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A one-pot synthesis of 3-nitro-2*H*-thiopyrans and their selective reduction to 3-nitro-3,4-dihydro-2*H*-thiopyrans

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Supplementary Information

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1. Crystallographic data for compound 7.

Single crystals of compound **7** were grown by slow evaporation of its solution in acetone. A suitable crystal was selected and intensity data were collected on a SuperNova, Single source at offset/far, HyPix3000 diffractometer. The crystal was kept at 99.97(16) K during data collection. Using Olex2[1], the structure was solved with the SHELXS[2] structure solution program using Direct Methods and refined with the SHELXL[3] refinement package using Least Squares minimization.

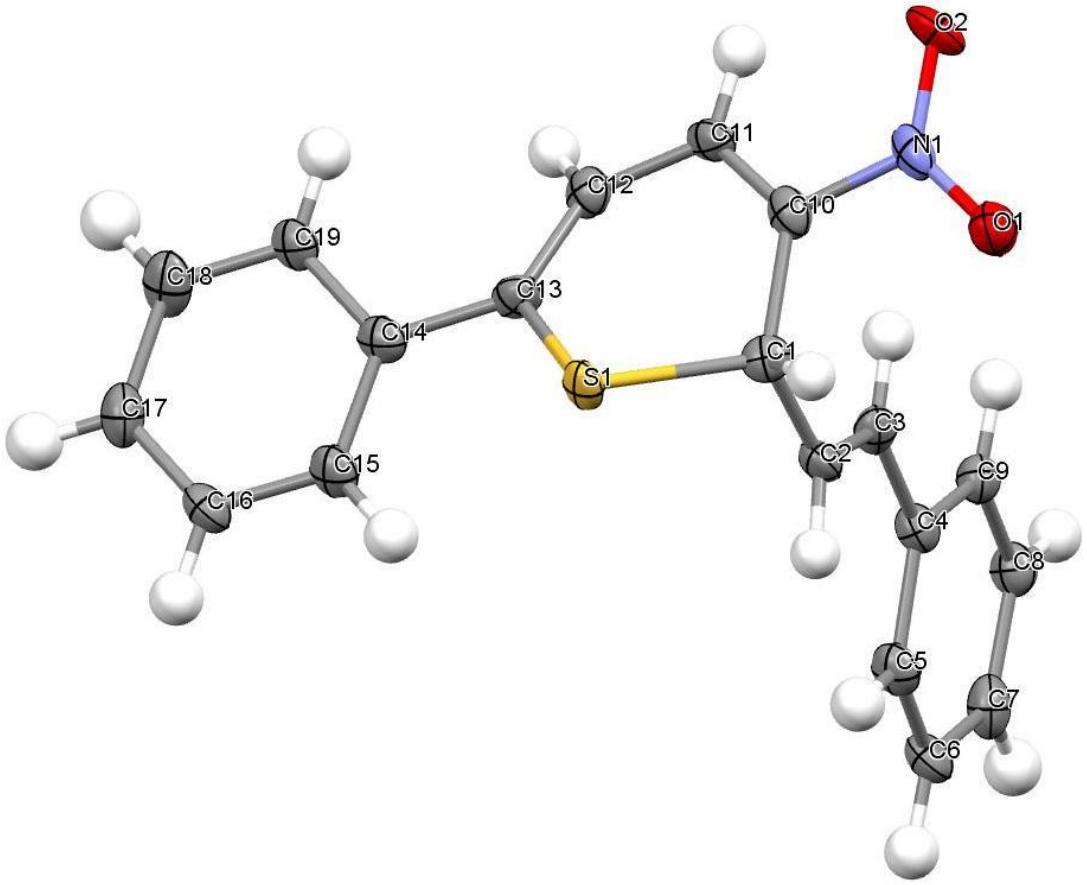


Figure S1. ORTEP representation of compound **7** displaying thermal ellipsoids at 50%.

