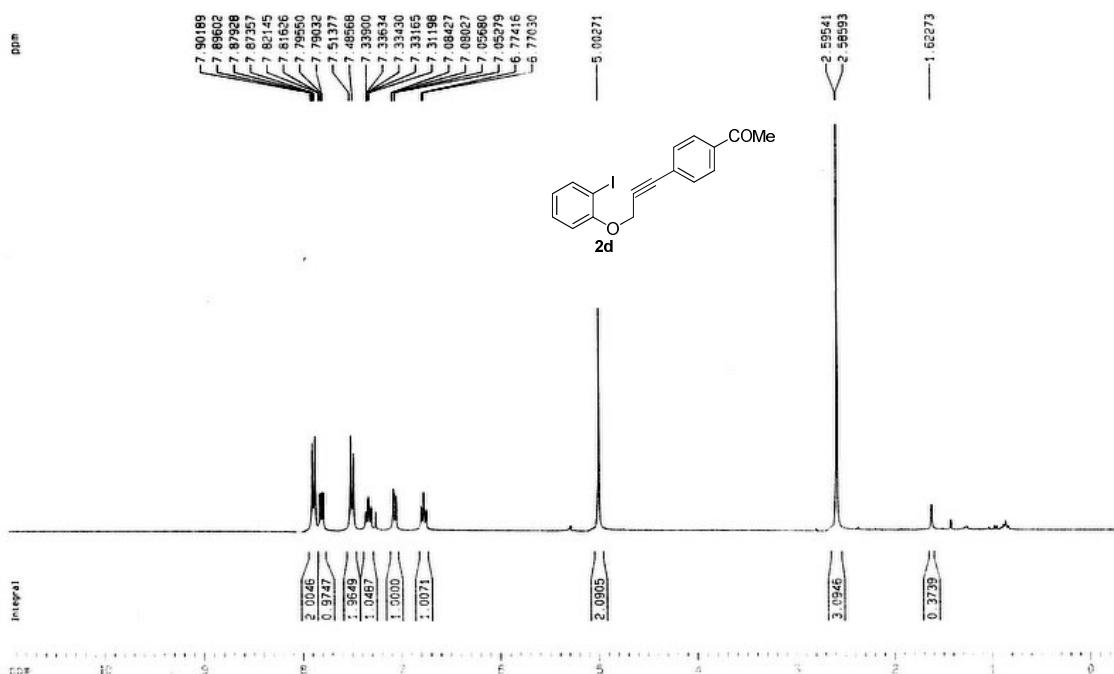
¹H NMR of **2c**, CDCl₃ 300 MHz



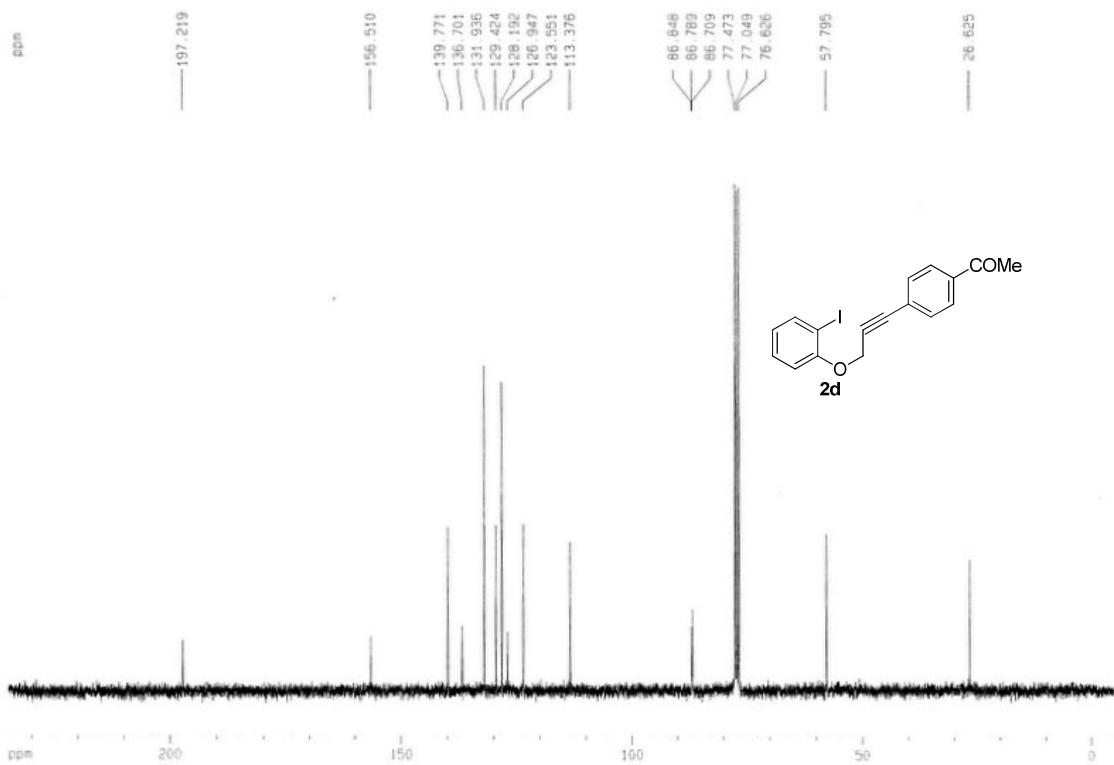
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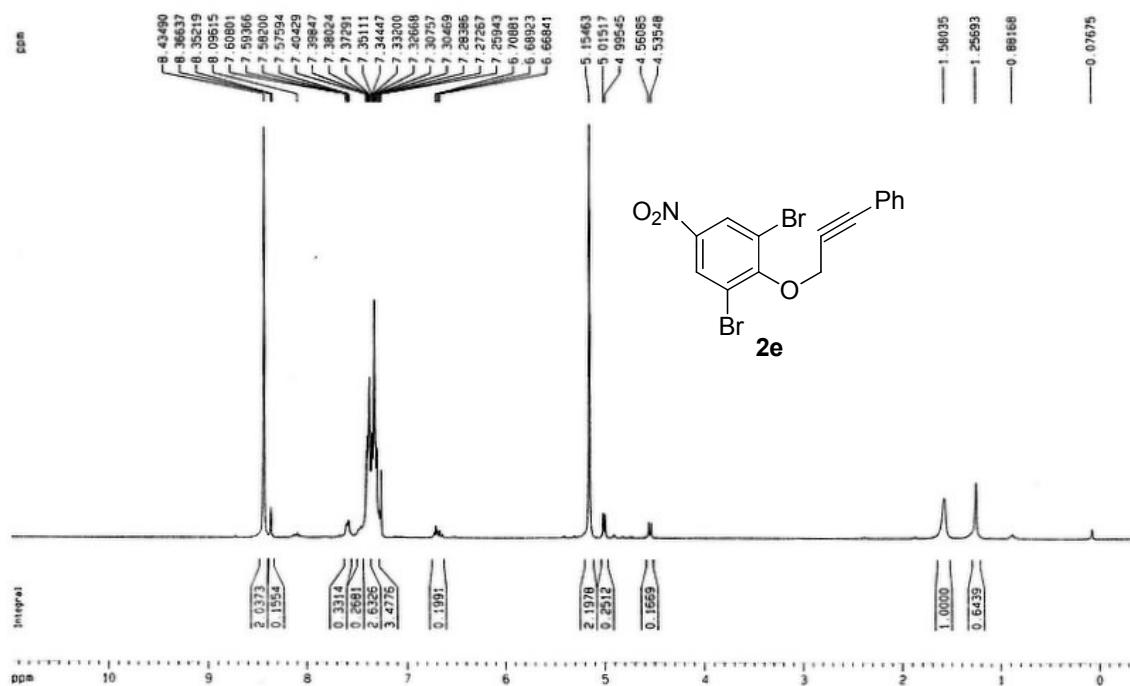
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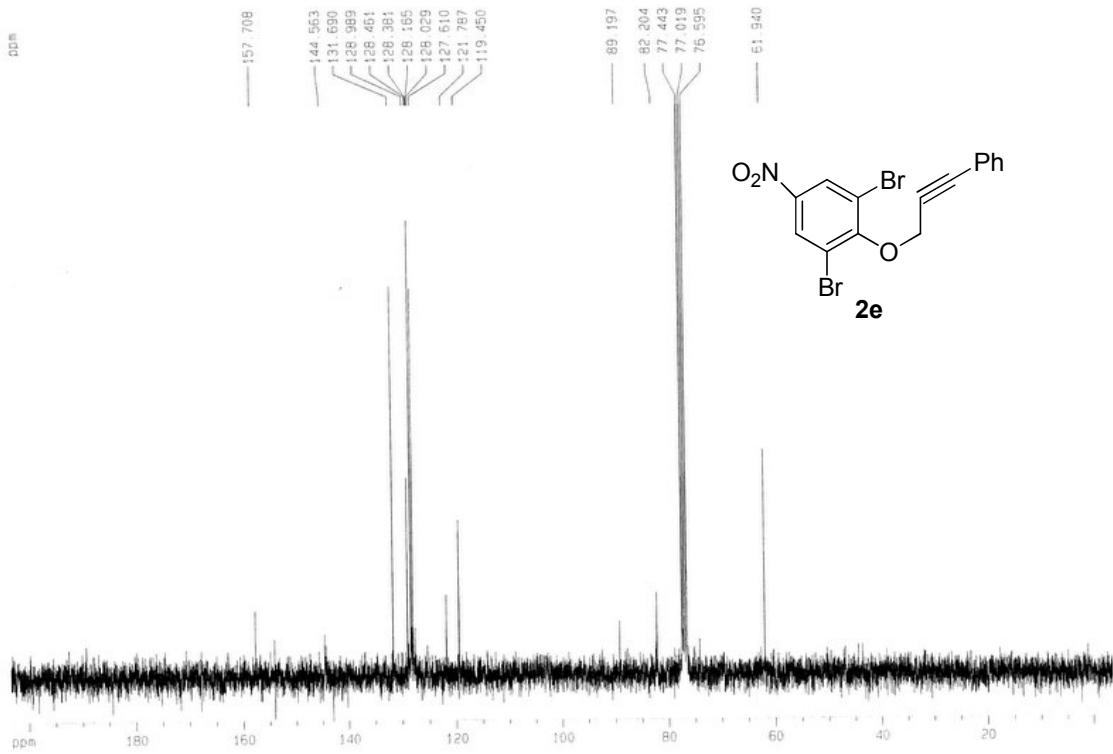
¹³C NMR of **2d**, CDCl₃, 75 MHz



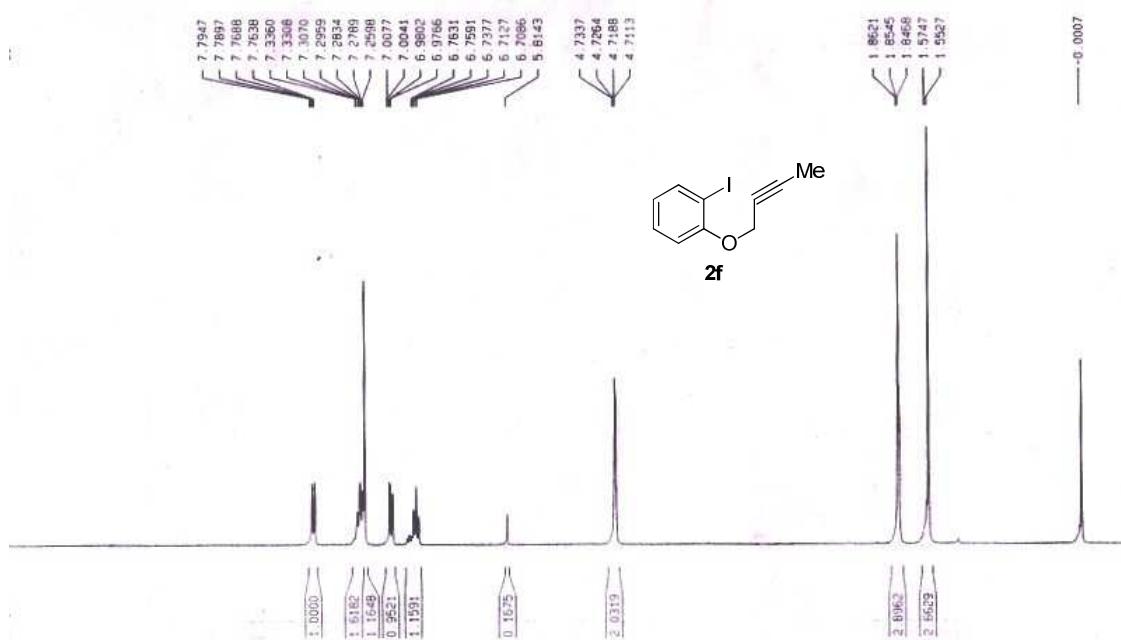
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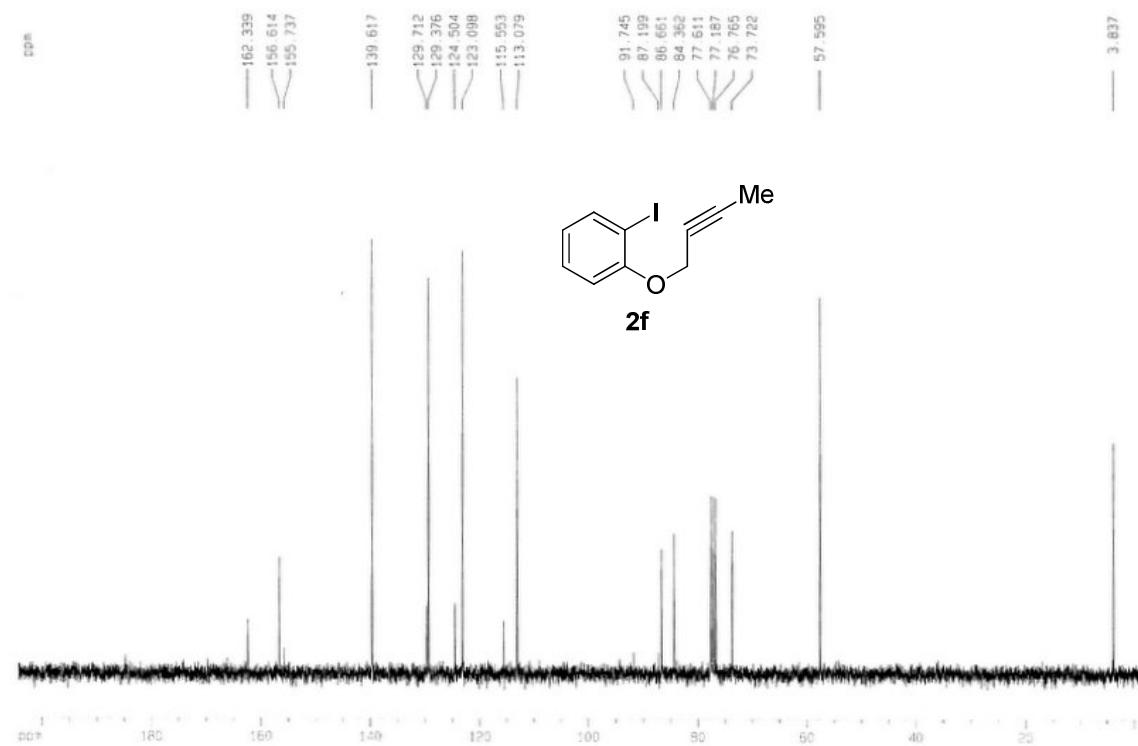
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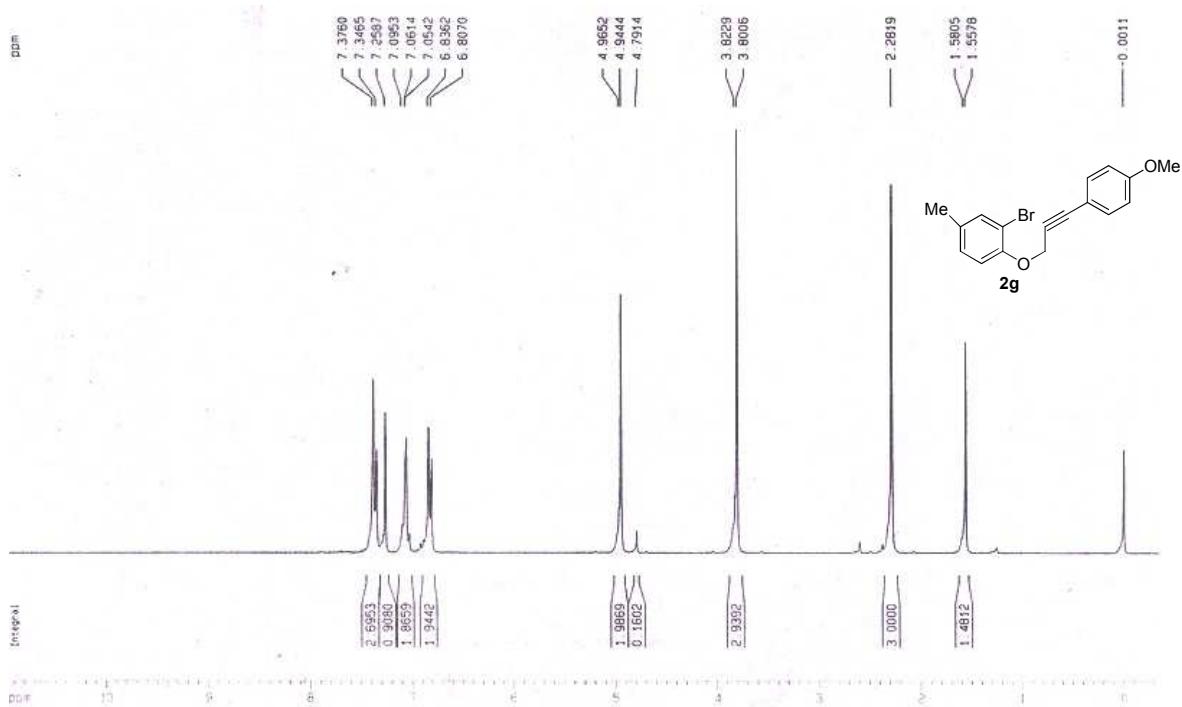
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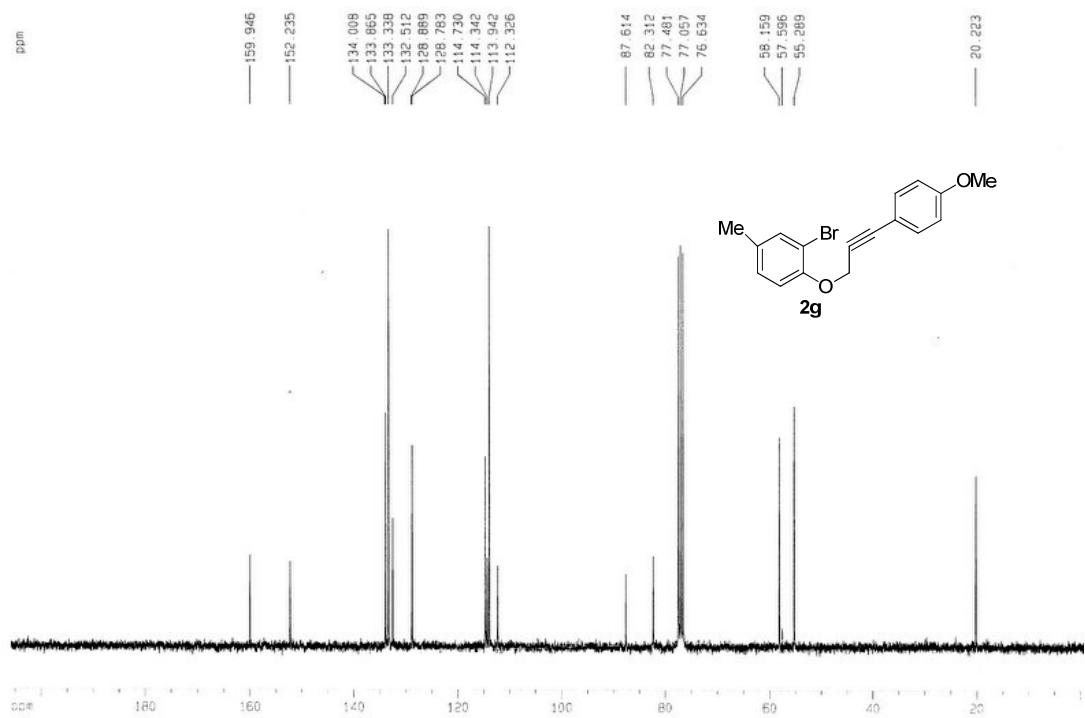
¹³C NMR of **2f**, CDCl₃, 75 MHz



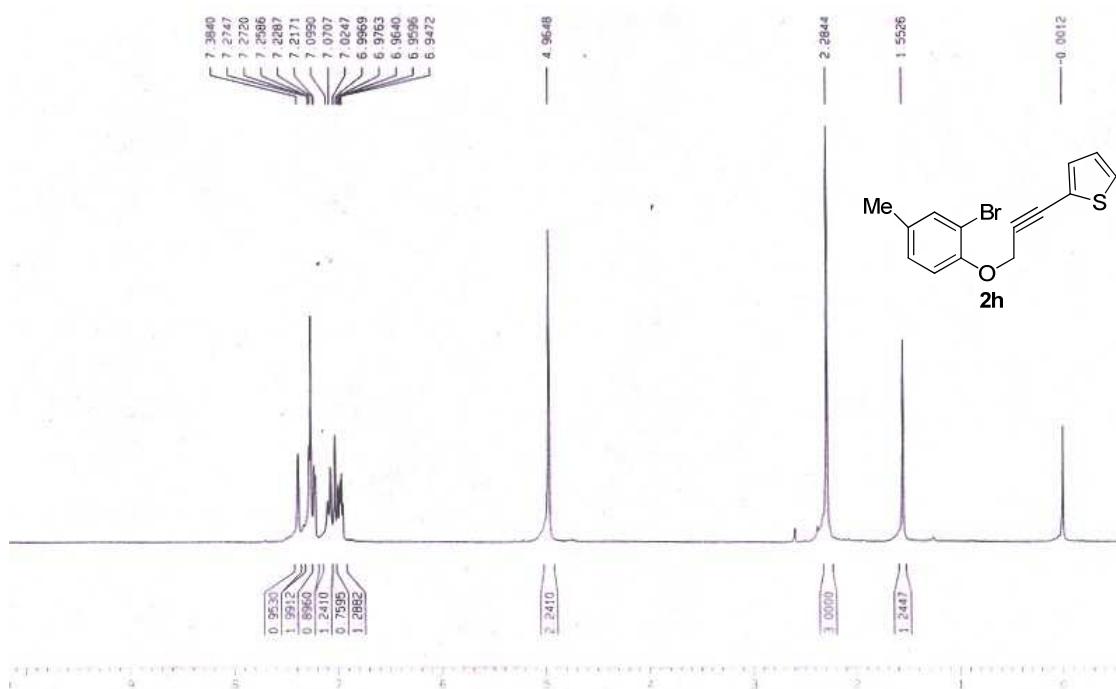
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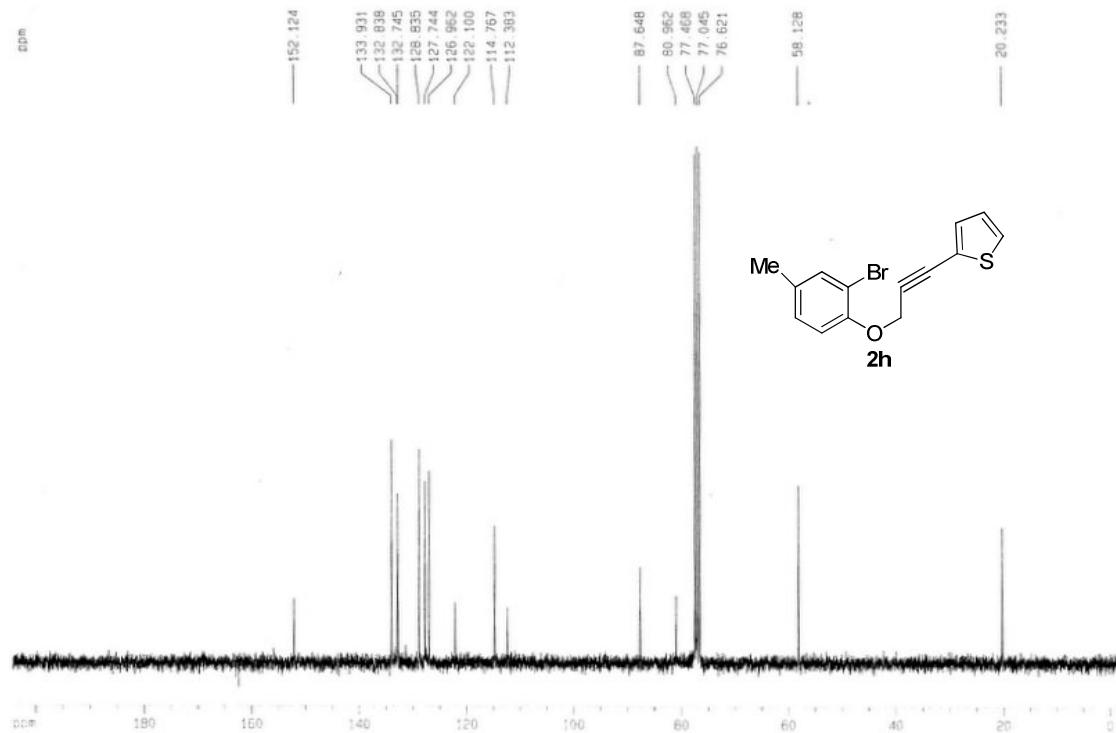
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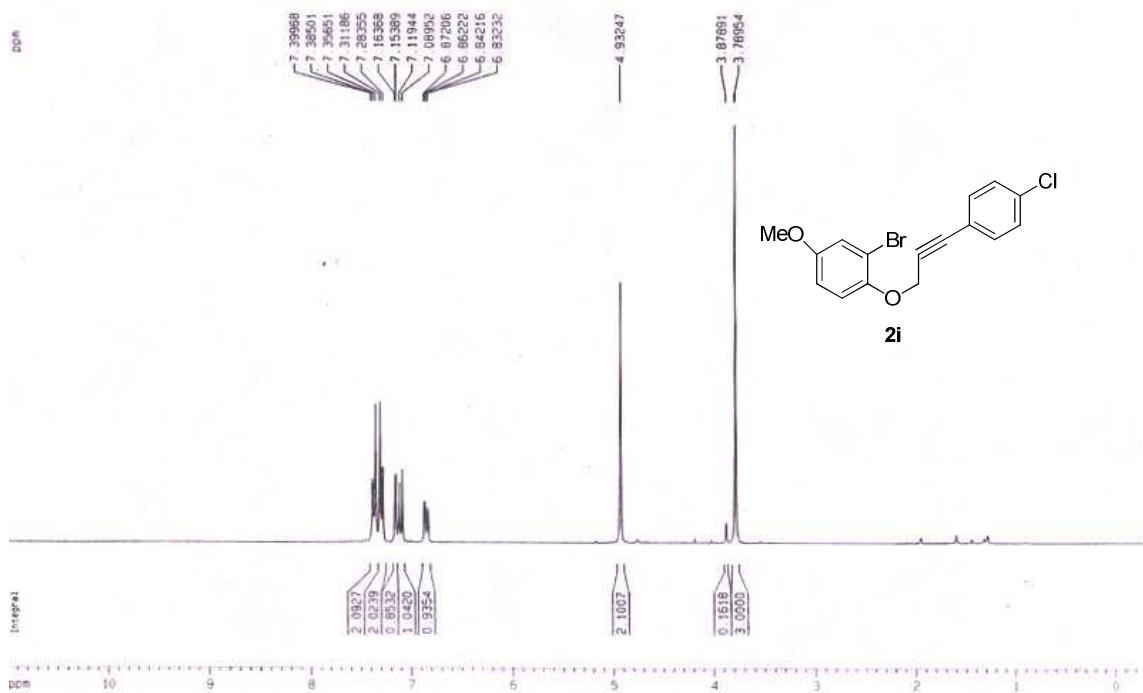
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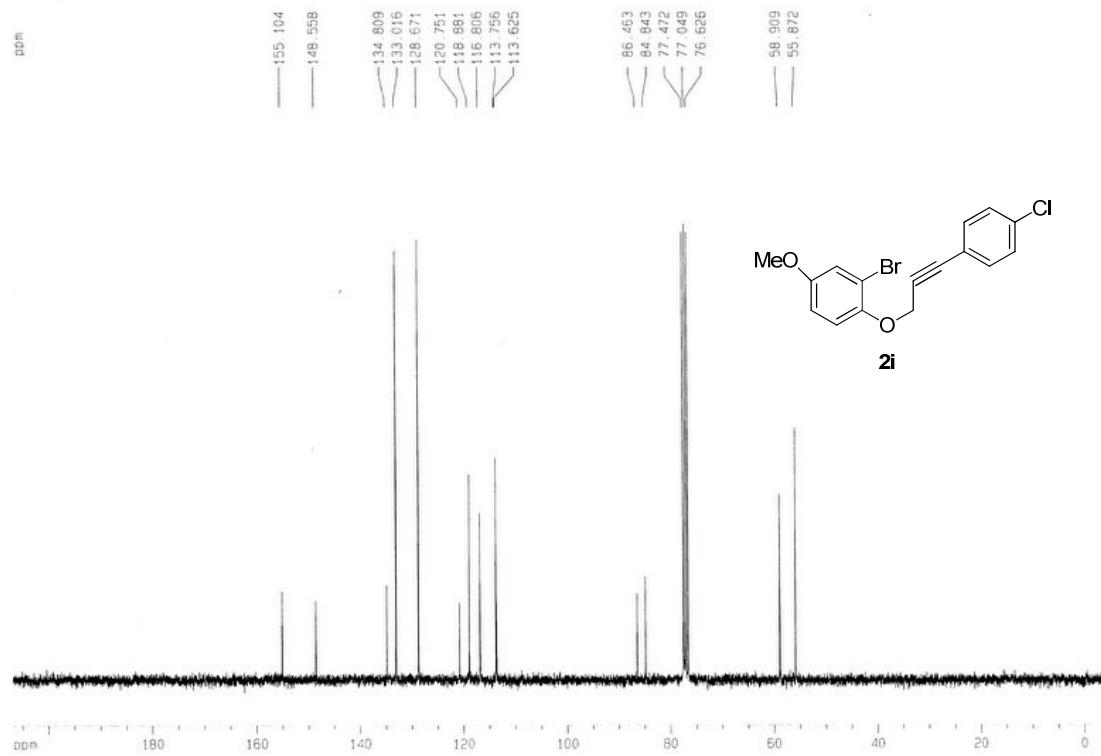