Table S1. Crystal data and structure refinement for **7**

Identification code	**7**
Empirical formula	$C_{19}H_{15}NO_2S$
Formula weight	321.38
Temperature/K	99.97(16)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	6.3509(3)
b/Å	12.8407(6)
c/Å	18.9824(16)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1548.01(18)
Z	4
ρ_{calc}g/cm^3	1.379
μ/mm^{-1}	1.930
F(000)	672.0
Crystal size/mm^3	0.1 × 0.08 × 0.08
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.314 to 150.508
Index ranges	$-7 \leq h \leq 7$, $-15 \leq k \leq 15$, $-22 \leq l \leq 22$
Reflections collected	6171
Independent reflections	2685 [R_{int} = 0.0943, R_{sigma} = 0.0786]
Data/restraints/parameters	2685/0/190
Goodness-of-fit on F^2	1.074
Final R indexes [I>=2σ (I)]	R_1 = 0.0869, wR_2 = 0.2147
Final R indexes [all data]	R_1 = 0.1084, wR_2 = 0.2330
Largest diff. peak/hole / e Å^{-3}	0.63/-0.75
Flack parameter	0.05(7)
CCDC	**2310879**

References

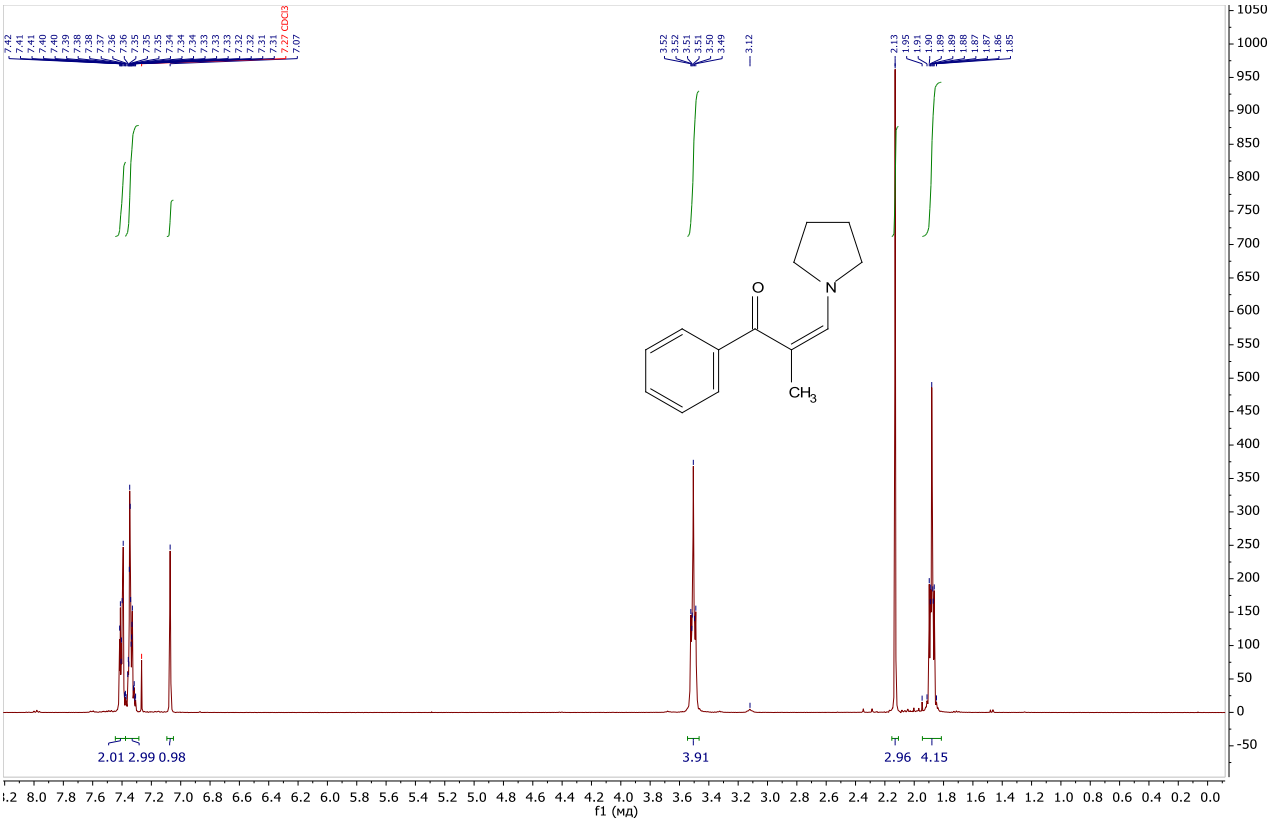
1. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. J. *Appl. Crystallogr*. **2009**, 42, 339. DOI: 10.1107/S0021889808042726.