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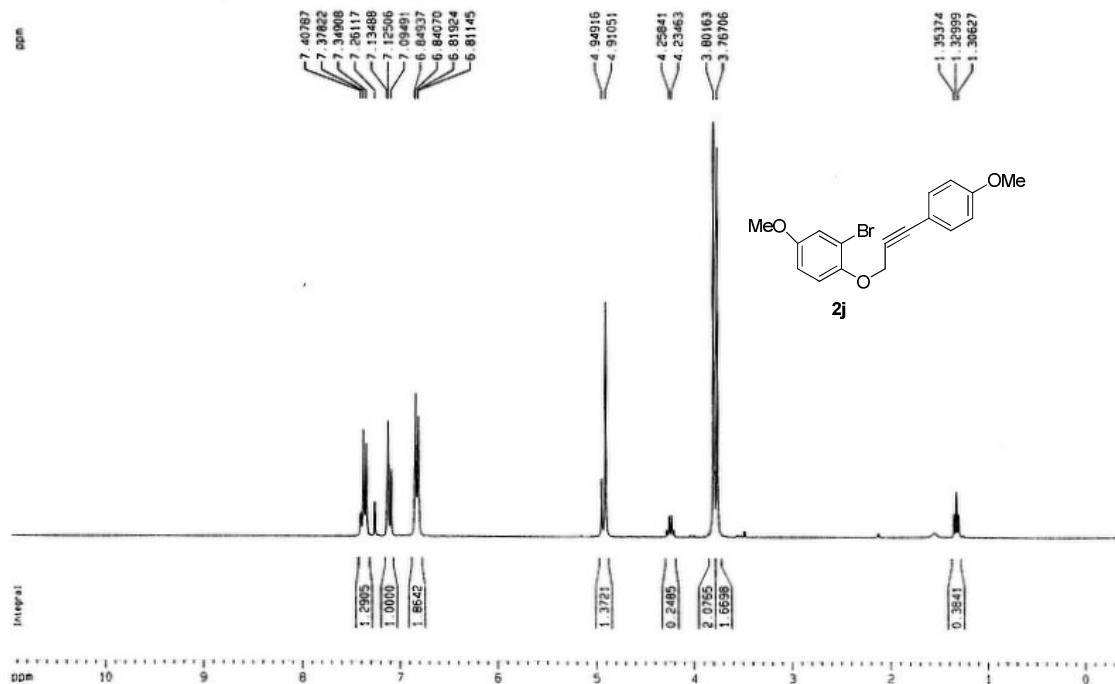
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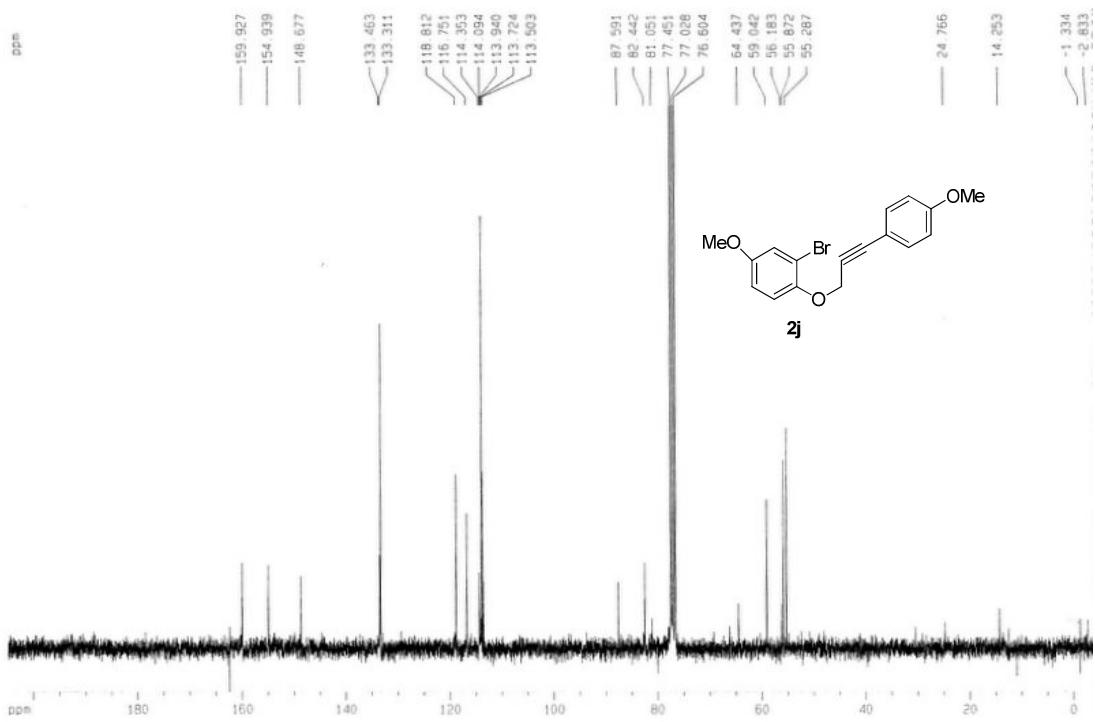
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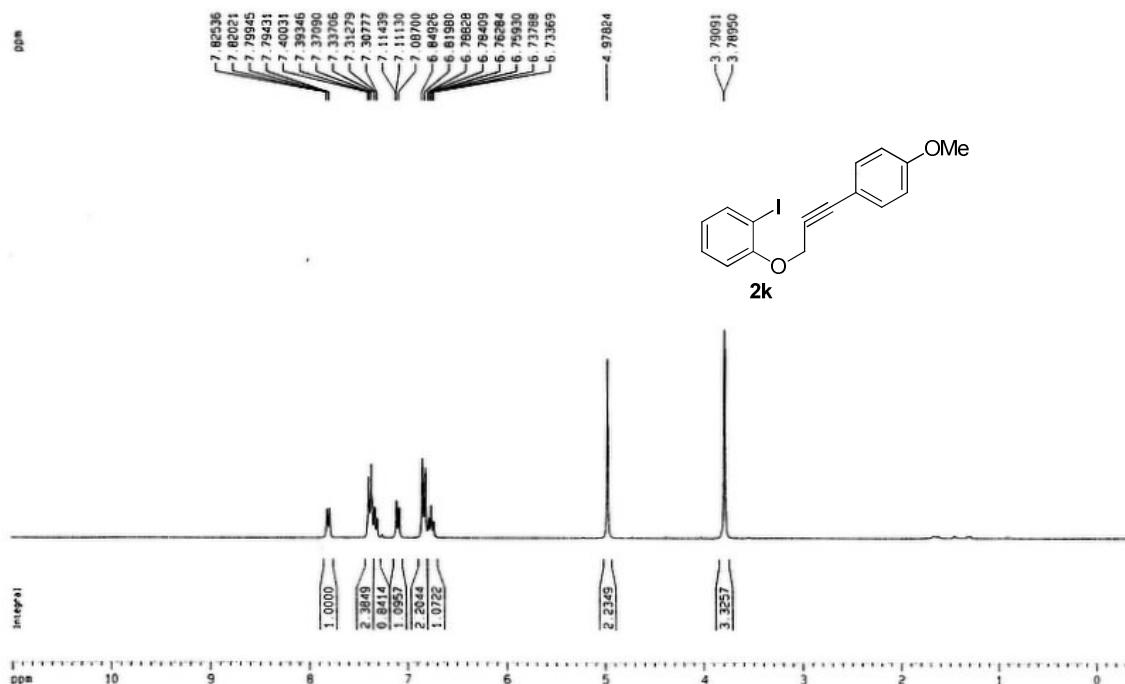
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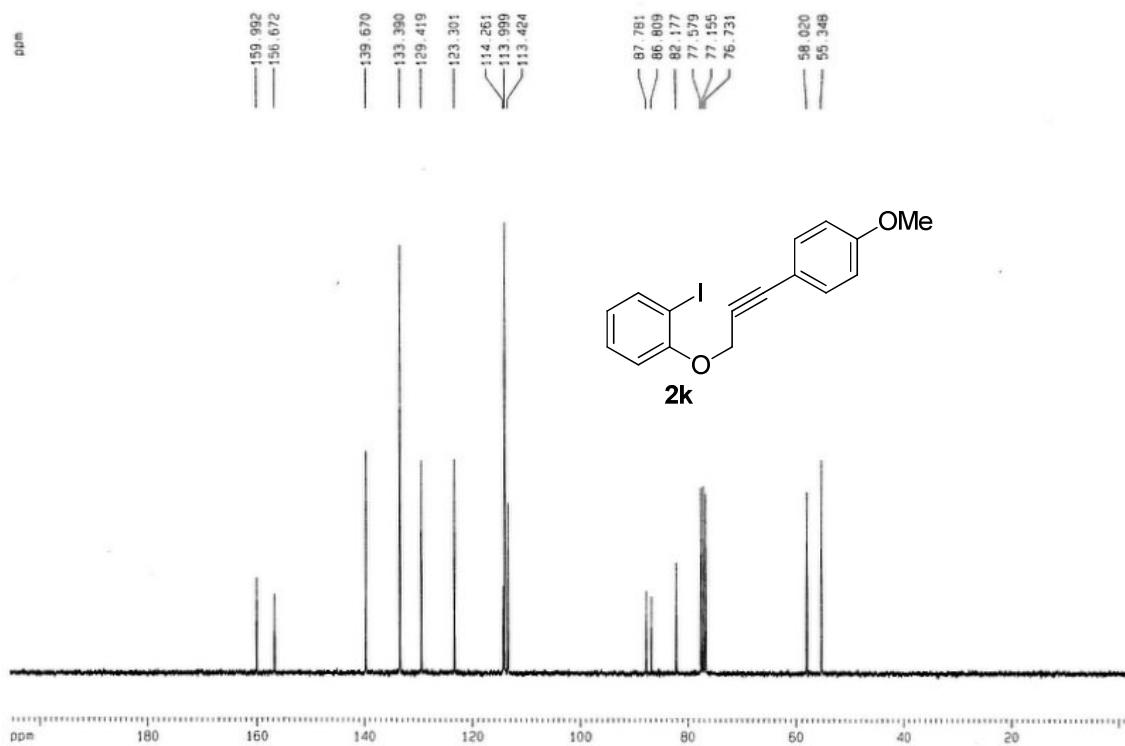
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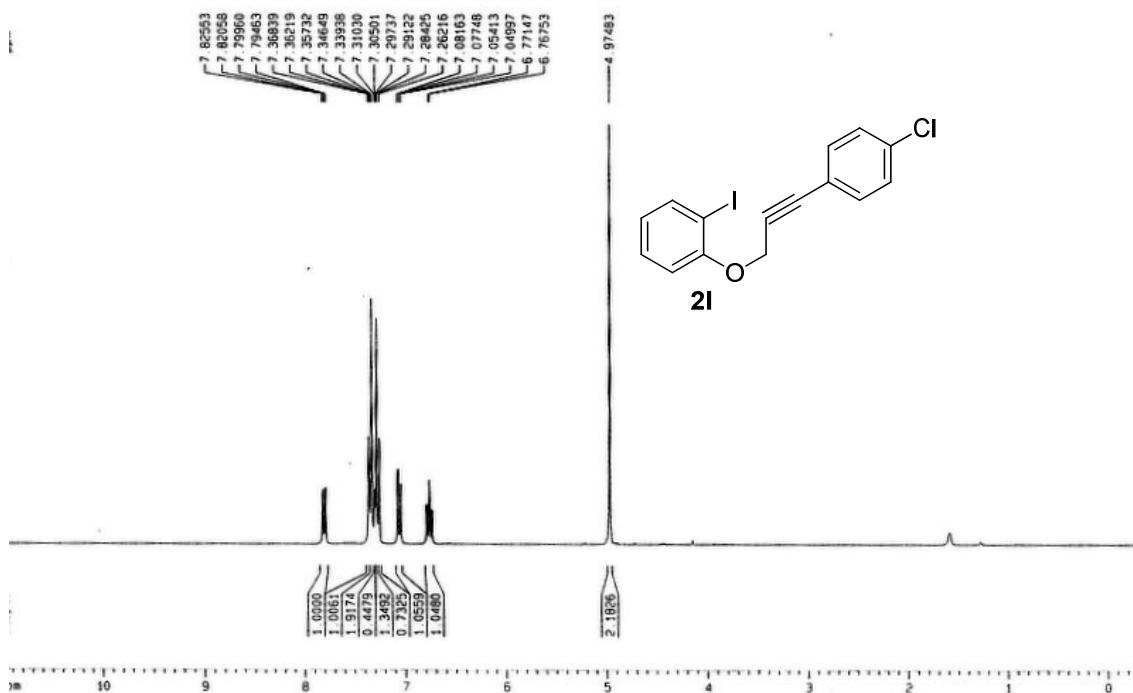
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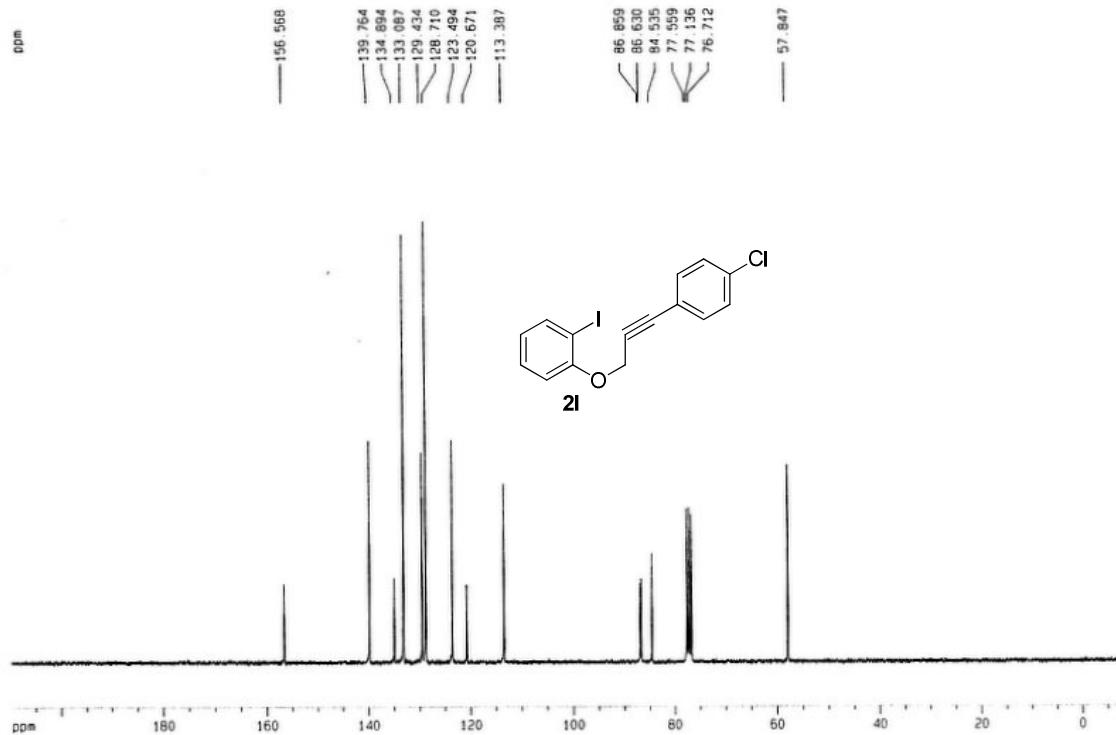
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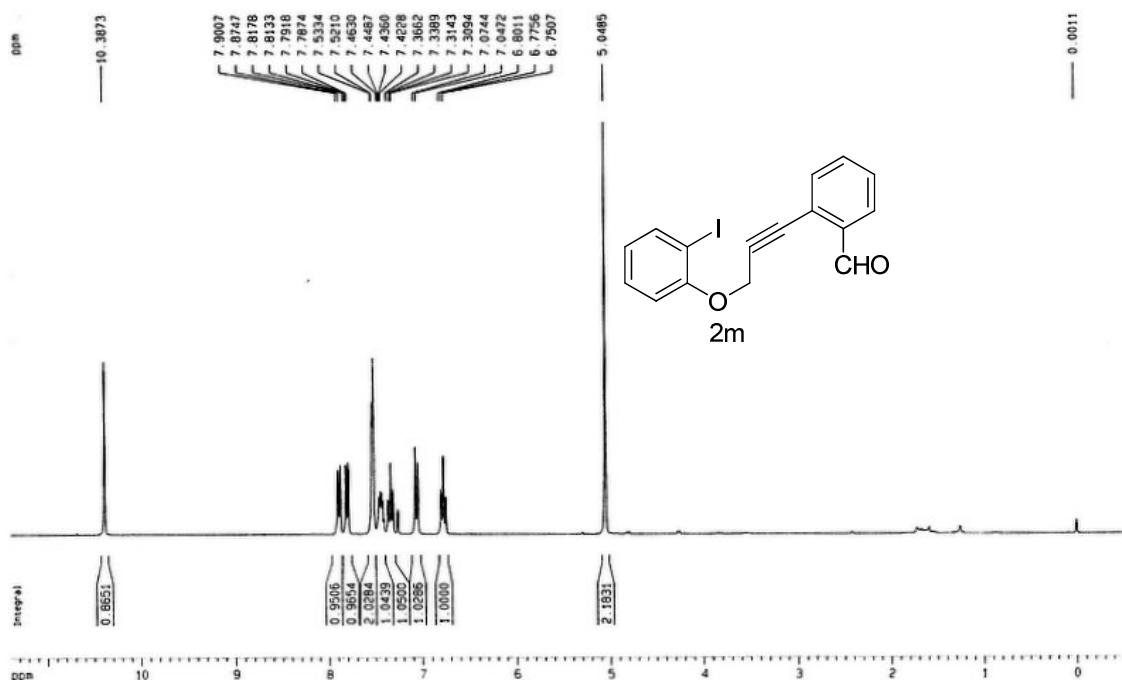
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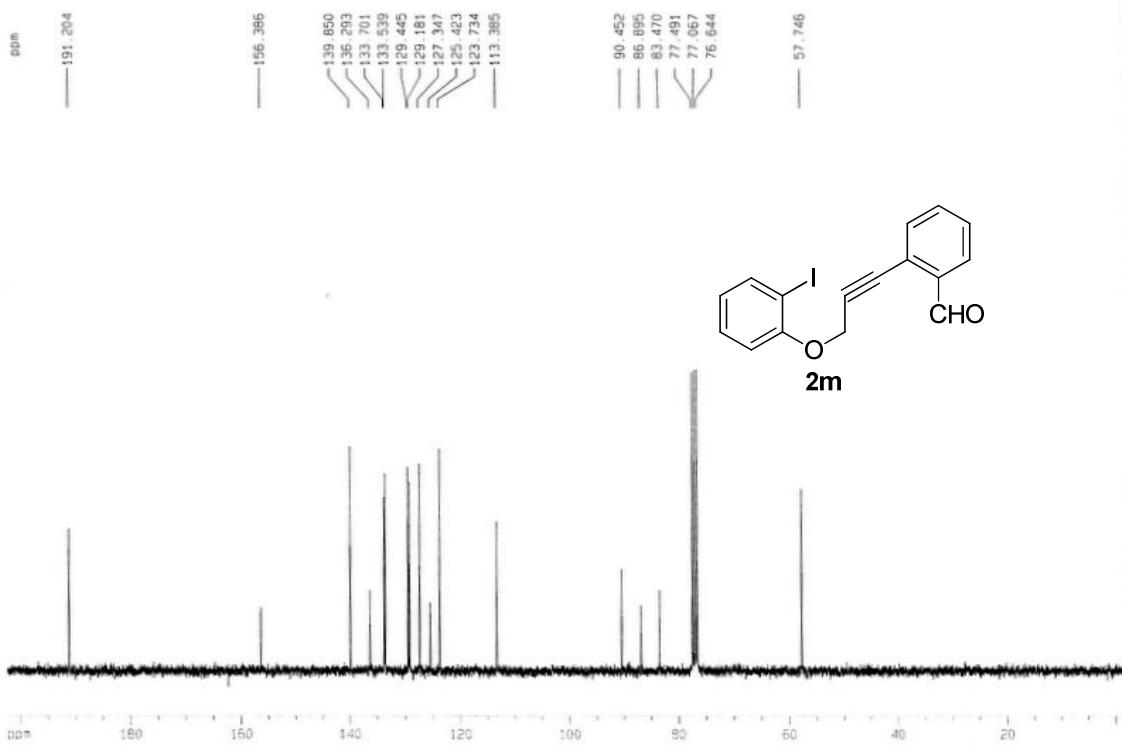
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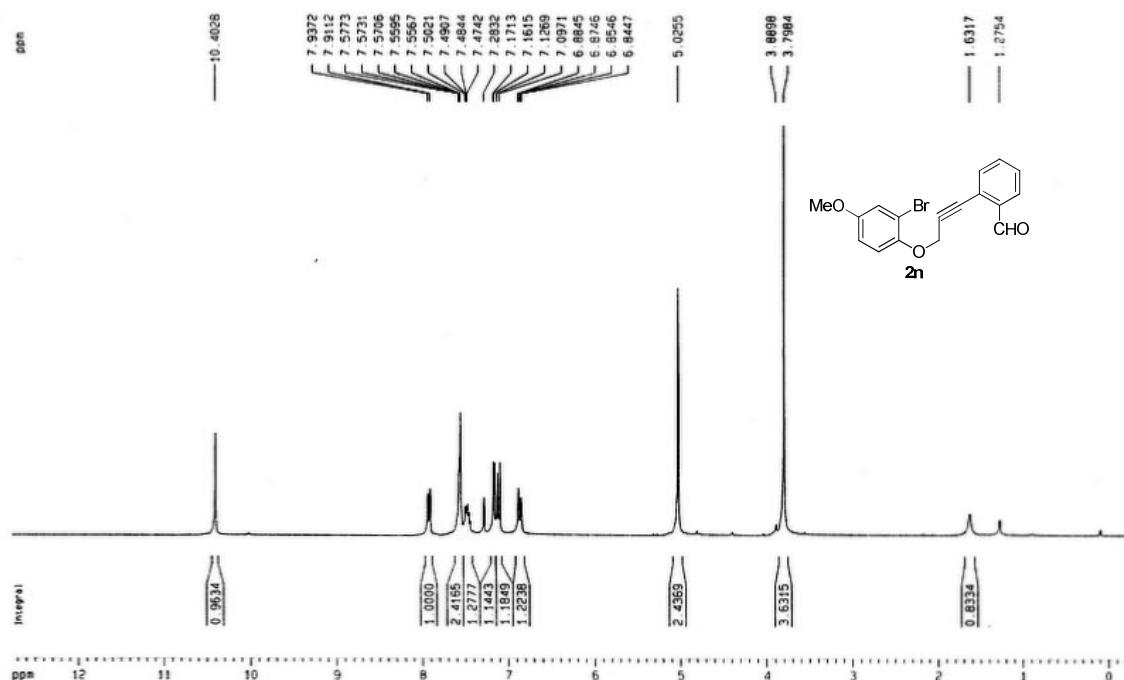
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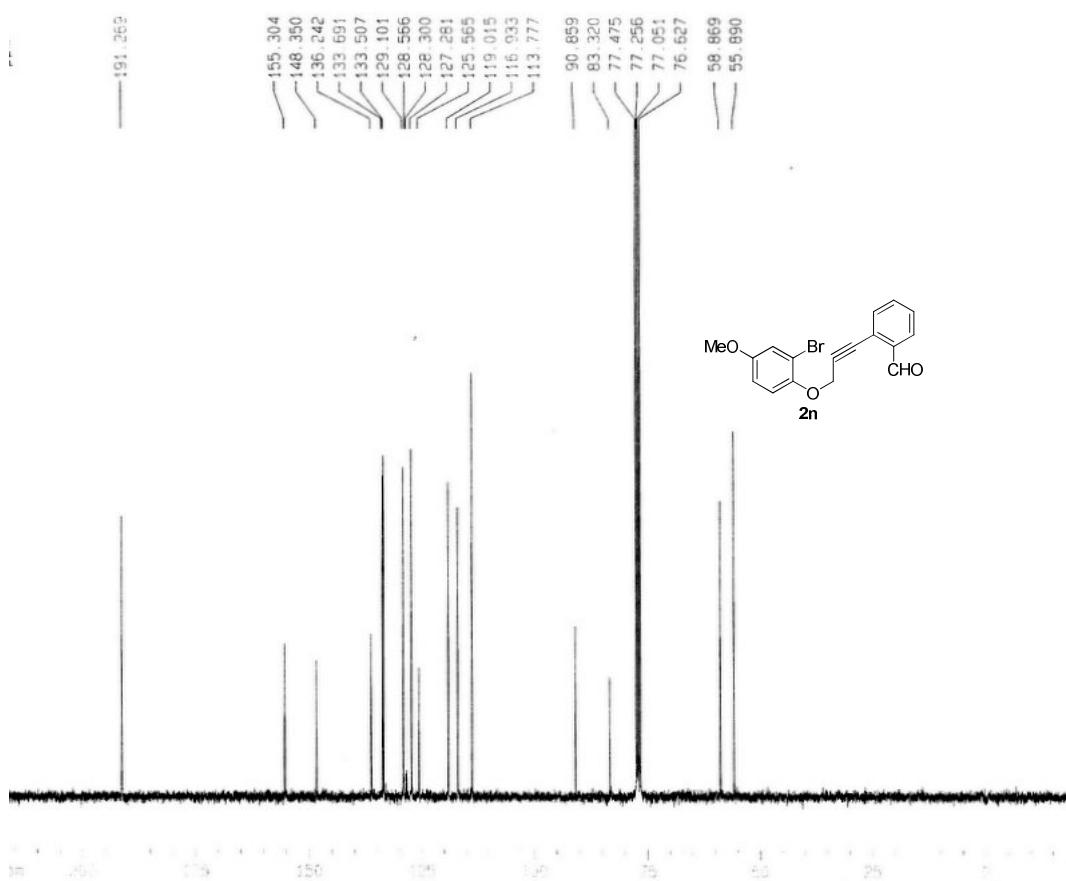
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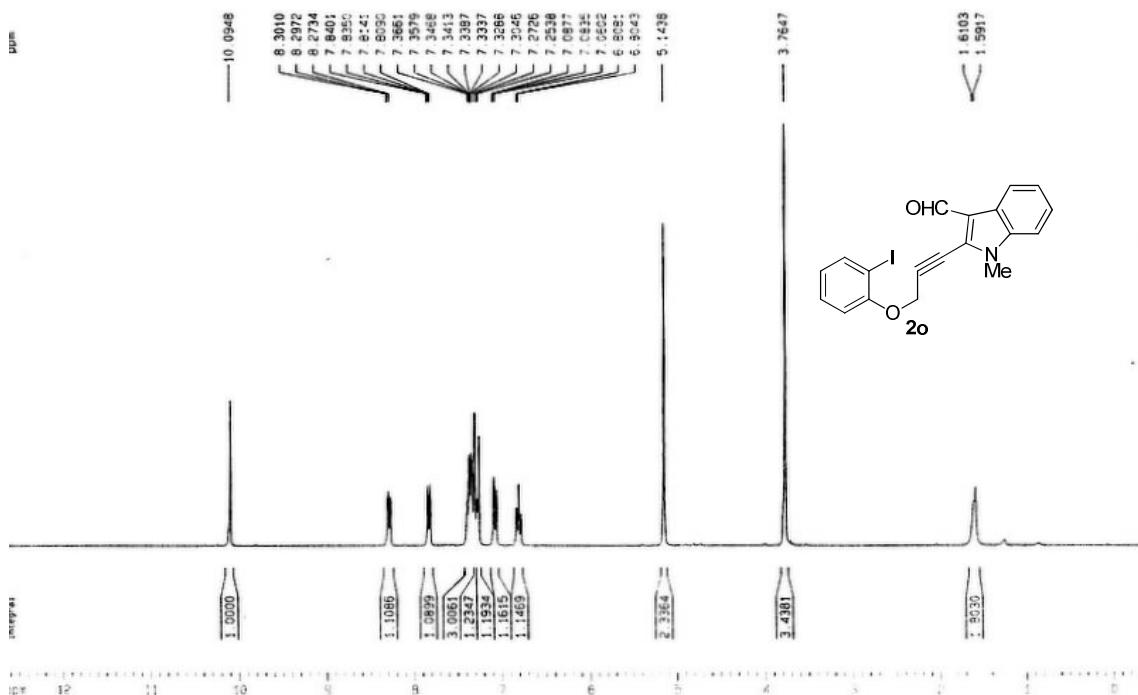