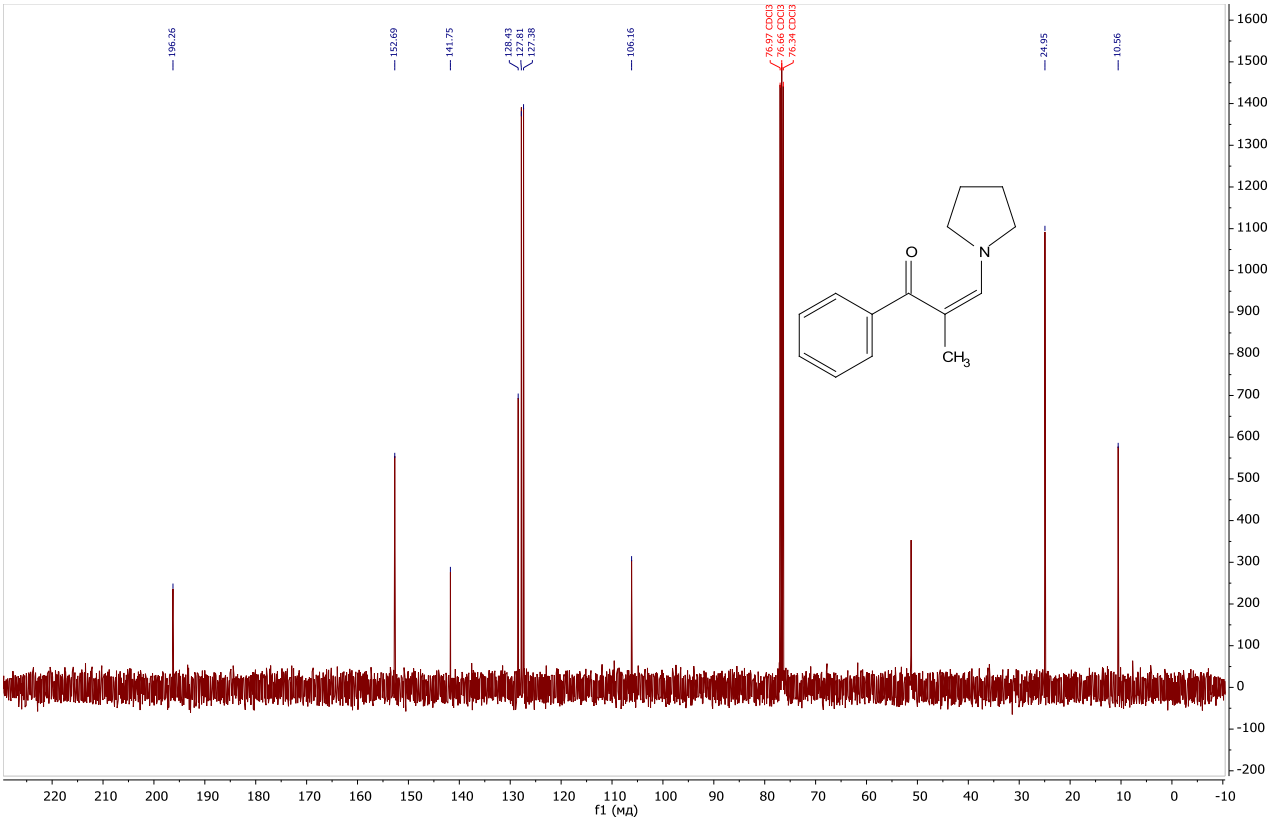
2. Sheldrick, G.M. *Acta Crystallogr., Sect. A.*. **2008**, 64, 112. DOI: 10.1107/S0108767307043930.

3. Sheldrick, G. M. *Acta Crystallogr., Sect. C:*. **2015**, 71, 3. DOI: 10.1107/S2053229614024218.

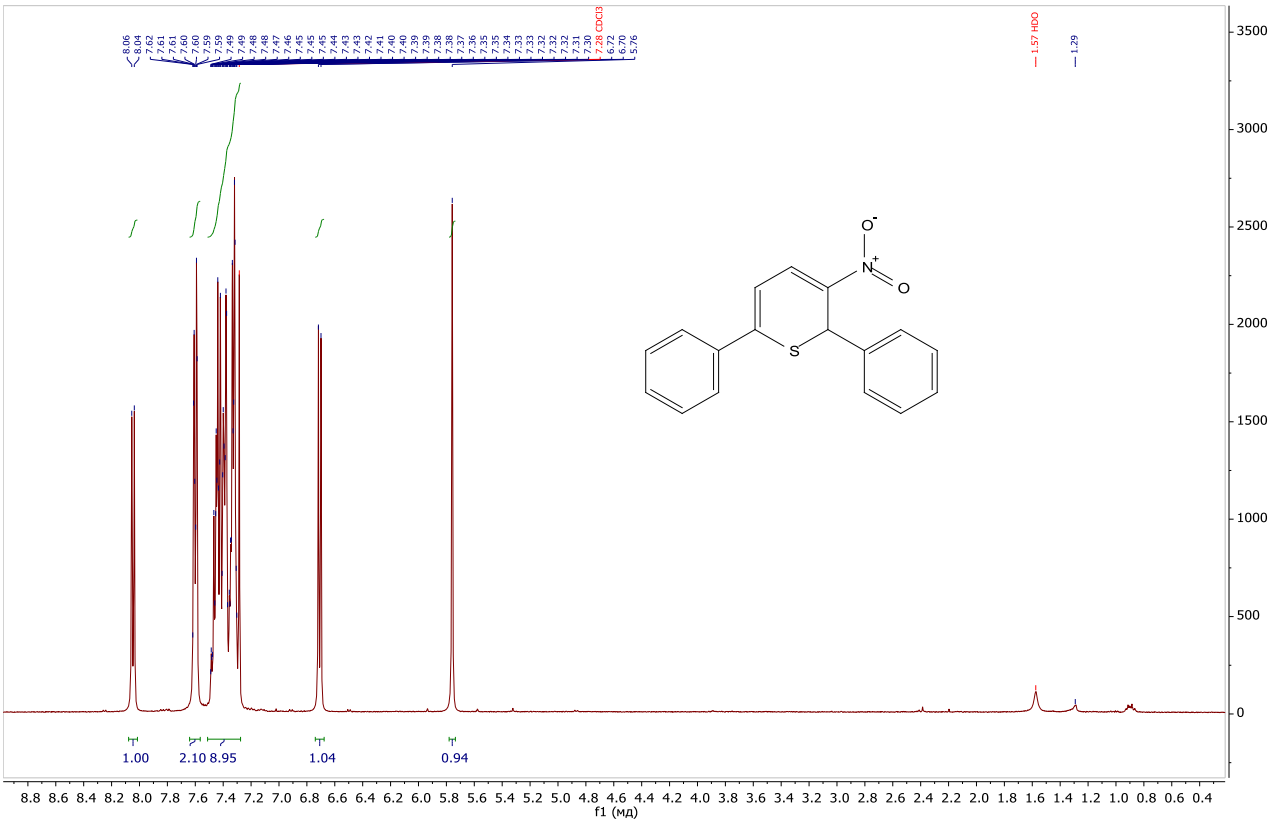
2. Copies of ^{1}H,^{13}C, ^{1}H-^{13}C-HSQC and ^{1}H-^{13}C-HMBC NMR spectra