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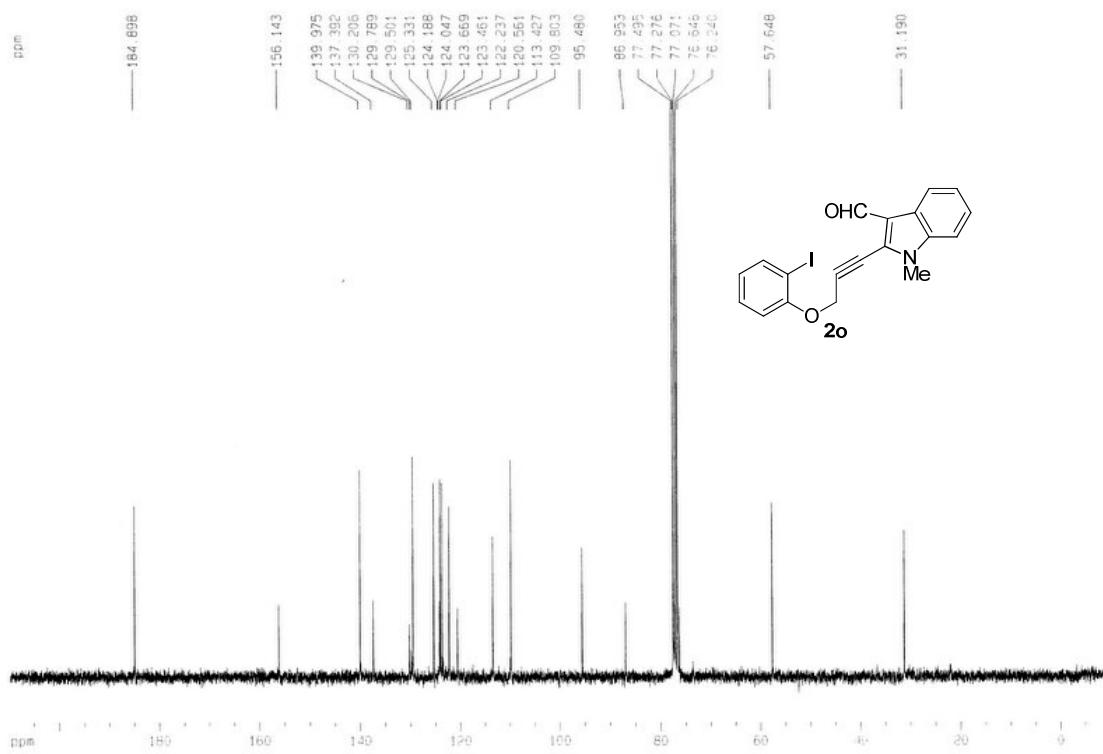
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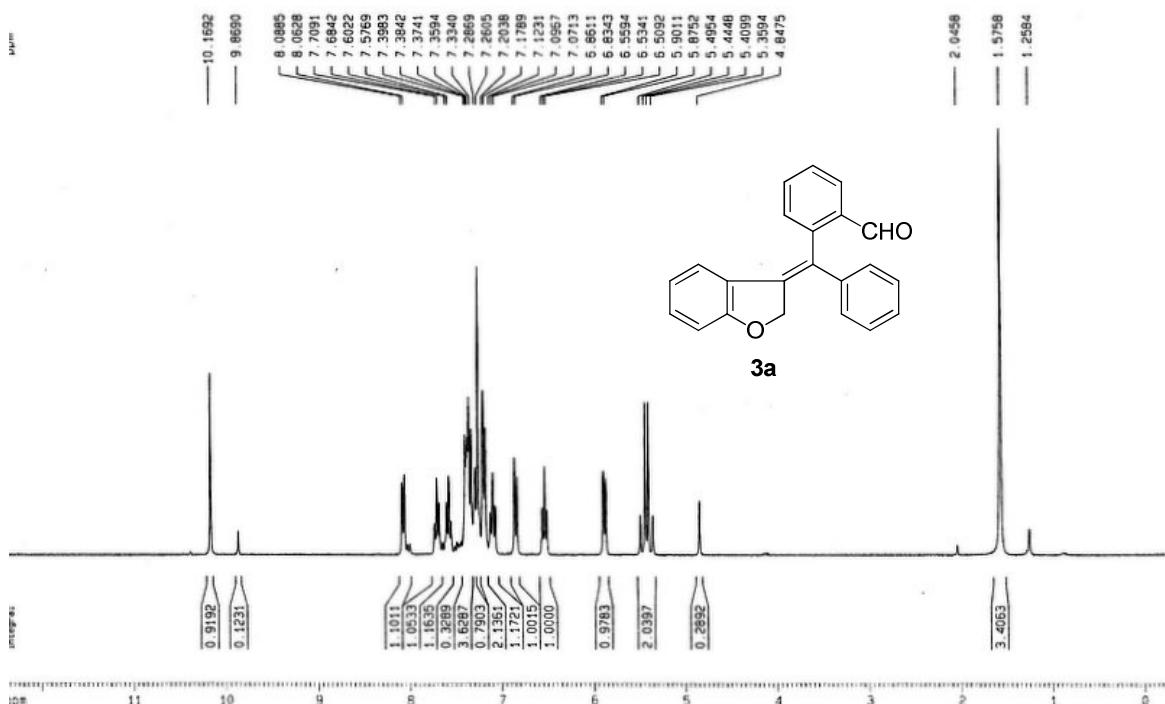
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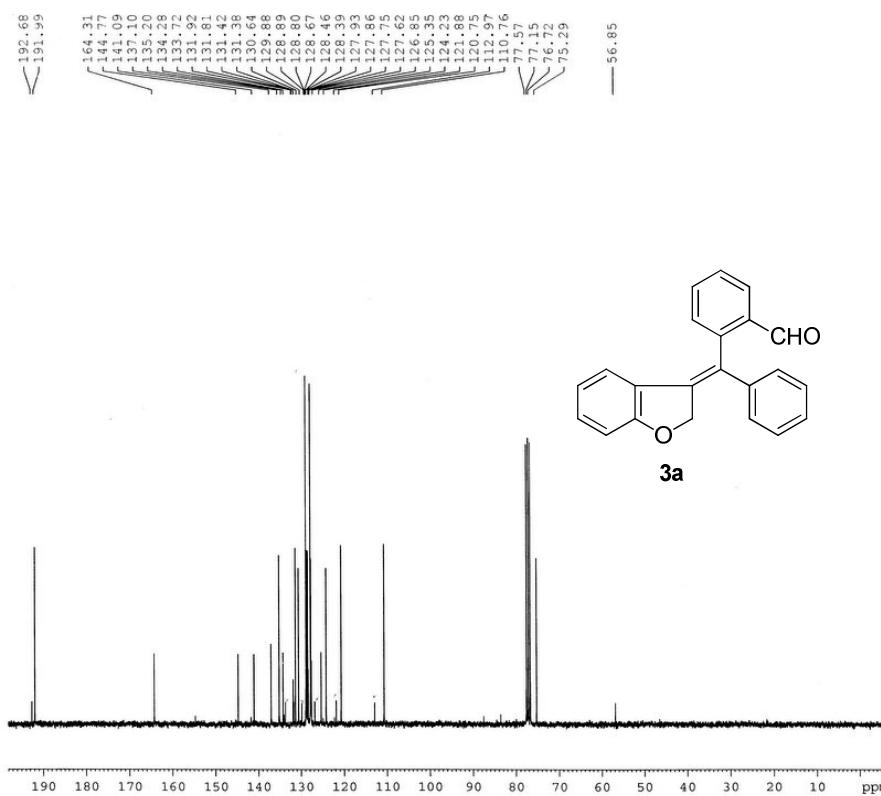
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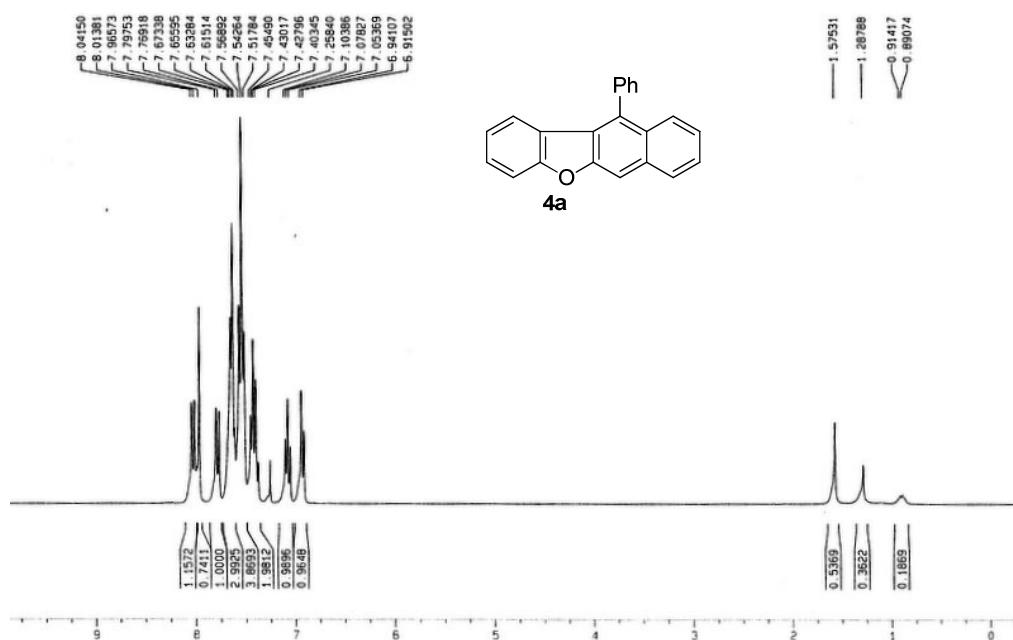
¹H NMR of **3a**, CDCl₃, 300 MHz



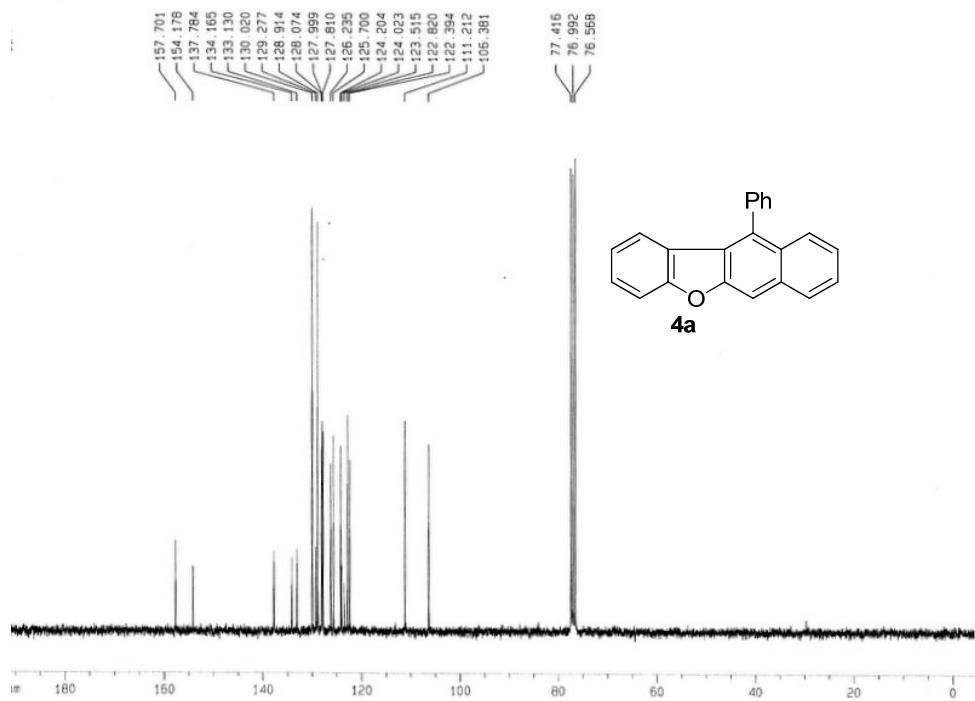
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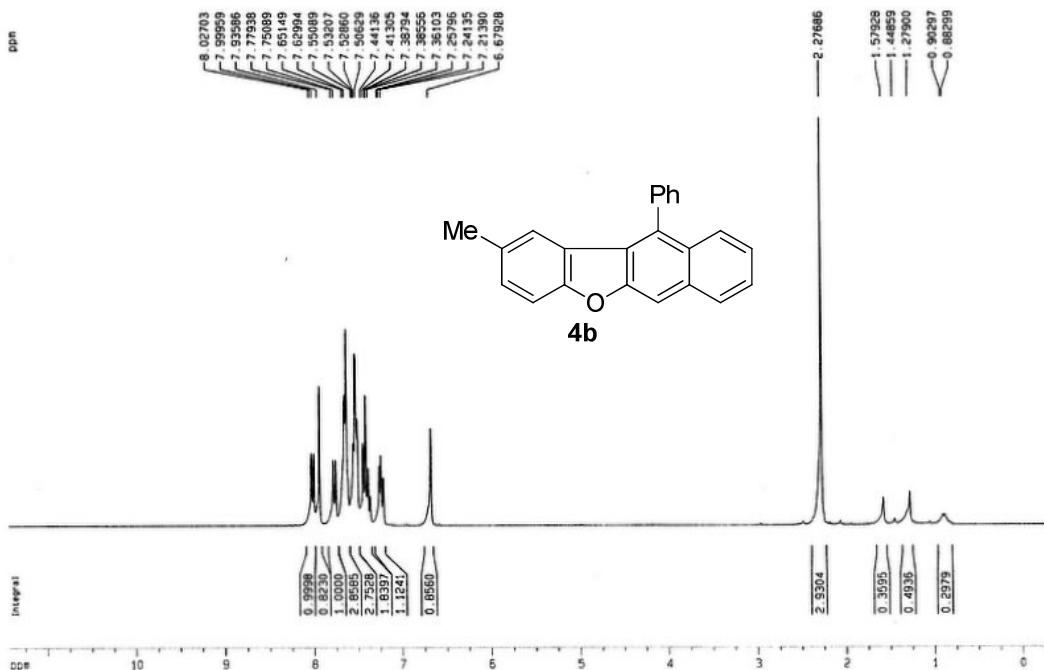
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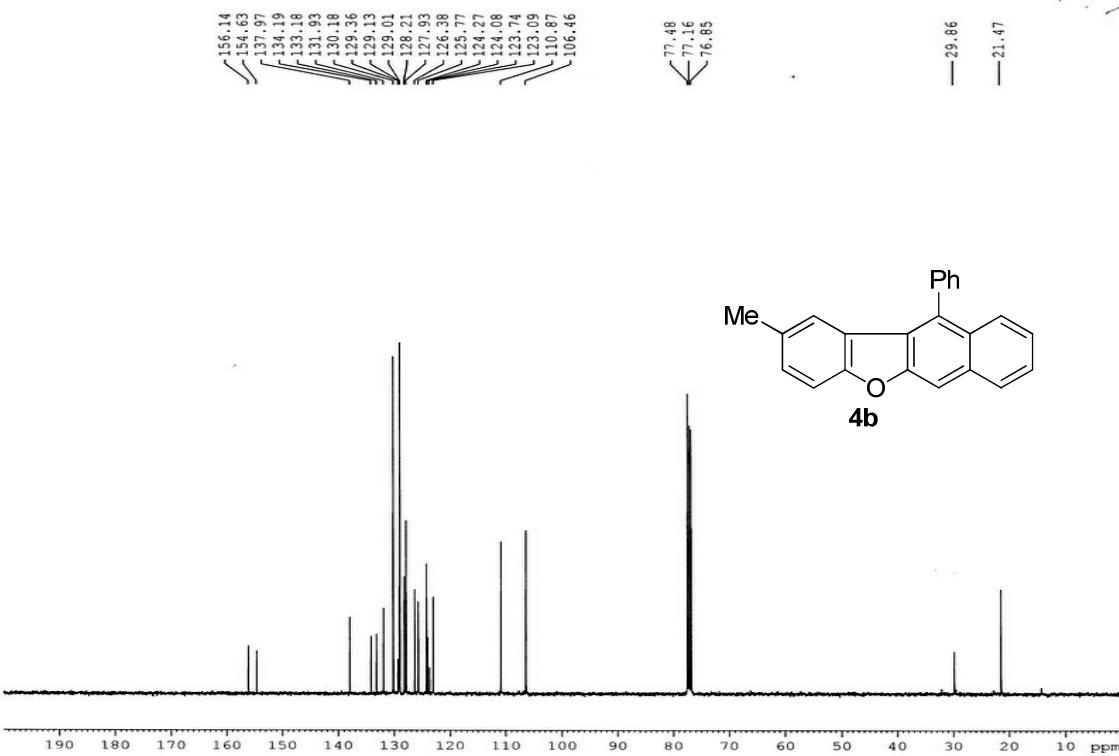
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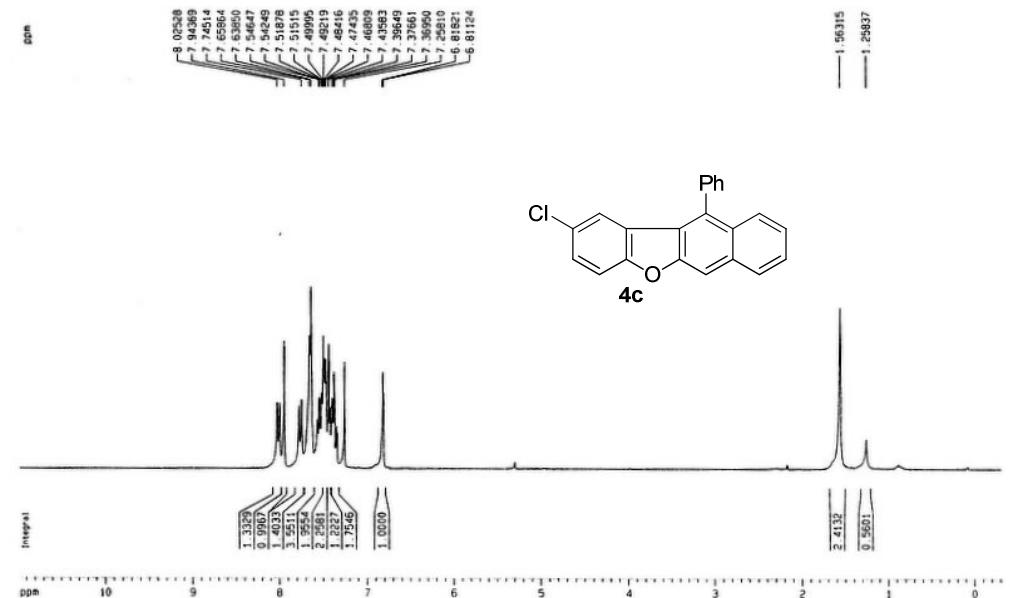
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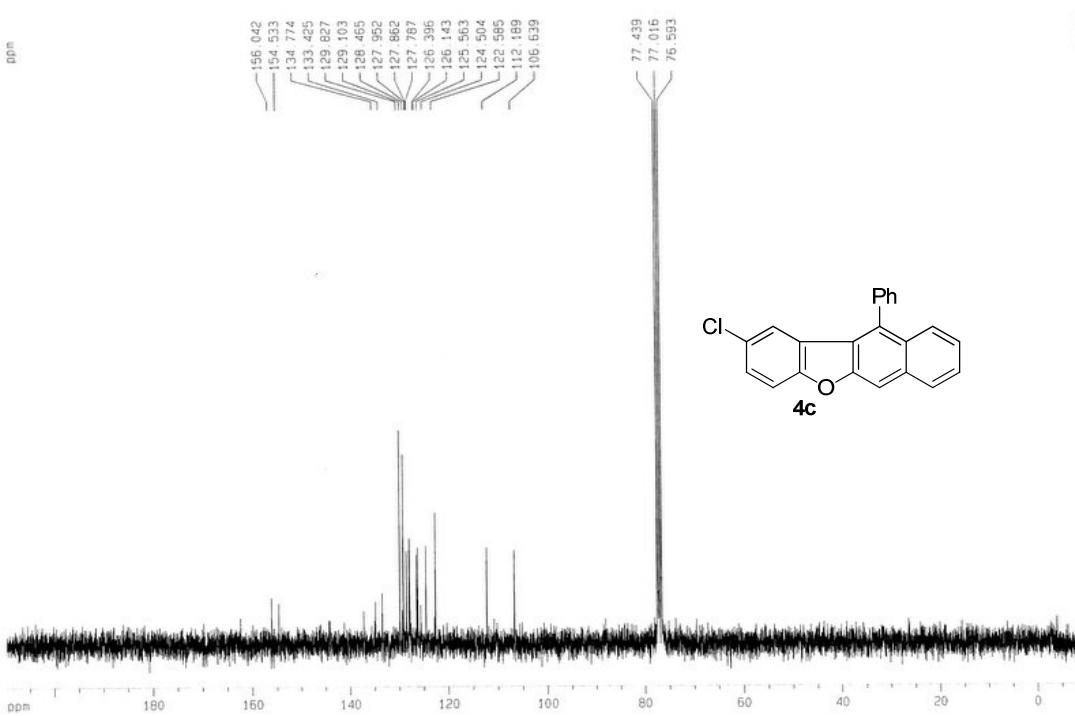
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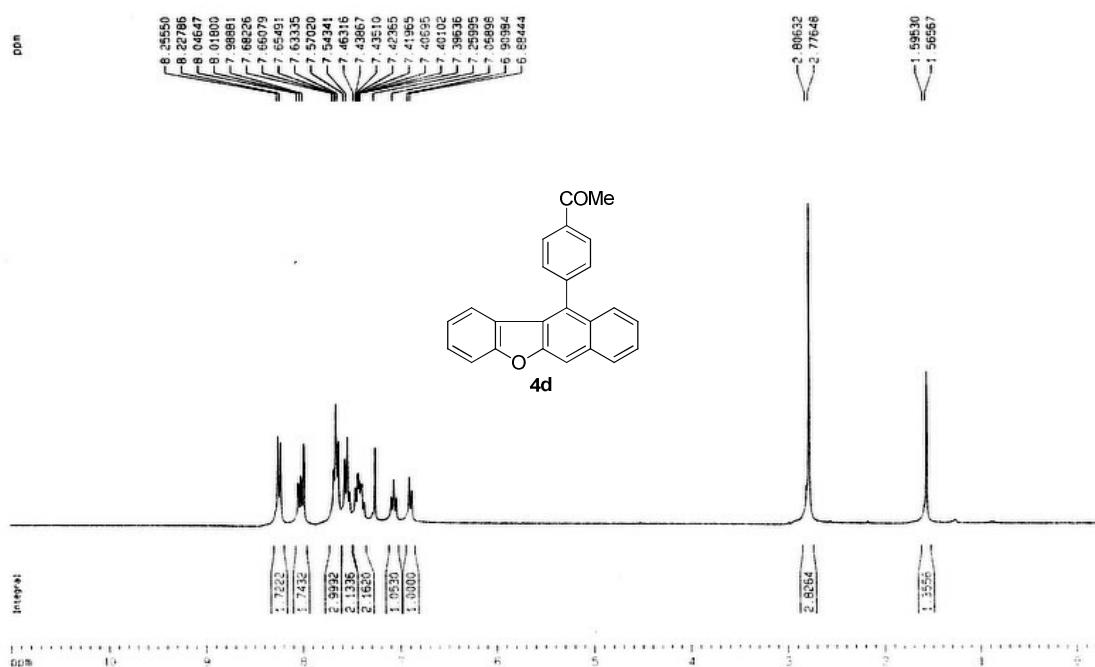
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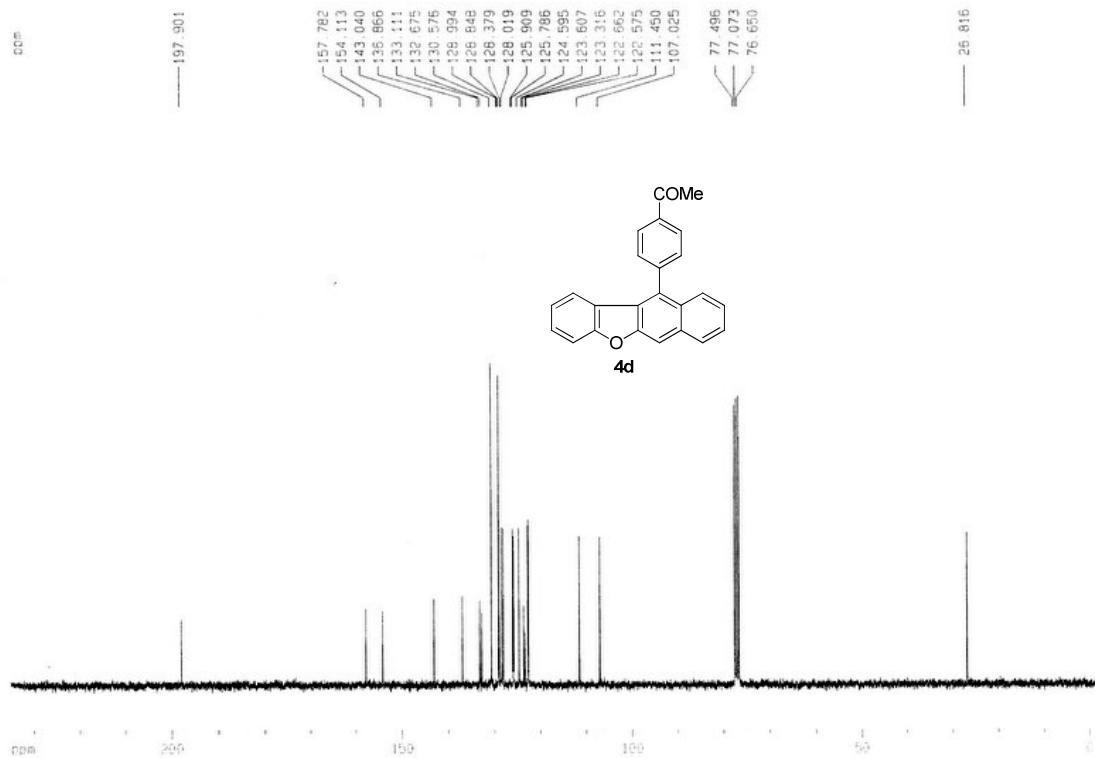
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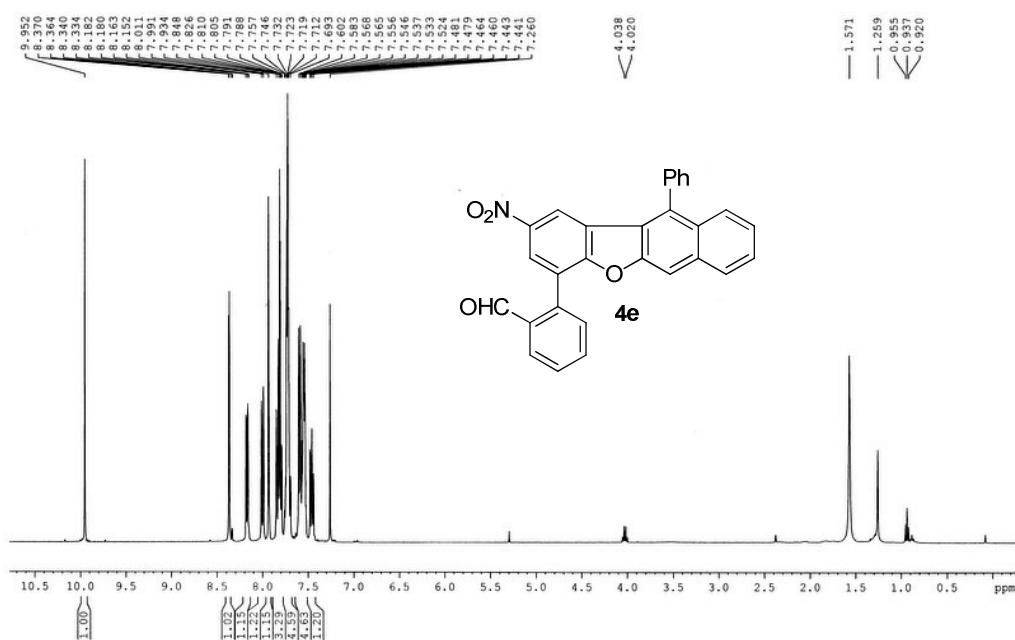