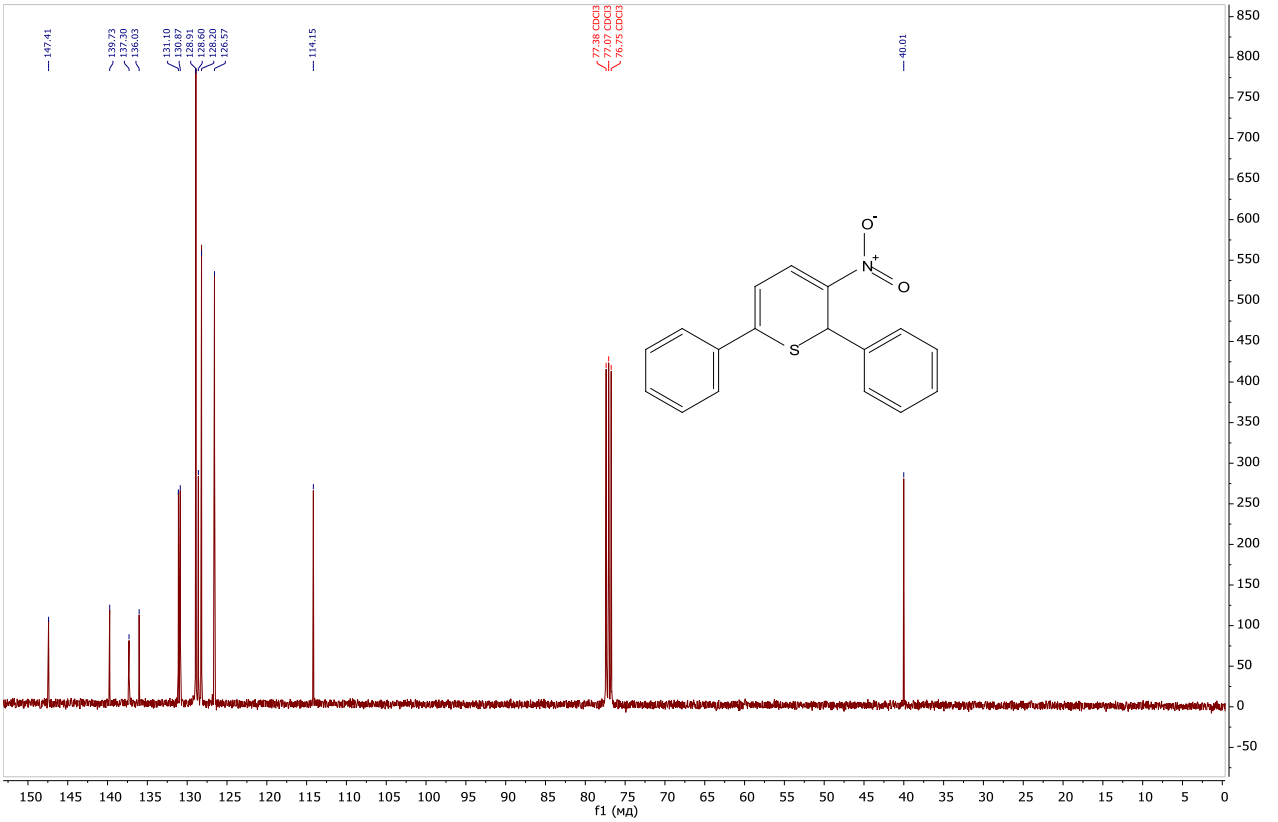
^{1}H and ^{13}C NMR spectra of compound **1c** ($CDCl_3$)