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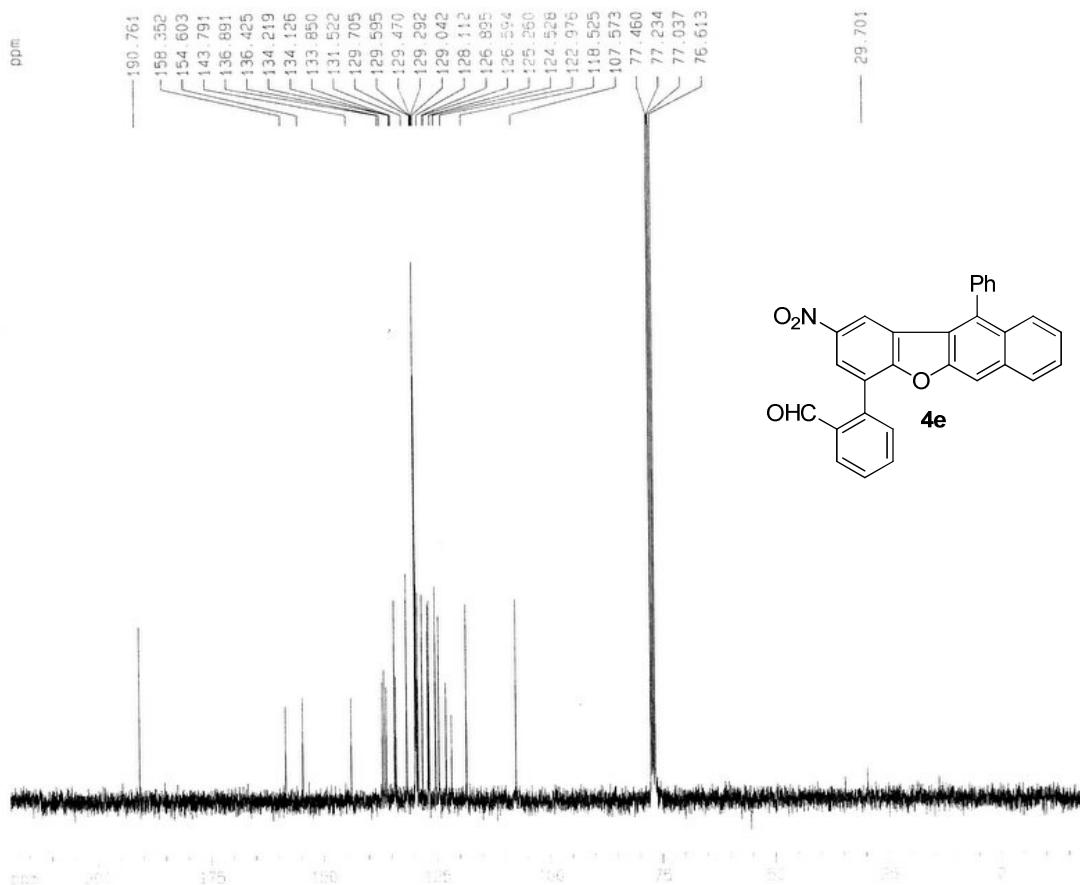
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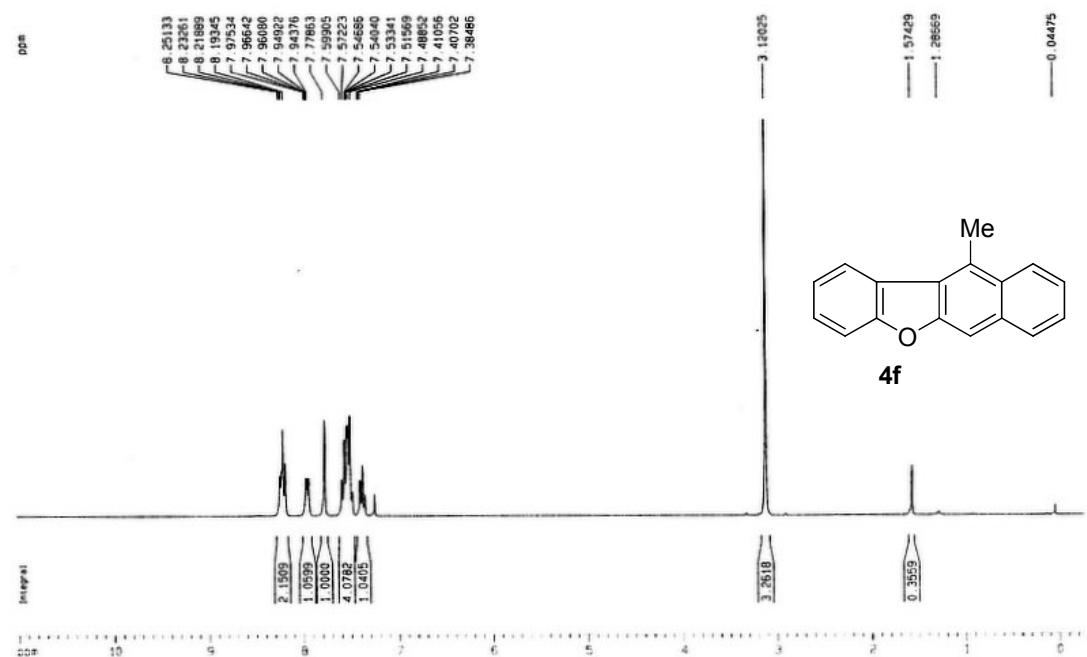
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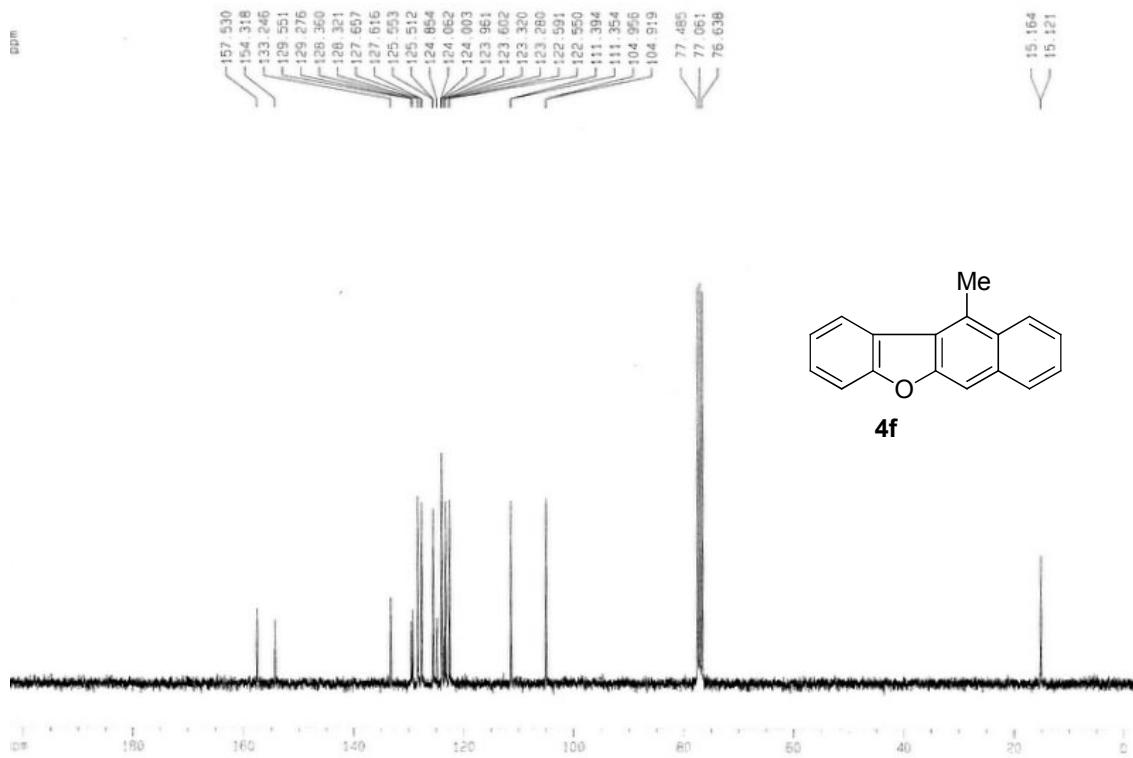
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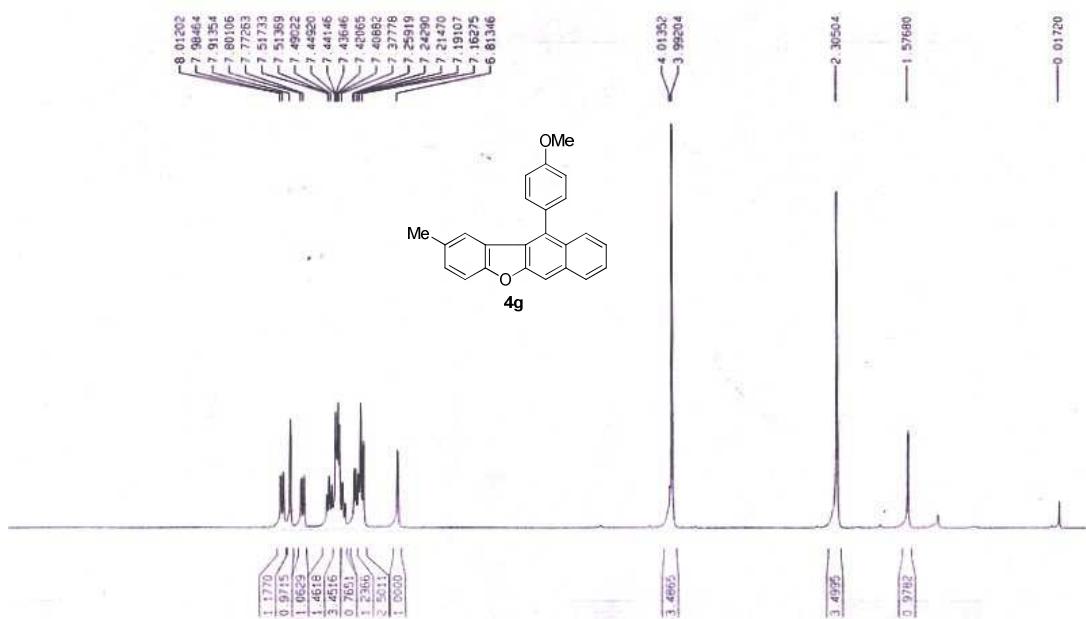
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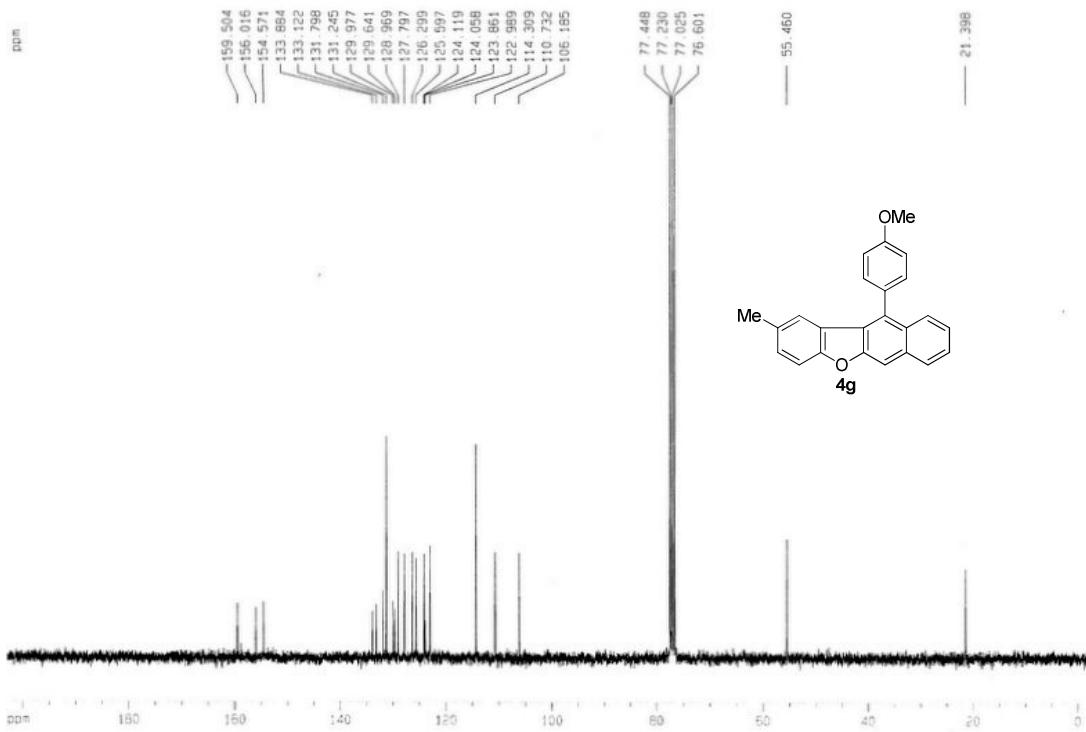
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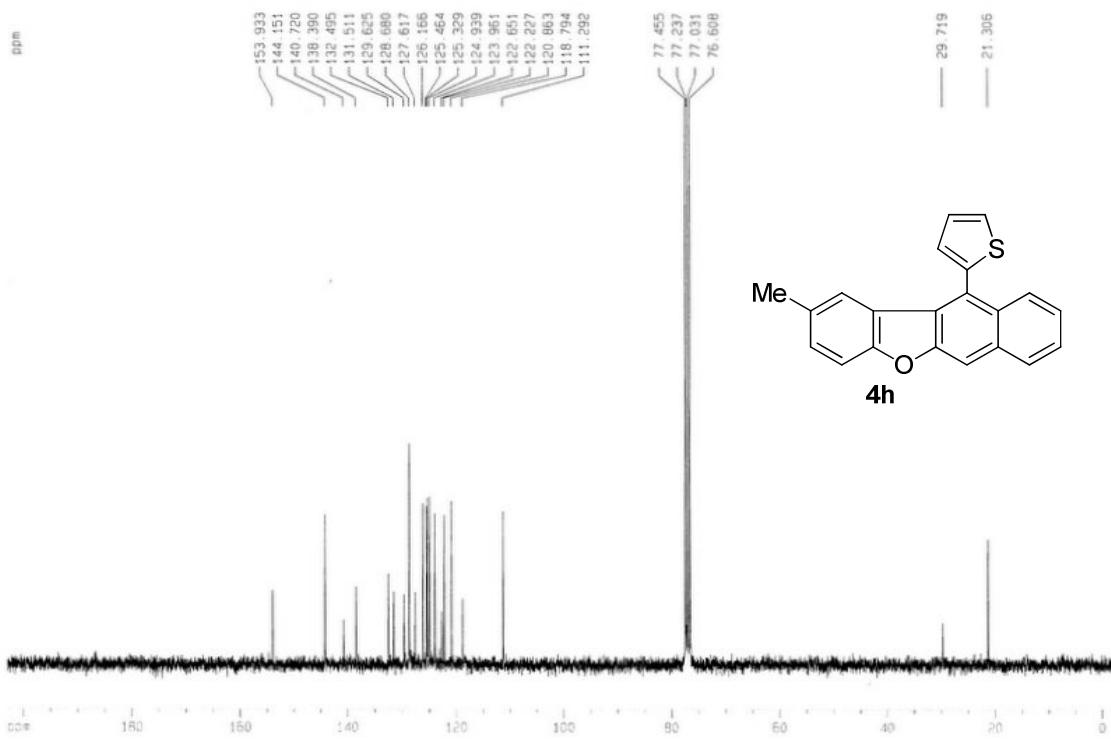
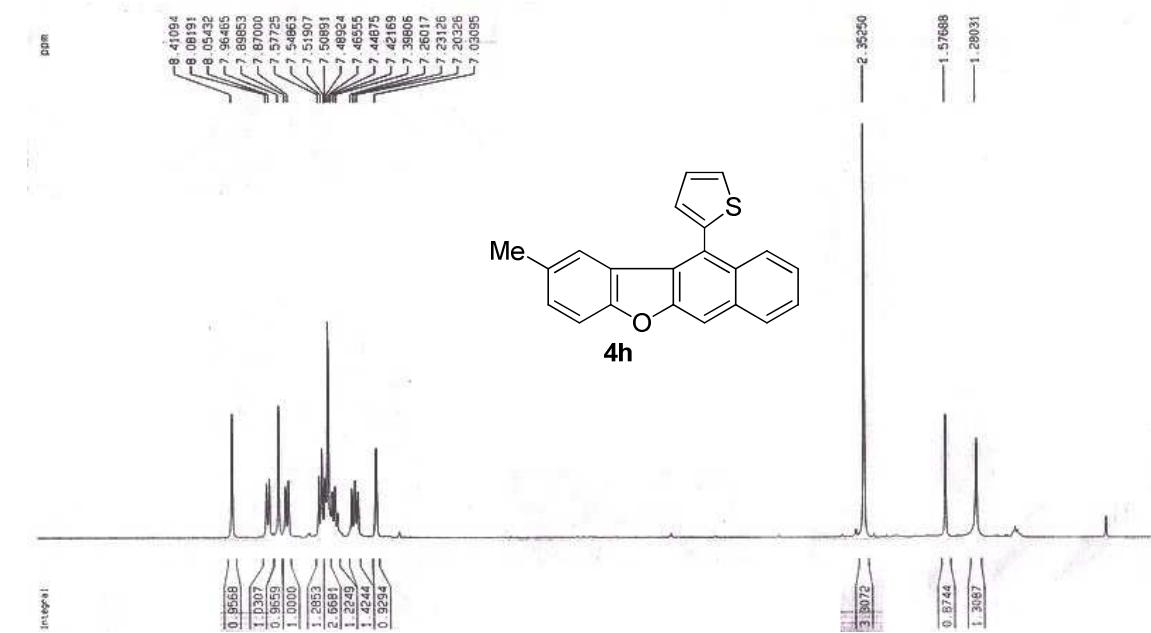
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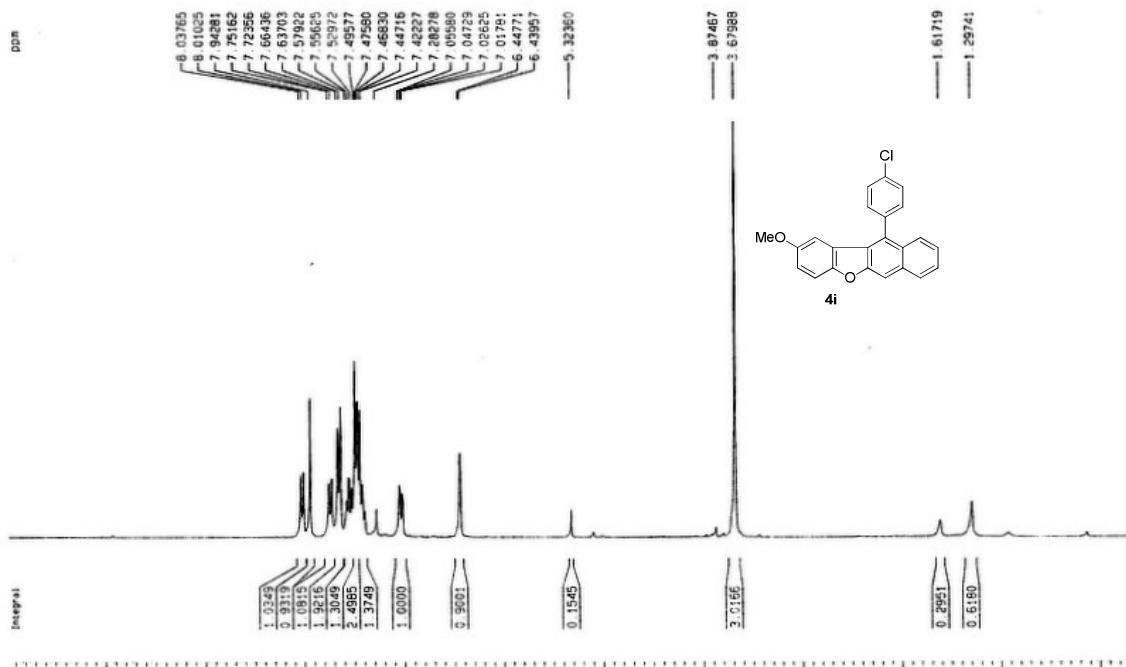
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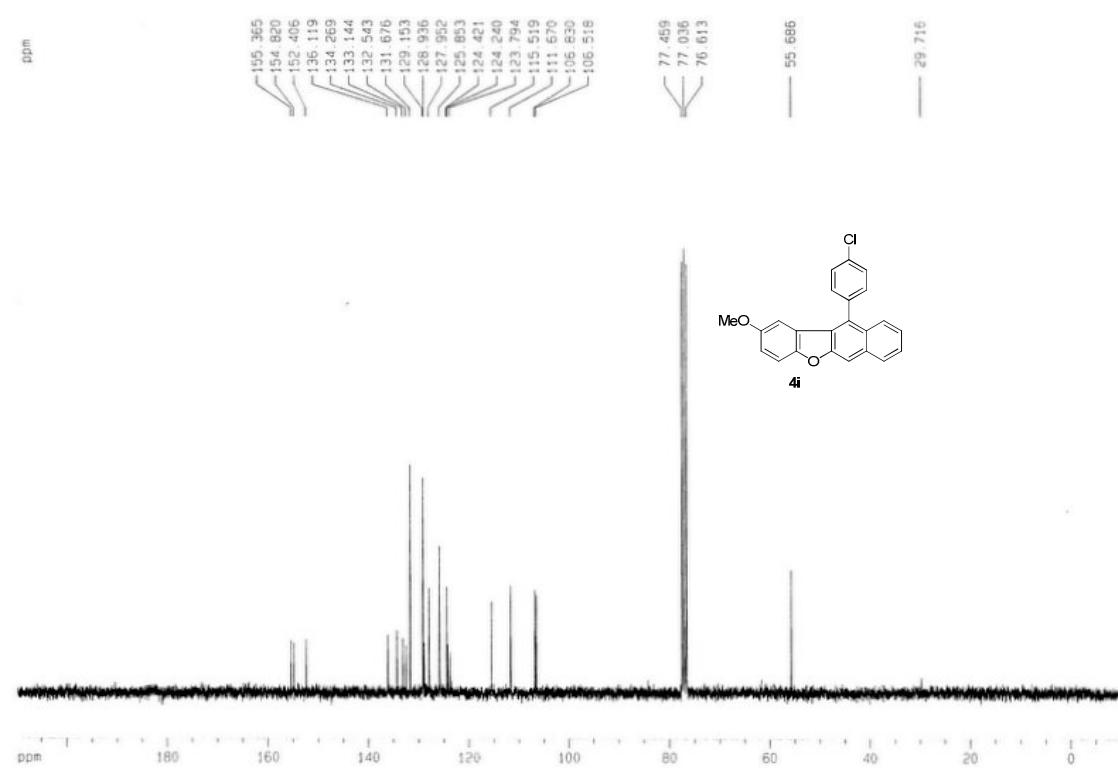
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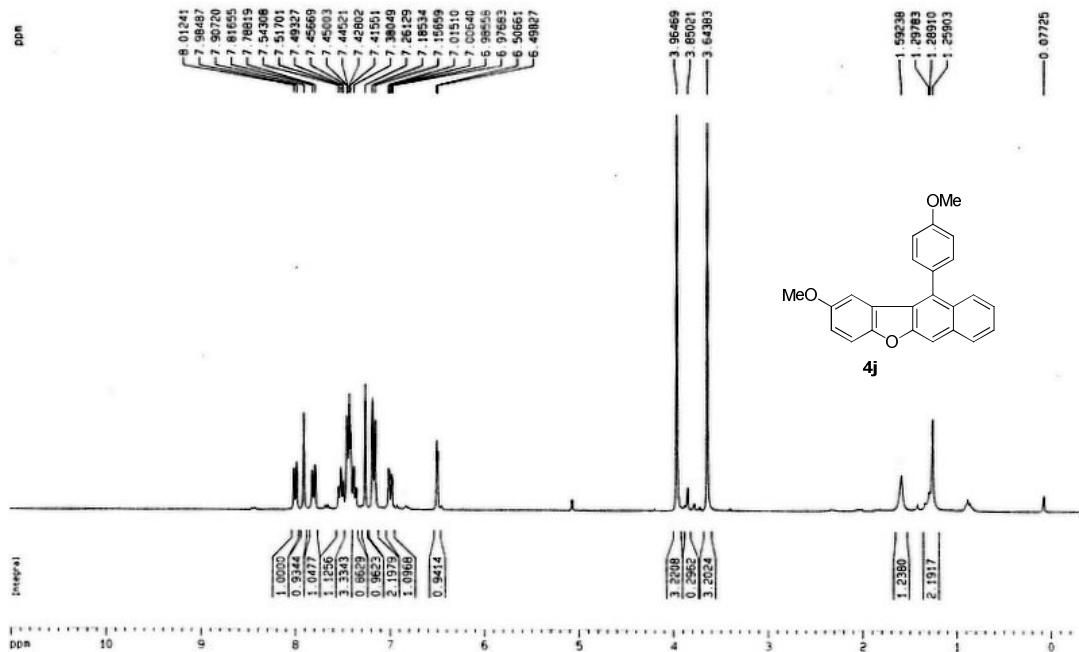
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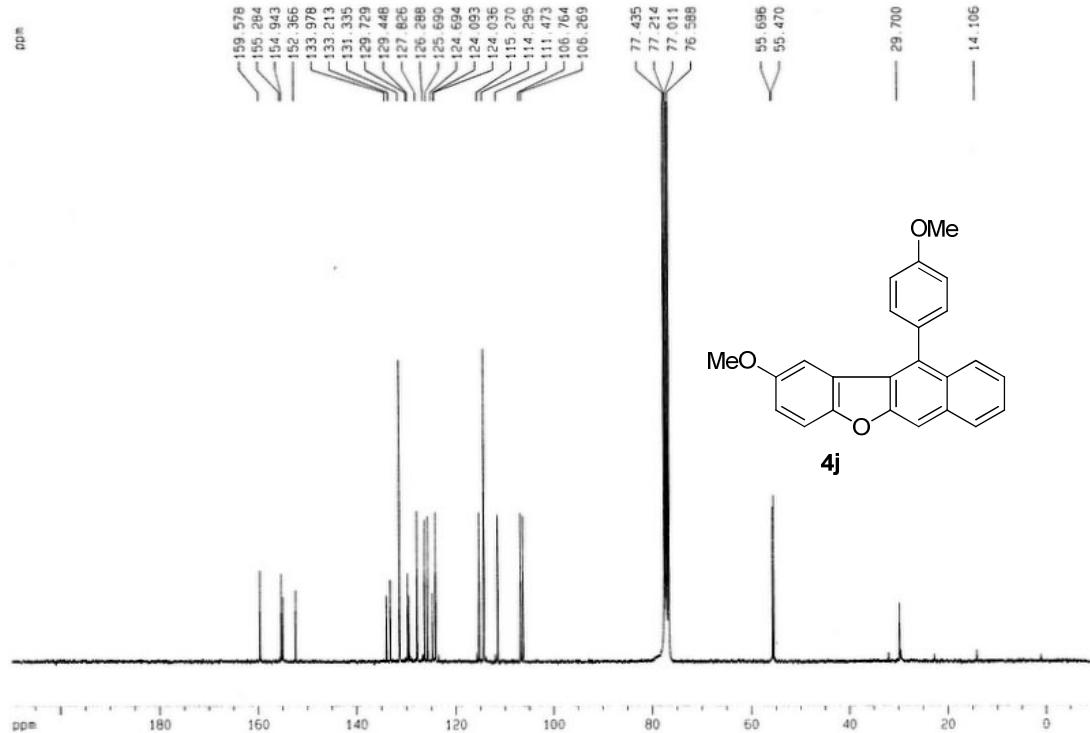