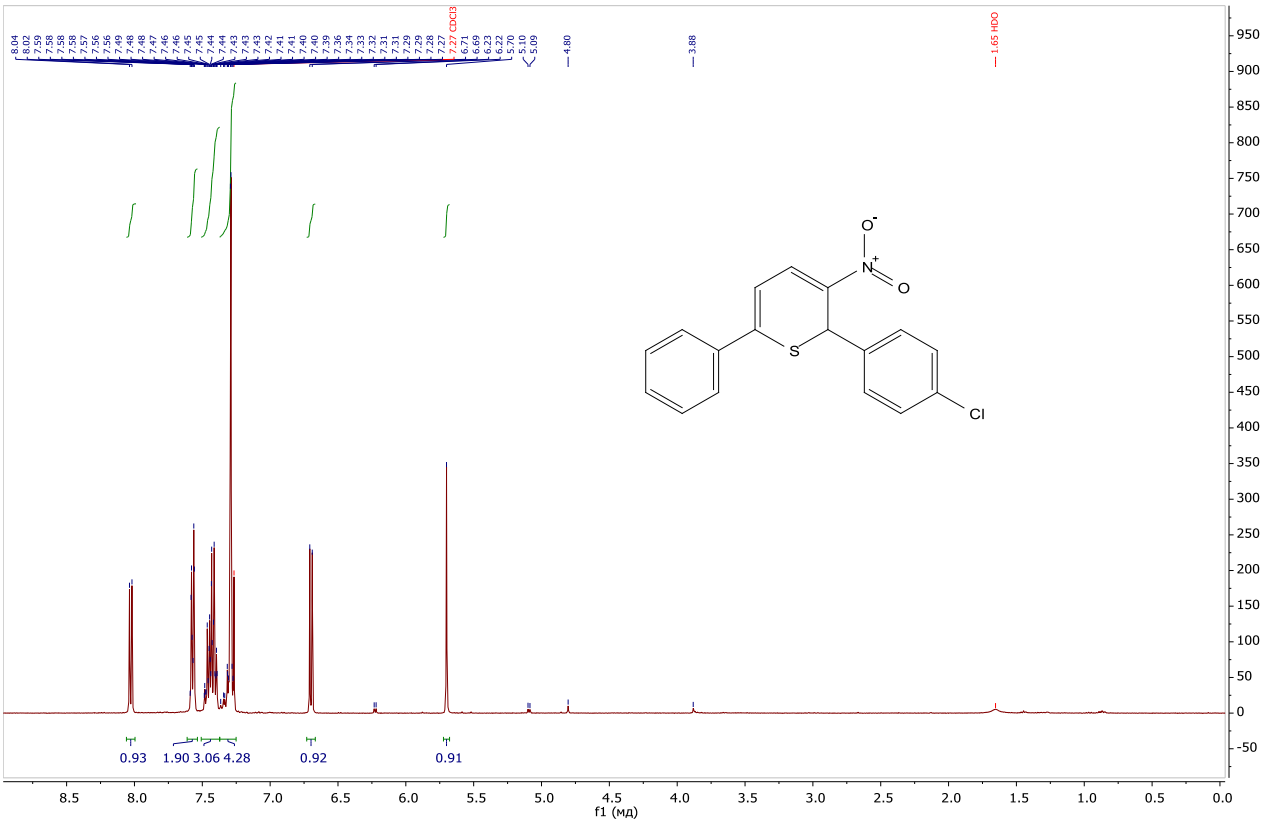


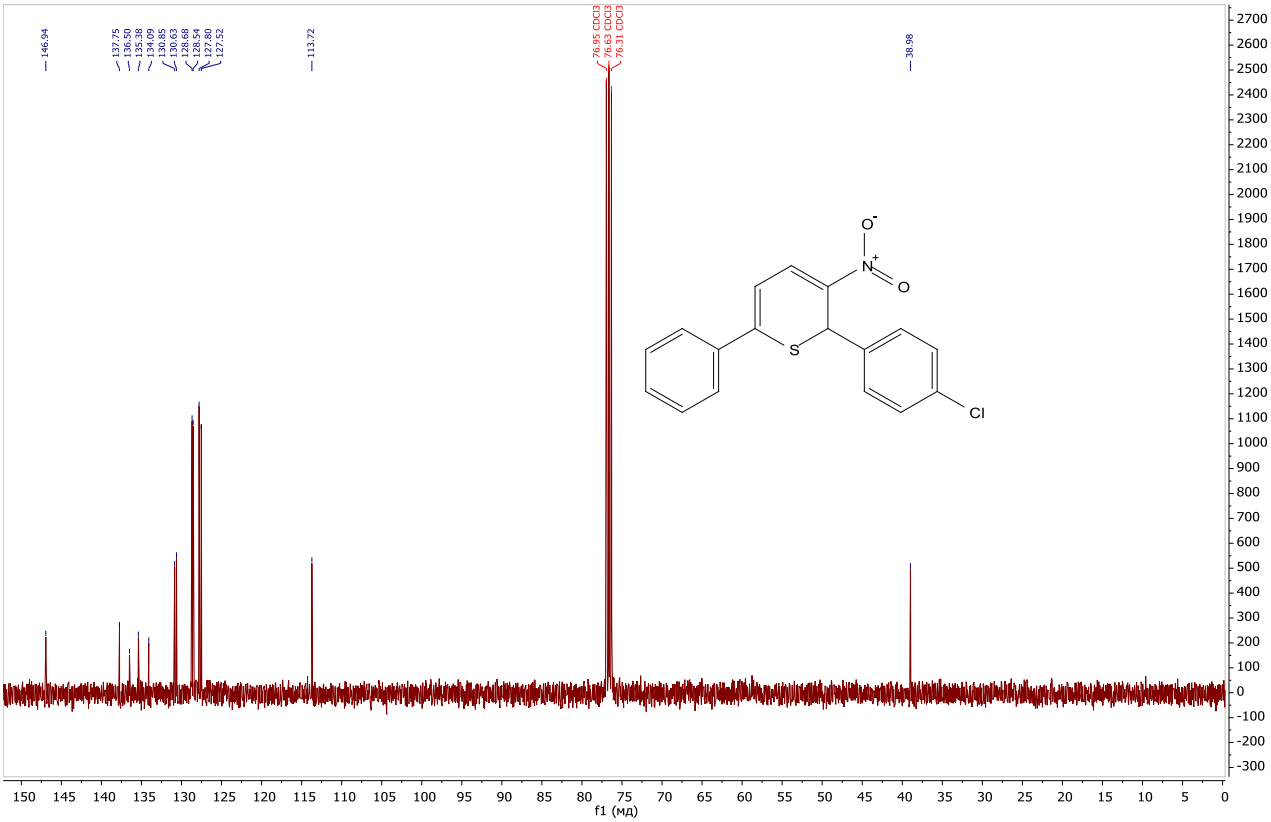
^{1}H and ^{13}C NMR spectra of compound **5a** ($CDCl_3$)