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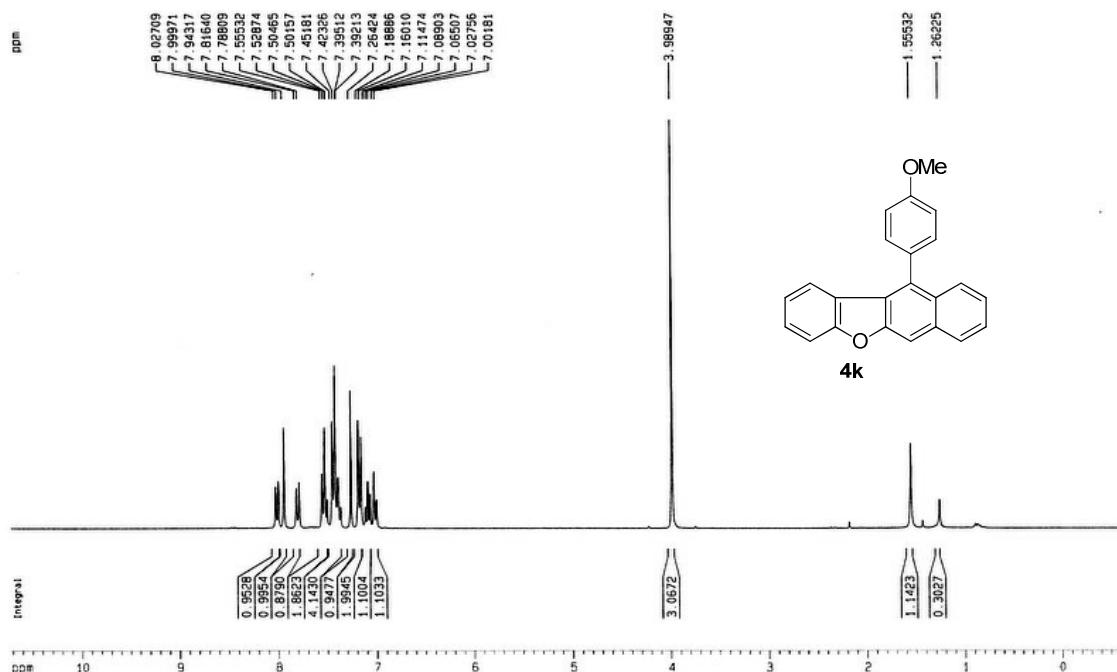
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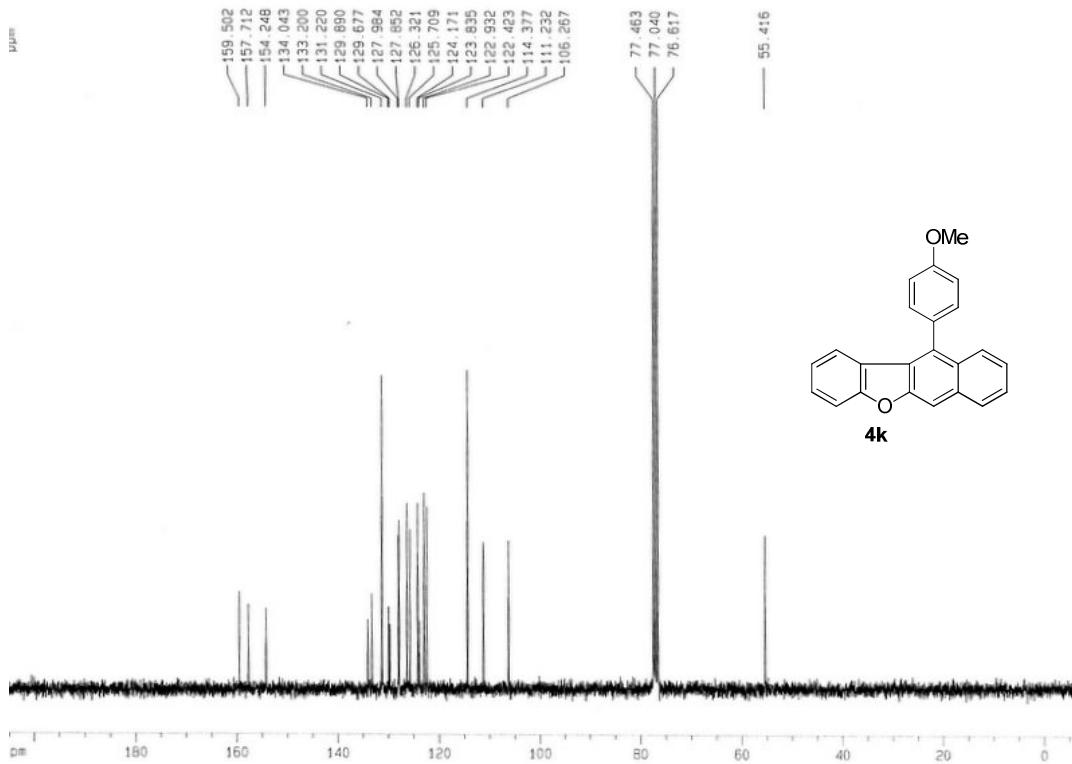
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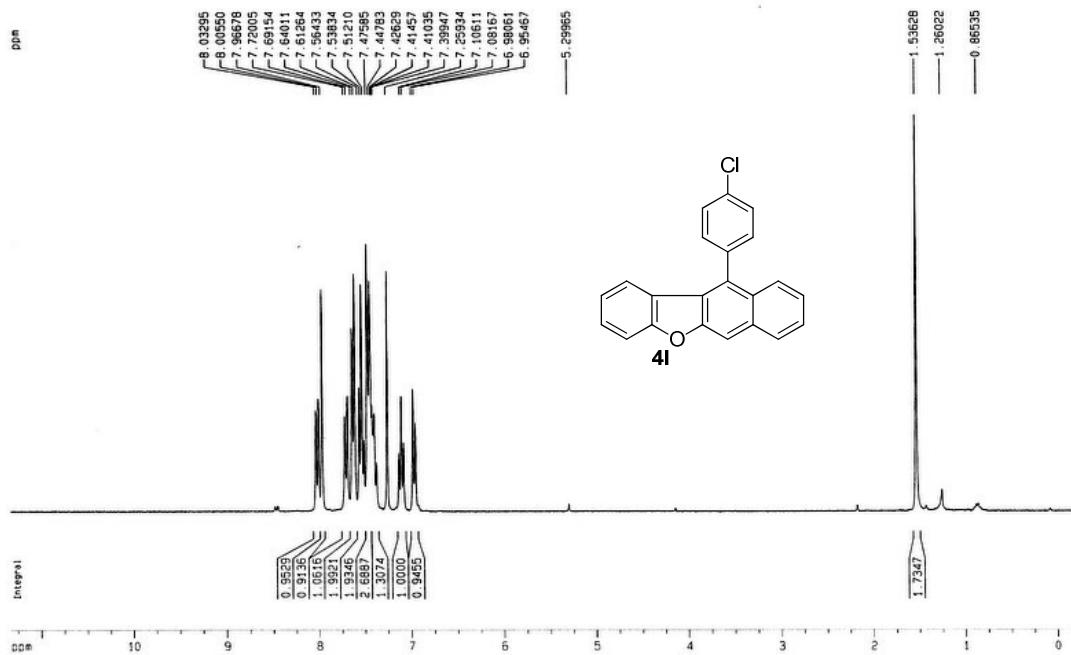
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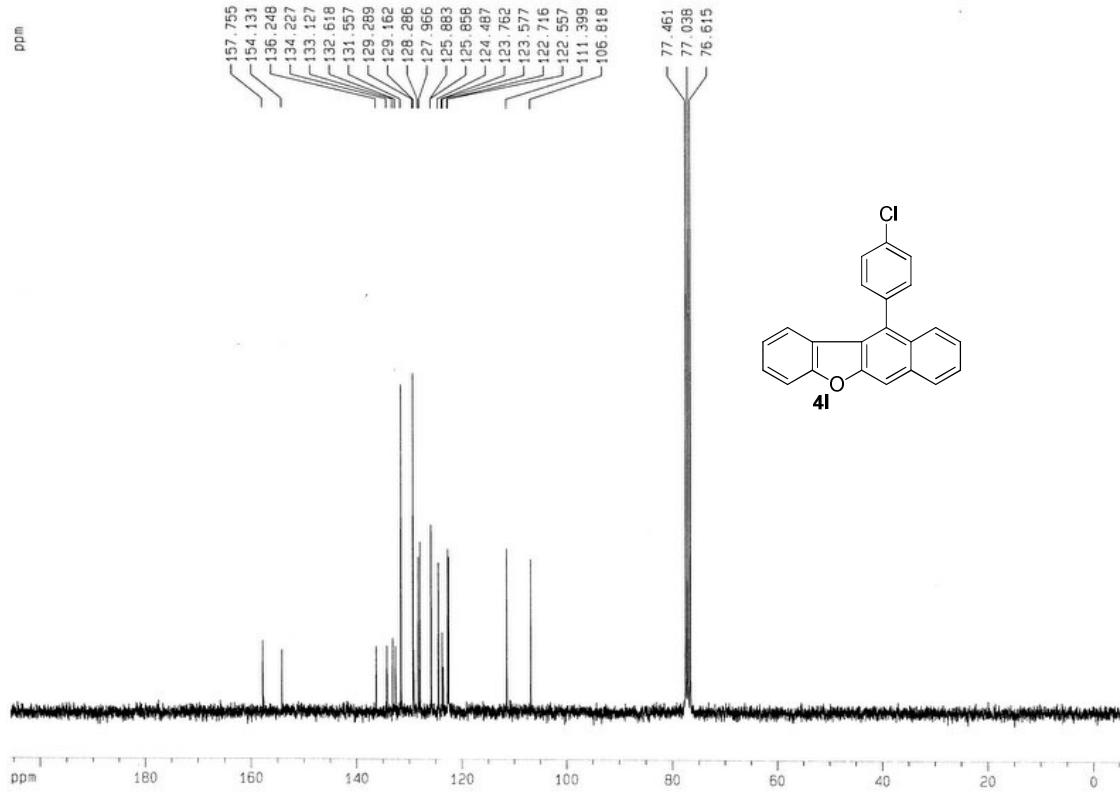
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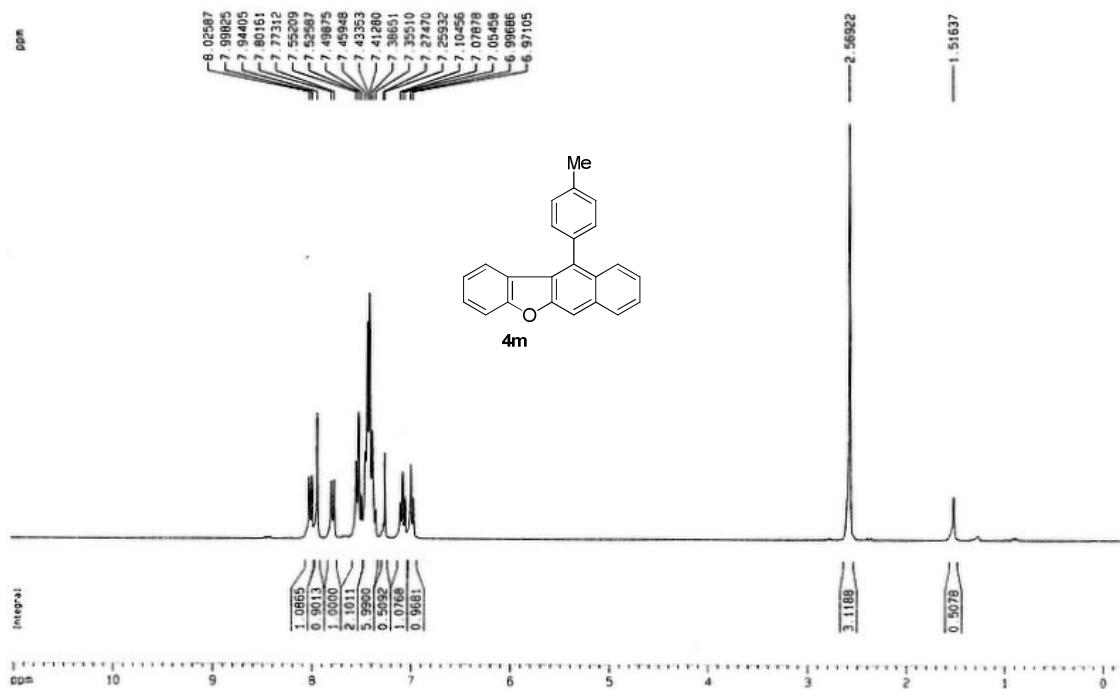
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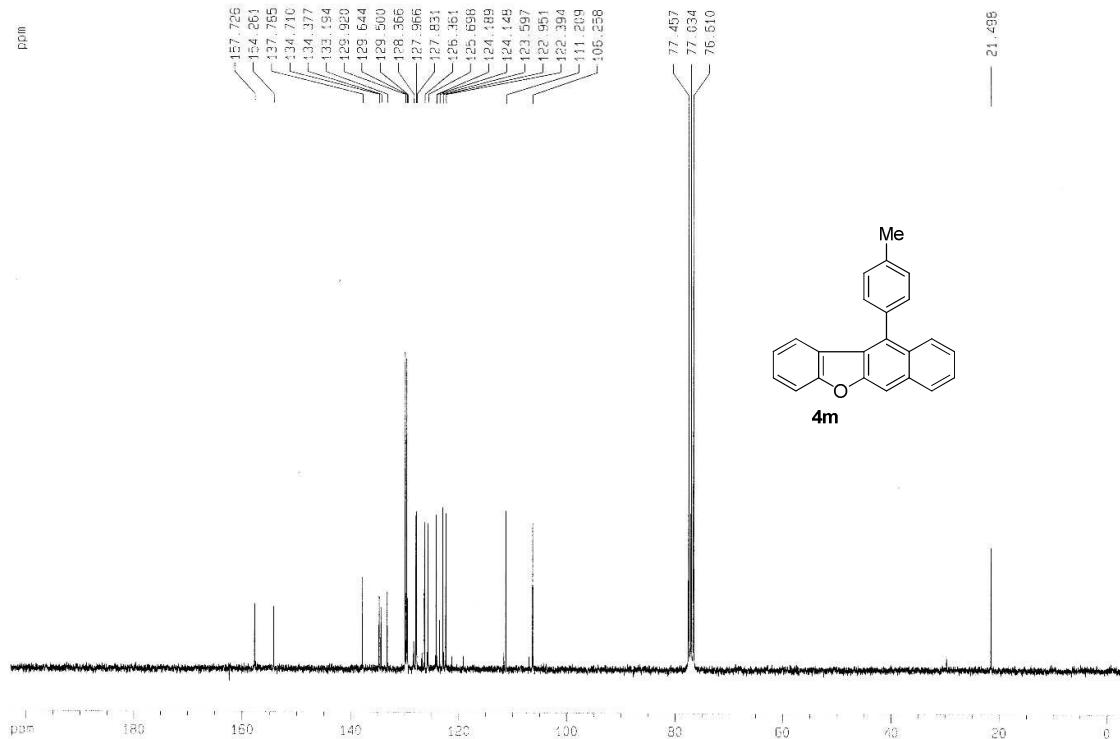
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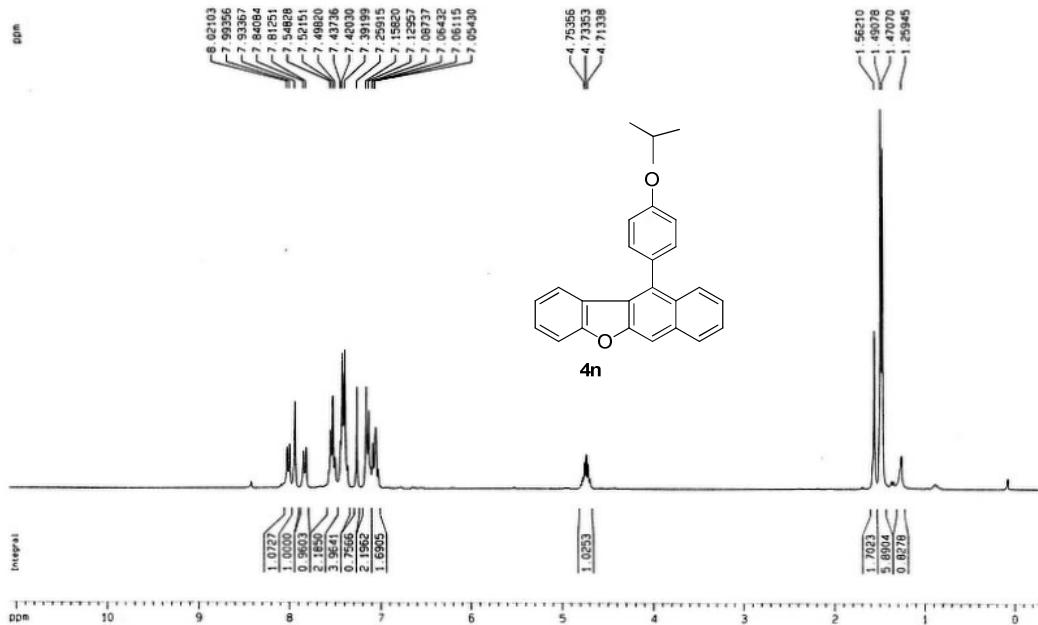
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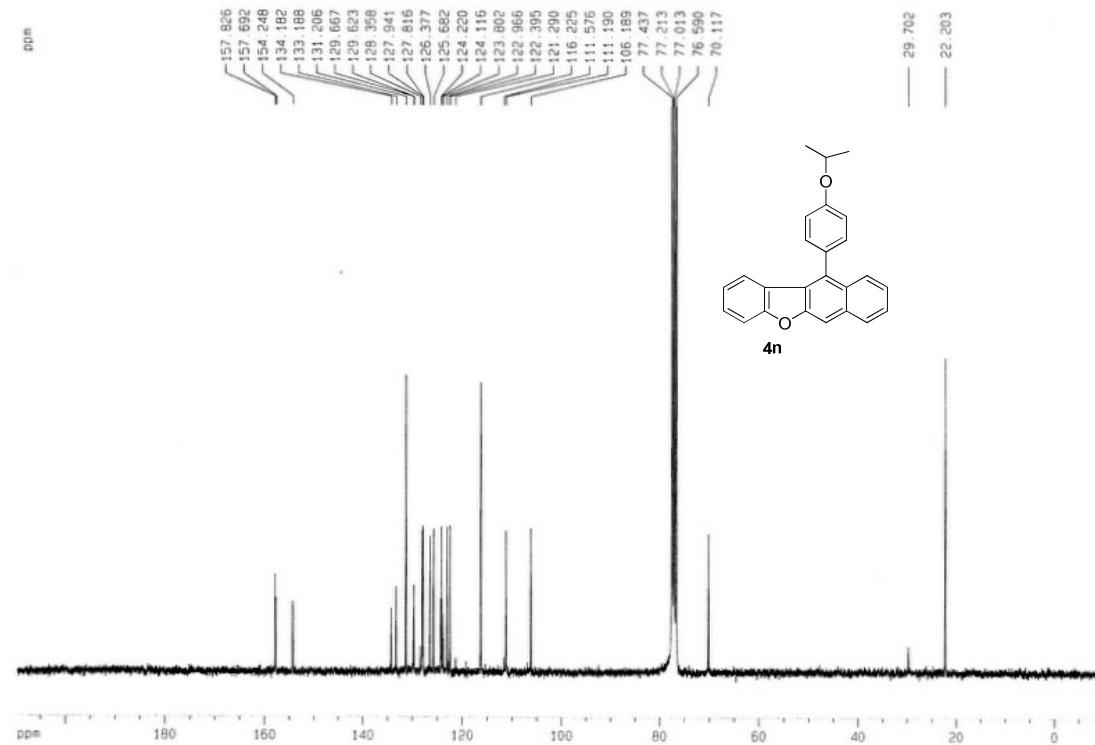
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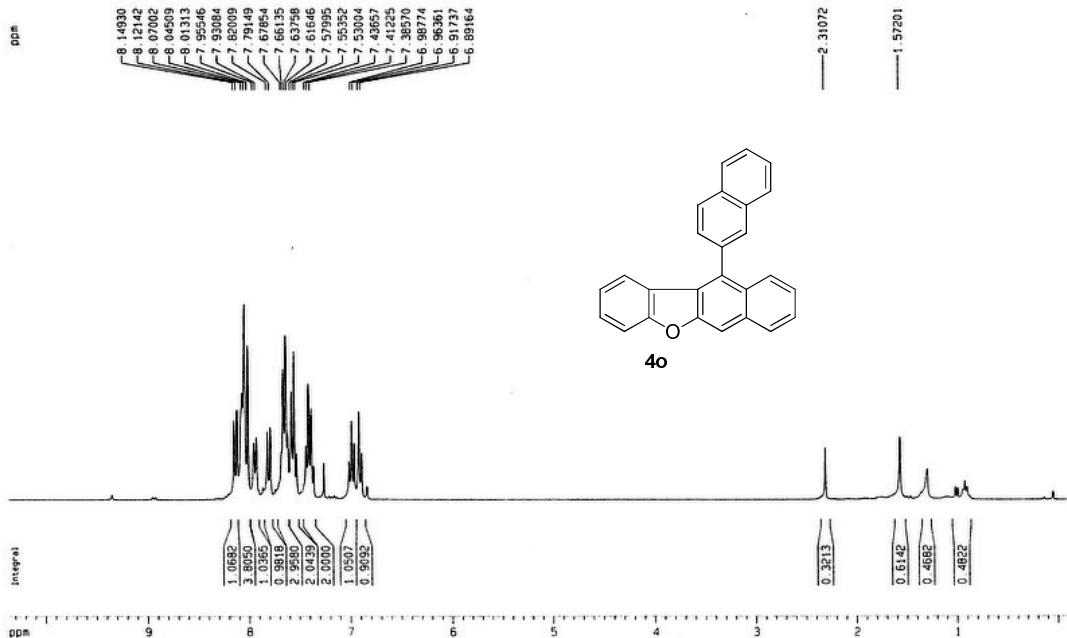
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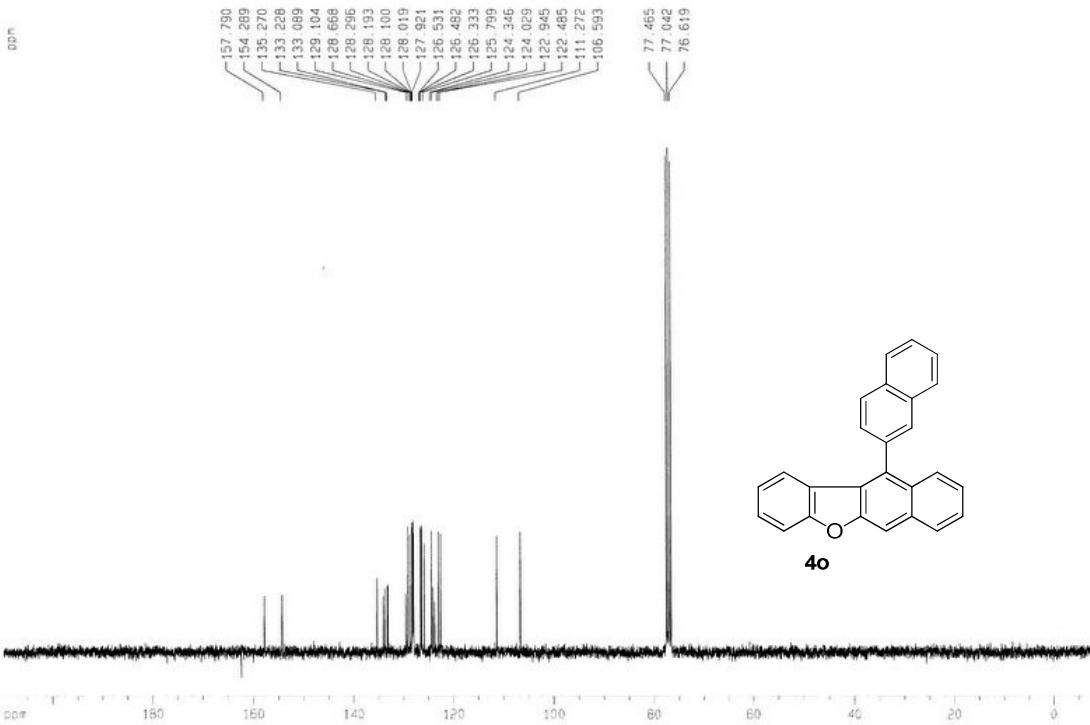
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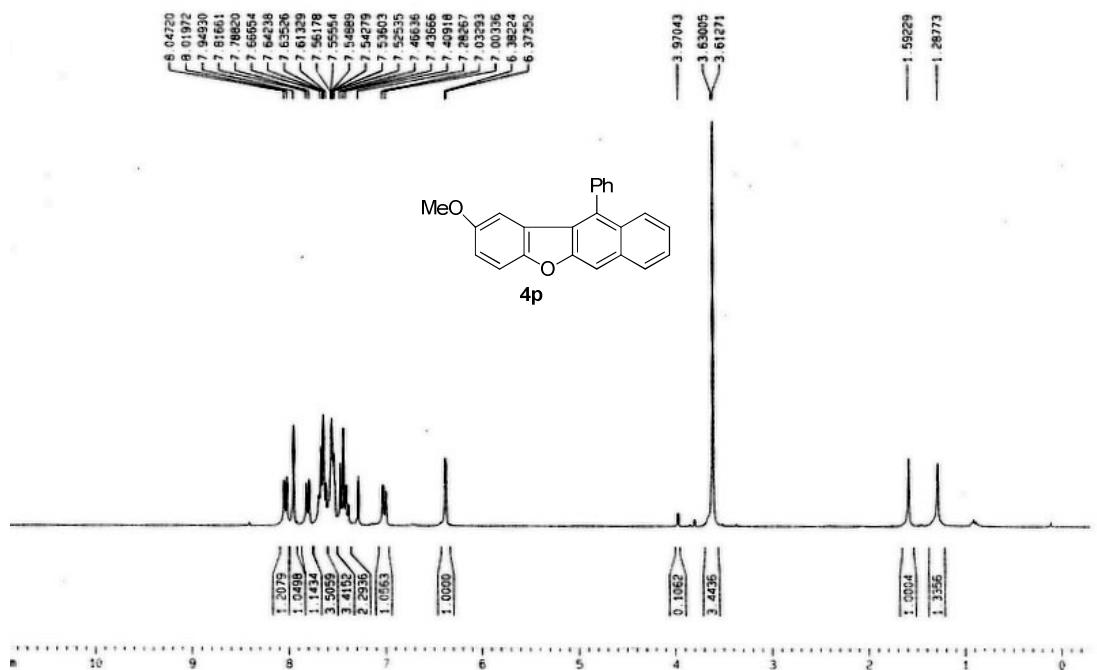
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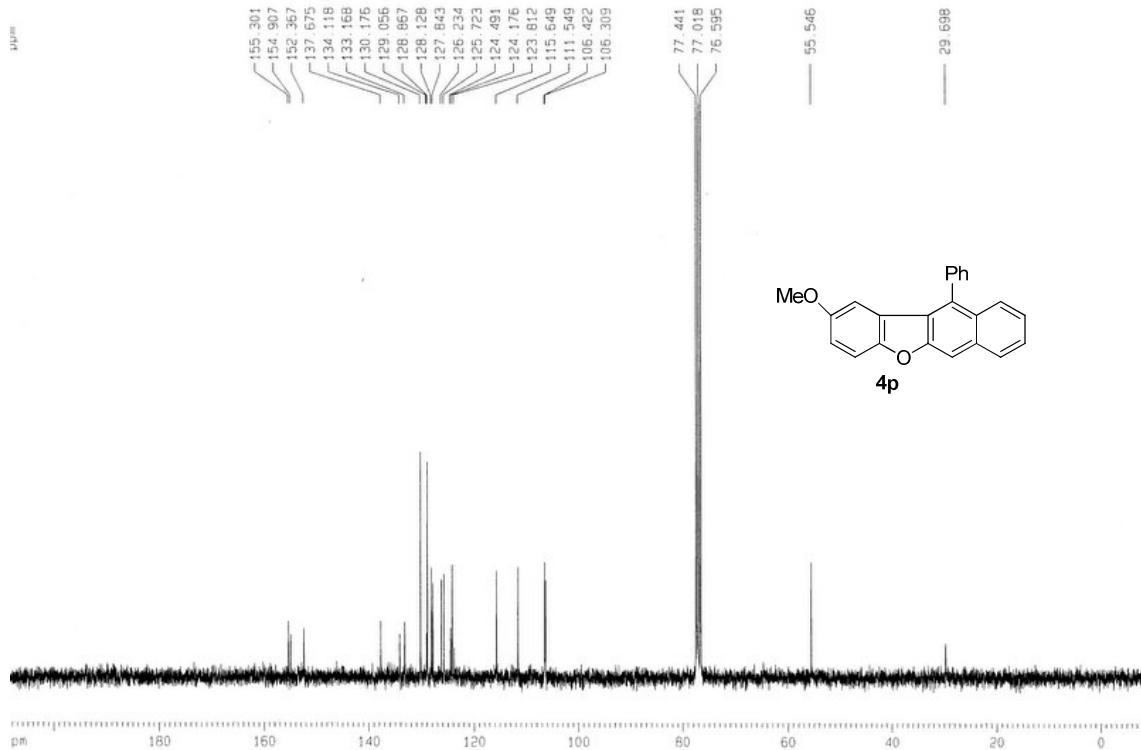
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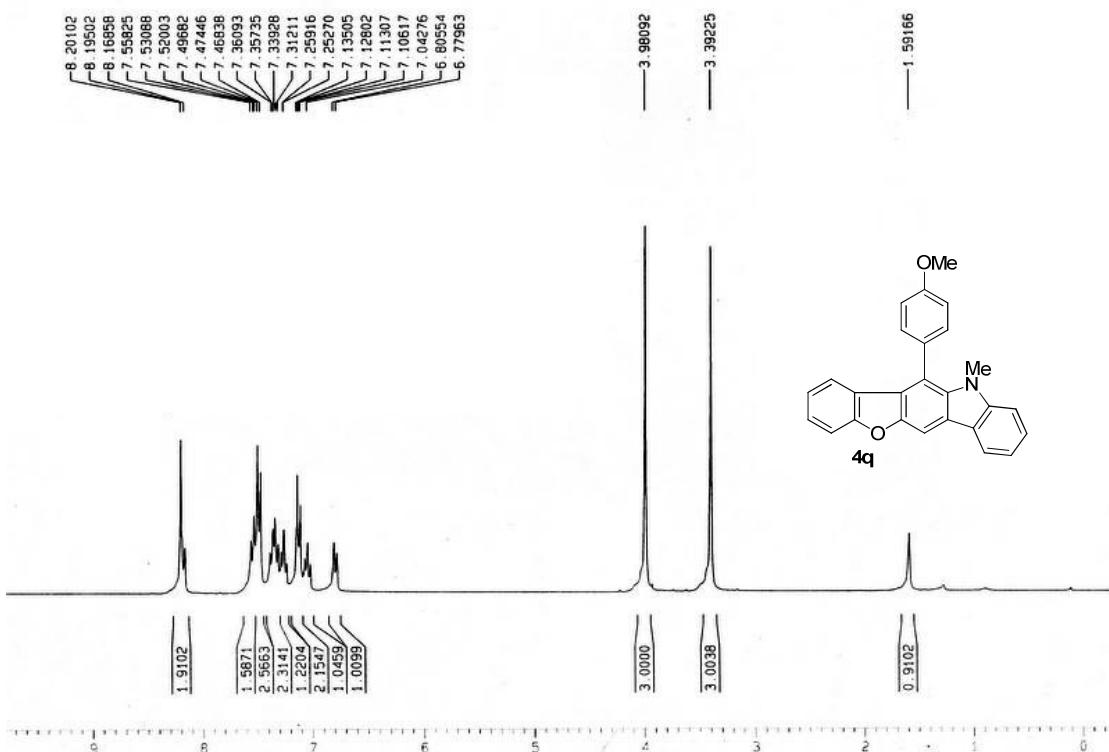
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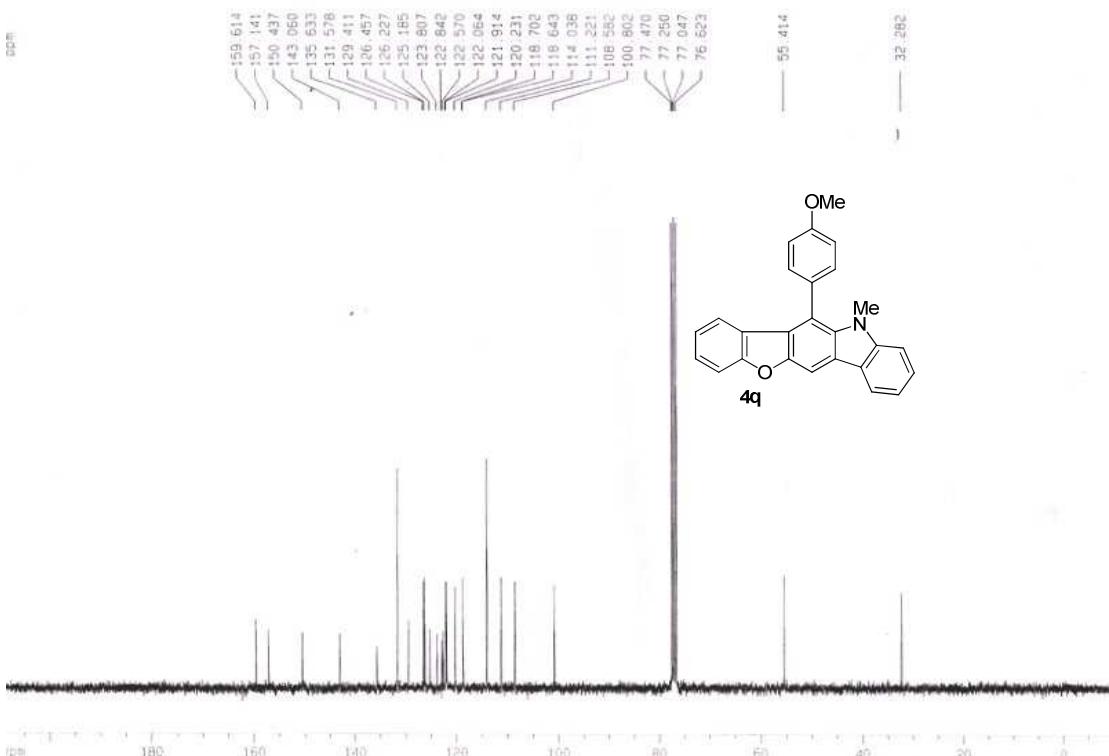
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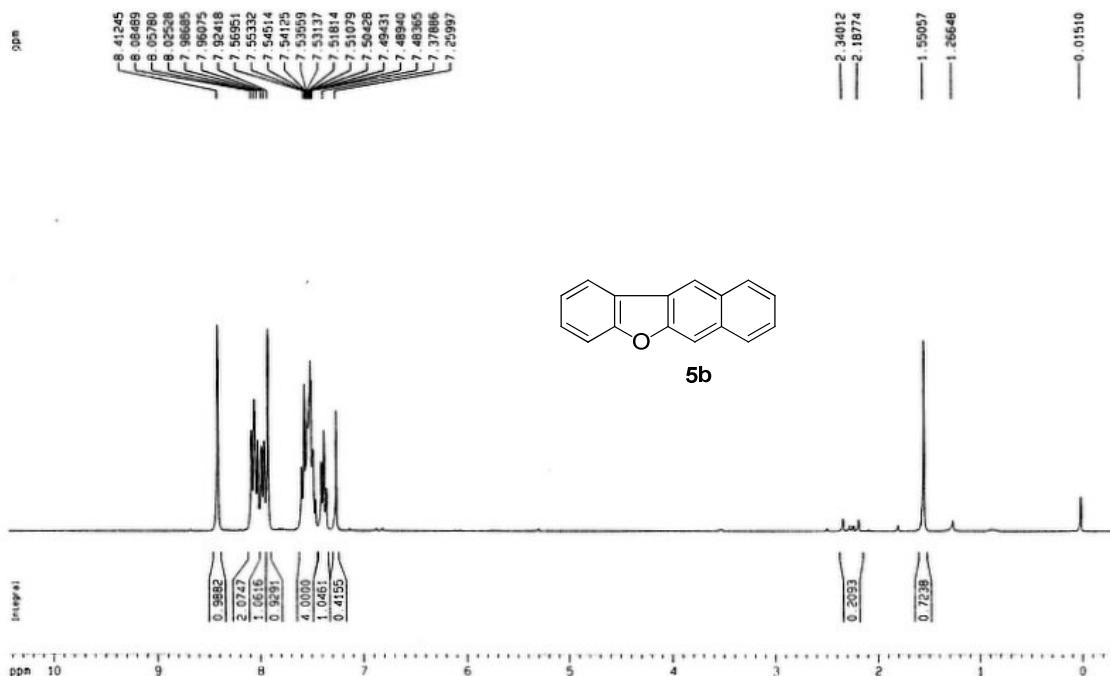
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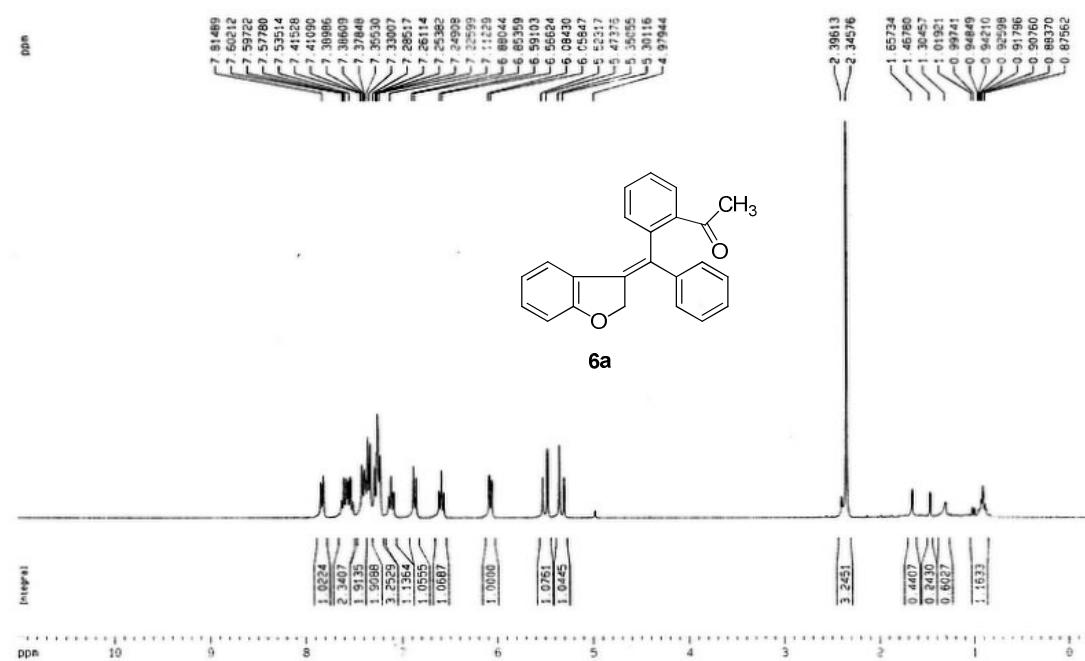
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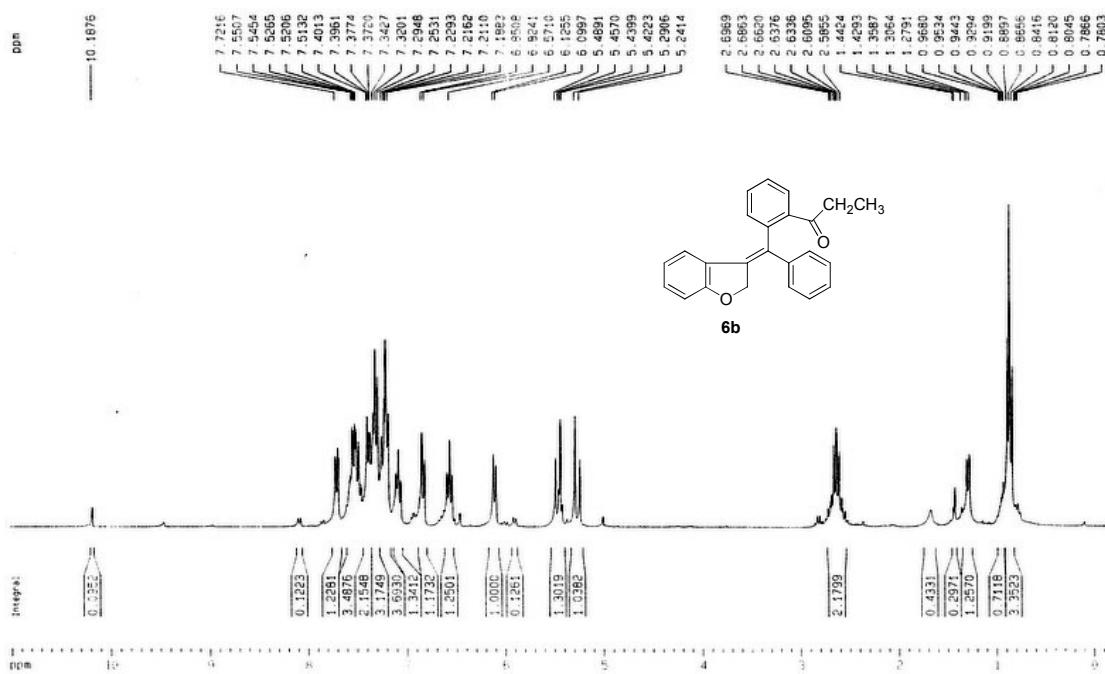
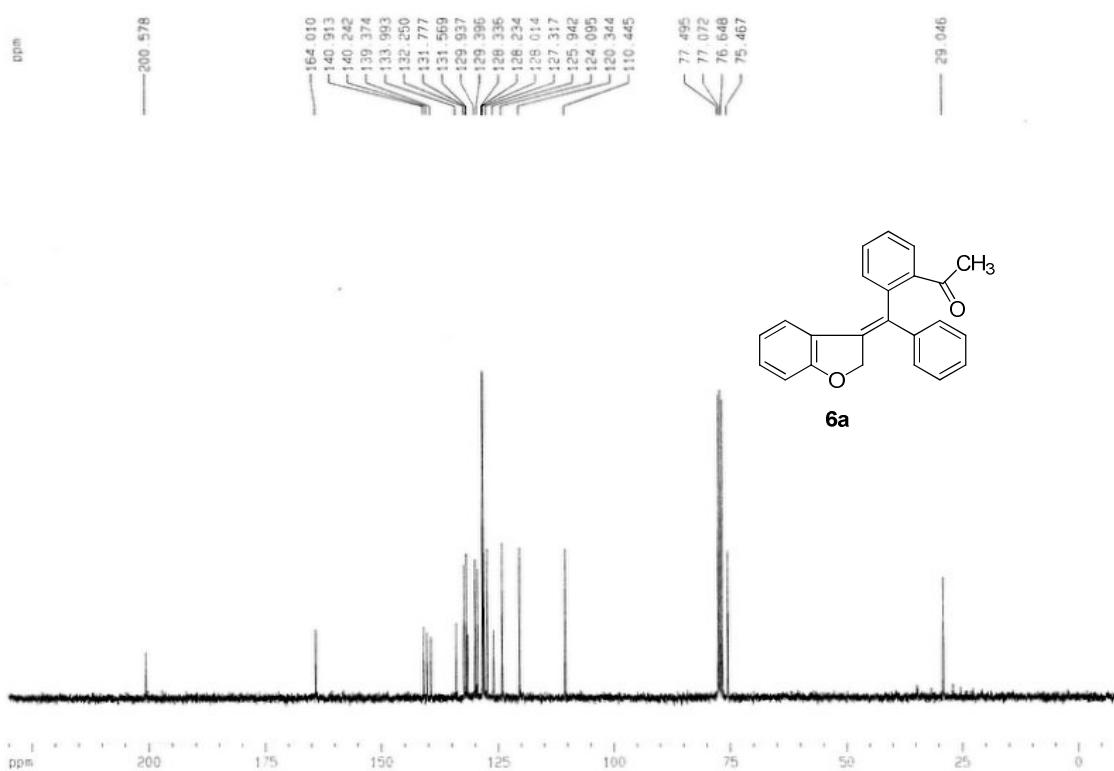
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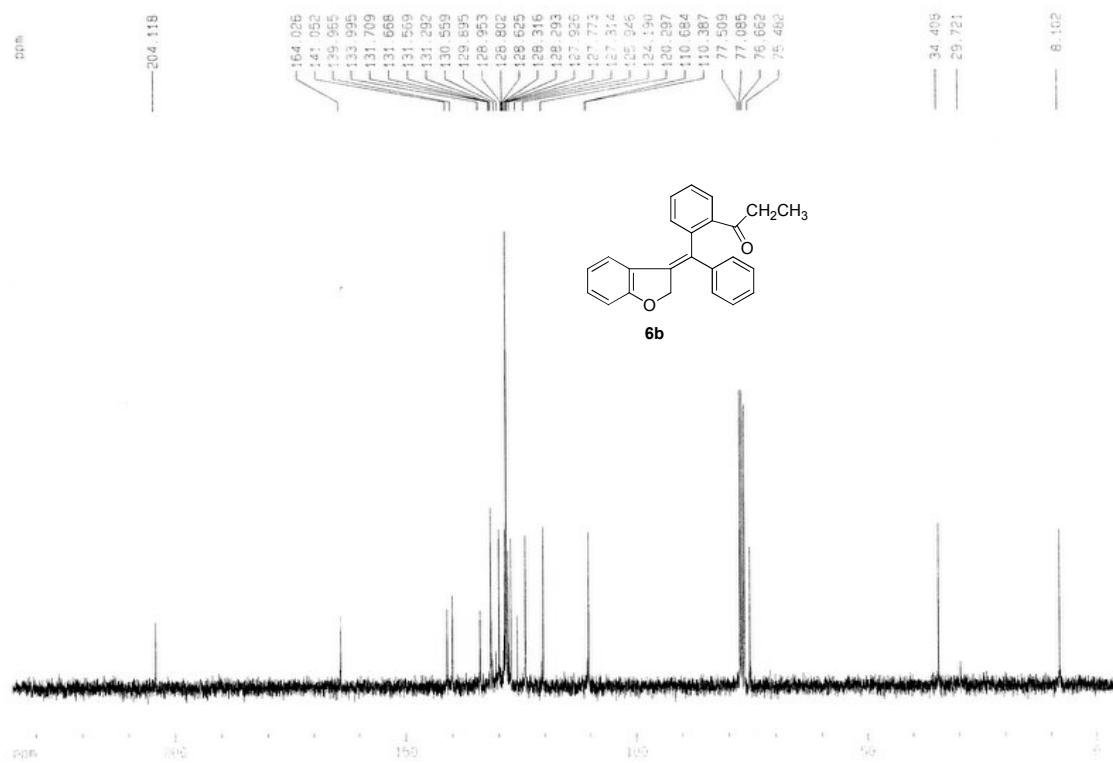
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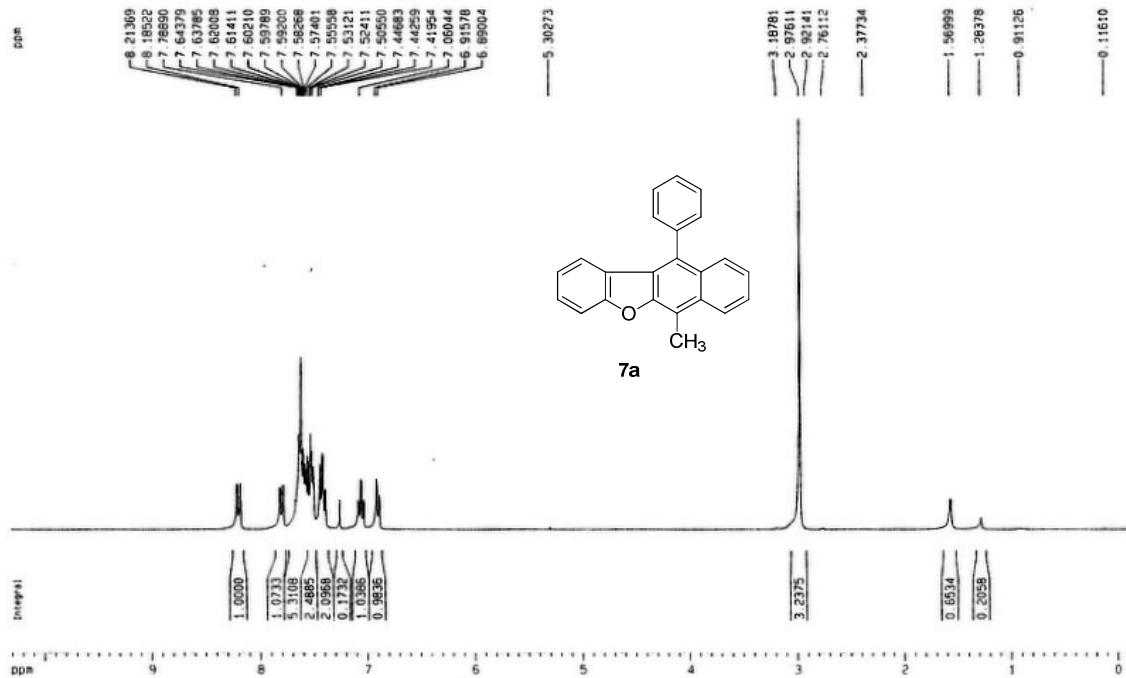
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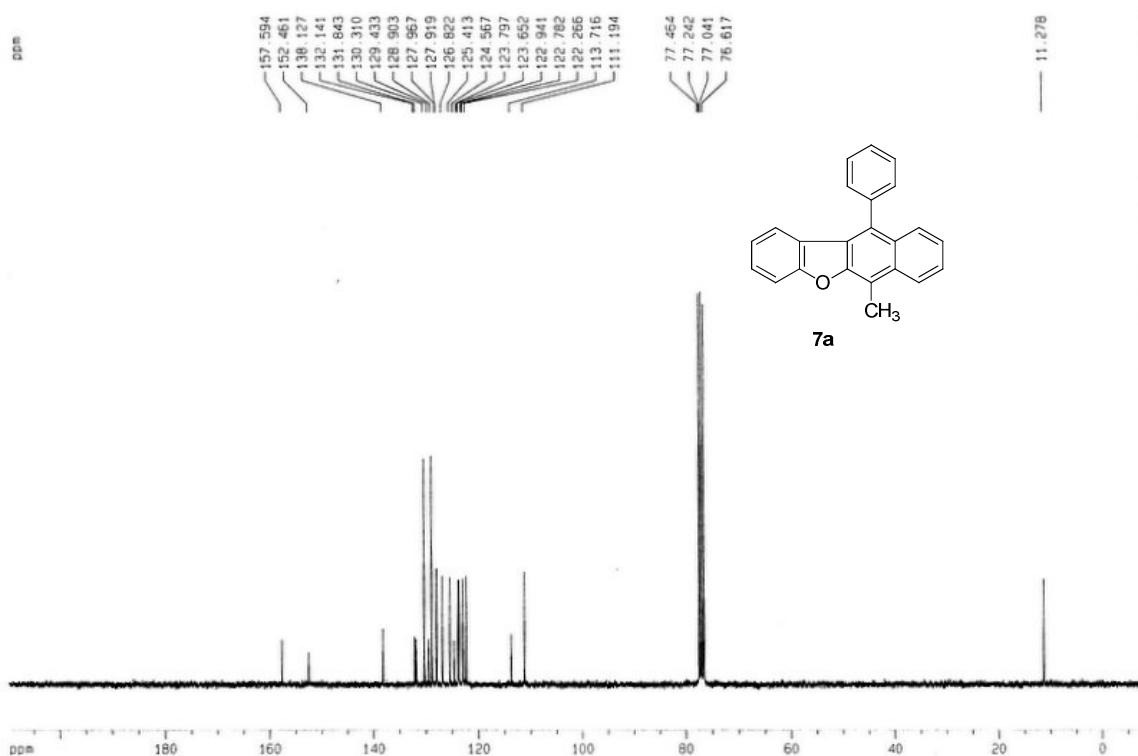
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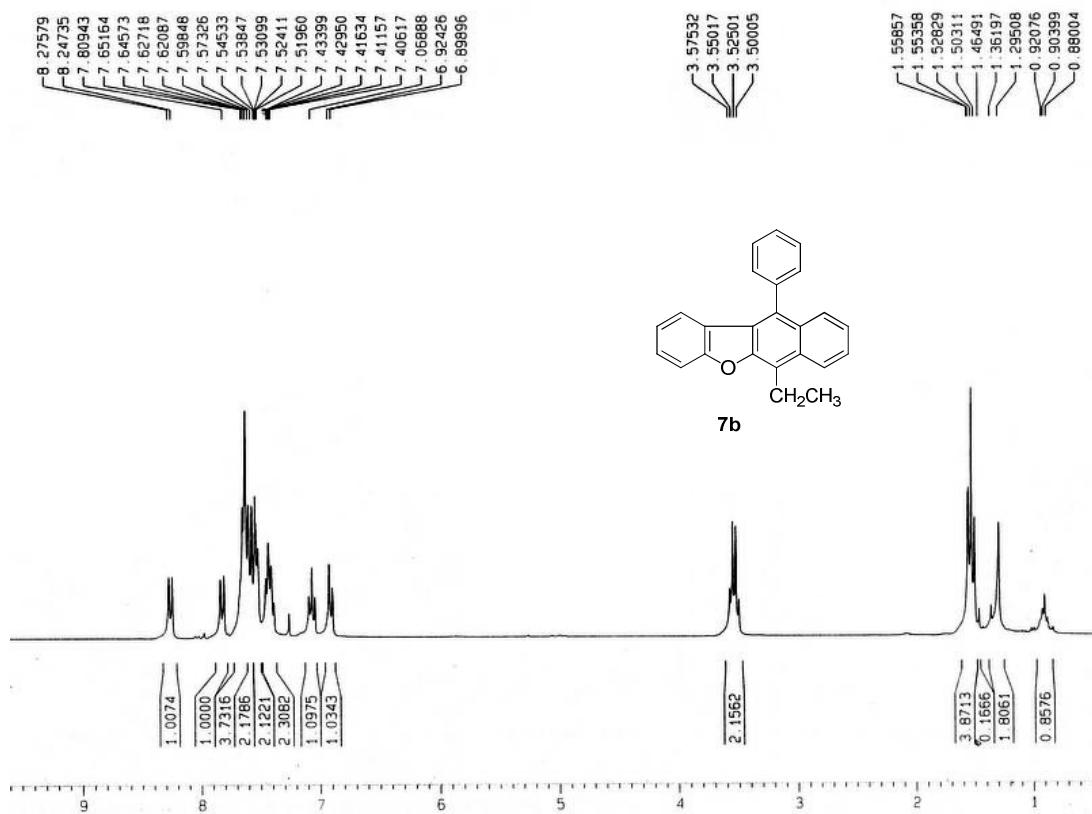
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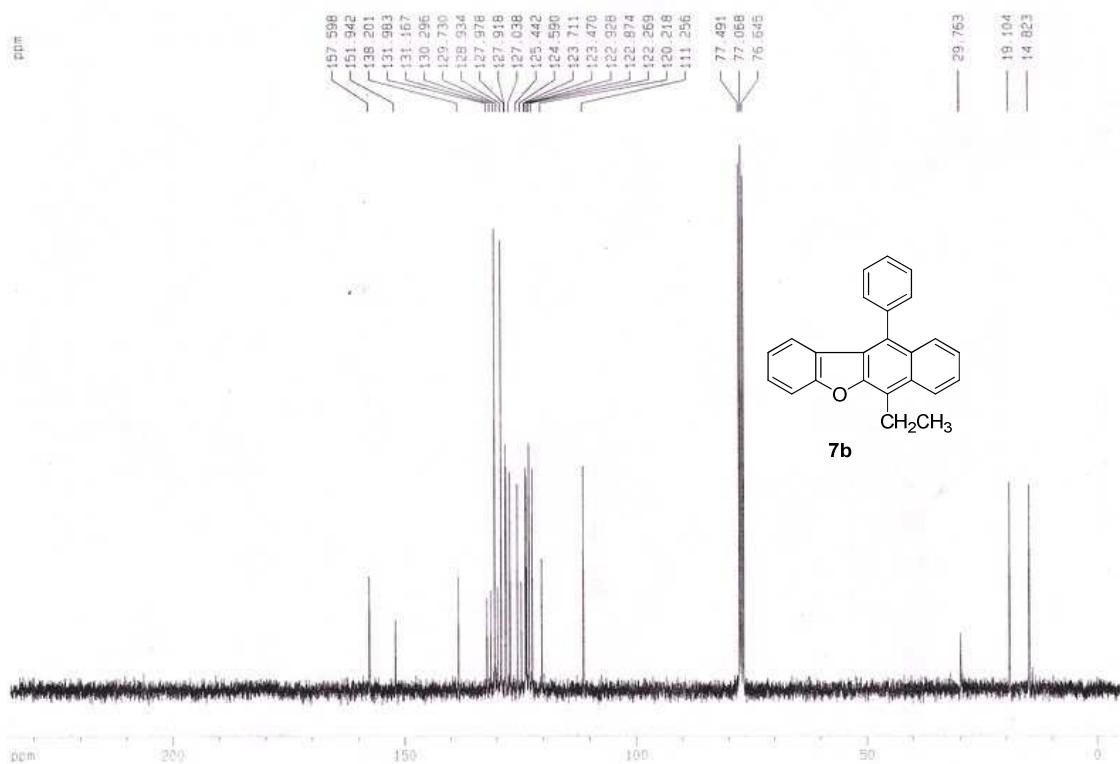
¹³C NMR of **7a**, CDCl₃, 75 MHz



¹H NMR of **7b**, CDCl₃, 300 MHz



¹³C NMR of **7b**, CDCl₃, 75 MHz



Crystallographic Data Collection and Refinement. The single crystal of compound **4a** was mounted on the tips of glass fibers with commercially available glue. X-ray data collection of **4a** was performed at room temperature using diffractometer equipped with a normal focus, sealed tube X-ray source with graphite monochromated Mo-K α radiation ($\lambda = 0.71073\text{\AA}$). The data were integrated using SAINT¹ program and the absorption corrections were made with SADABS.² The structure of **4a** was solved by SHELXS-97³ using Patterson method and followed by successive Fourier and difference Fourier synthesis. Full matrix least-squares refinements were performed on F² using SHELXL-97³ with anisotropic displacement parameters for all non-hydrogen atoms. All the hydrogen atoms were fixed geometrically by HFIX command and placed in ideal positions. Calculations were carried out using SHELXS 97,³ SHELXL 97,³ PLATON v1.15,⁴ ORTEP-3v2,⁵ and WinGX system Ver-1.80.⁶ Data collection and structure refinement parameters along with crystallographic data of **4a** are given in Table 1.

References

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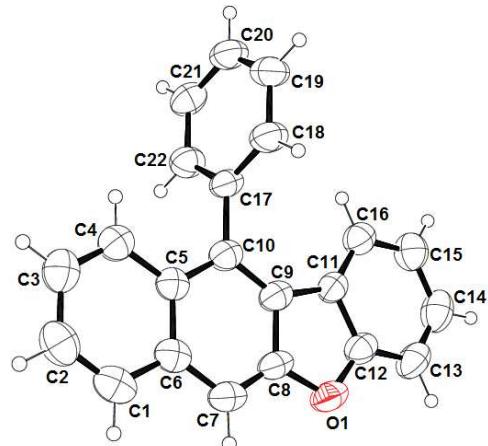
Table 1. Crystallographic and Structural Refinement Parameters of **4a**.

Formula	C ₂₂ H ₁₄ O
Formula Weight	294.33
Crystal System	Triclinic
Space group	P $\bar{1}$
a/ Å	9.1784(2)
b/ Å	10.2343(2)
c/ Å	10.5109(3)
$\alpha/^\circ$	98.755(2)
$\beta/^\circ$	113.273(2)
$\gamma/^\circ$	114.440(1)
V/ Å ³	764.64(4)
Z	2
$D_c/\text{g cm}^{-3}$	1.278
μ/mm^{-1}	0.077
F(000)	308
θ range/°	2.3-27.5
Reflections collected	12329

Unique reflections	3462
Reflections $I > 2\sigma(I)$	2110
R_{int}	0.026
goodness-of-fit (F^2)	1.03
$R1 (I > 2\sigma(I))$ ^[a]	0.0509
$wR2(I > 2\sigma(I))$ ^[a]	0.1476

$$^{[a]} R_1 = \sum | |F_o| - |F_c| | / \sum |F_o|, wR_2 = [\sum (w(F_o^2 - F_c^2)^2) / \sum w(F_o^2)^2]^{1/2}$$

X-ray crystal structure of **4a** (CCDC 143499)



(50% ellipsoidal probability)