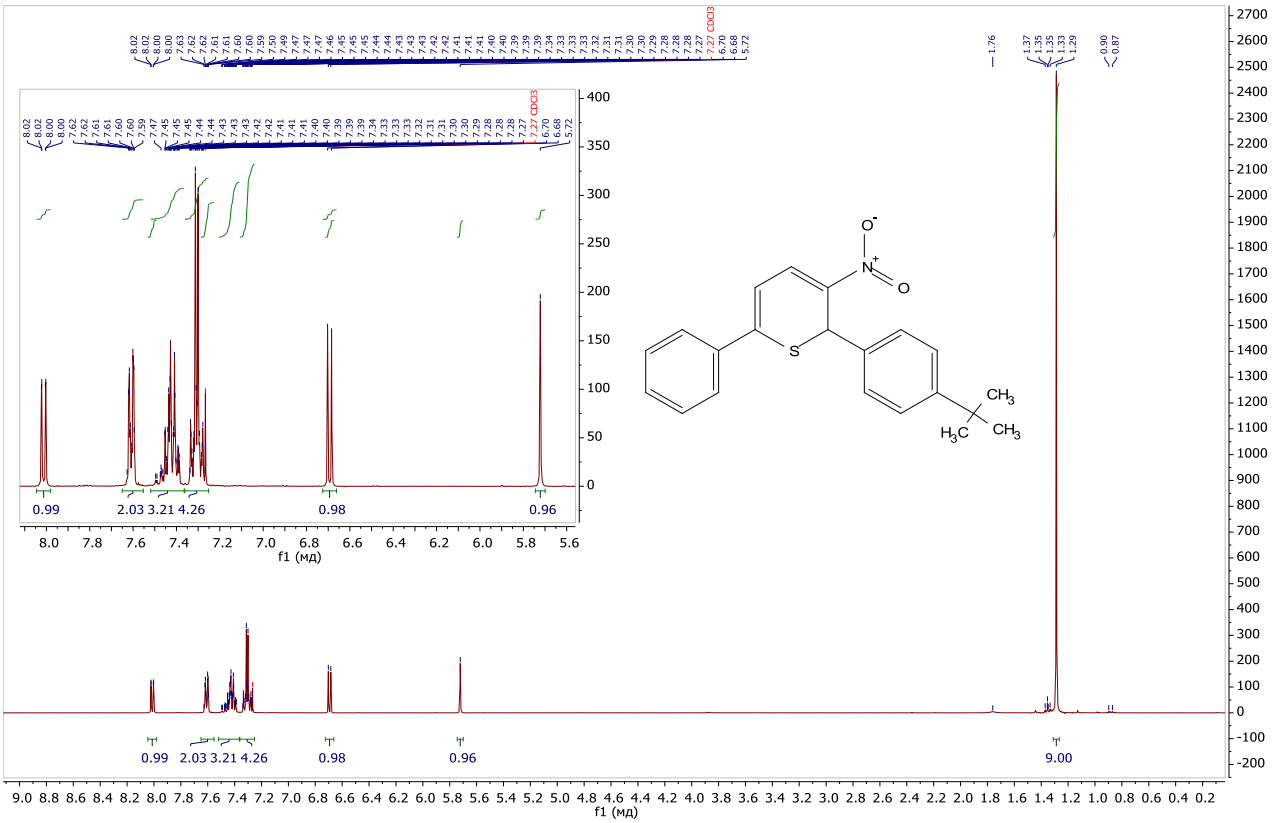


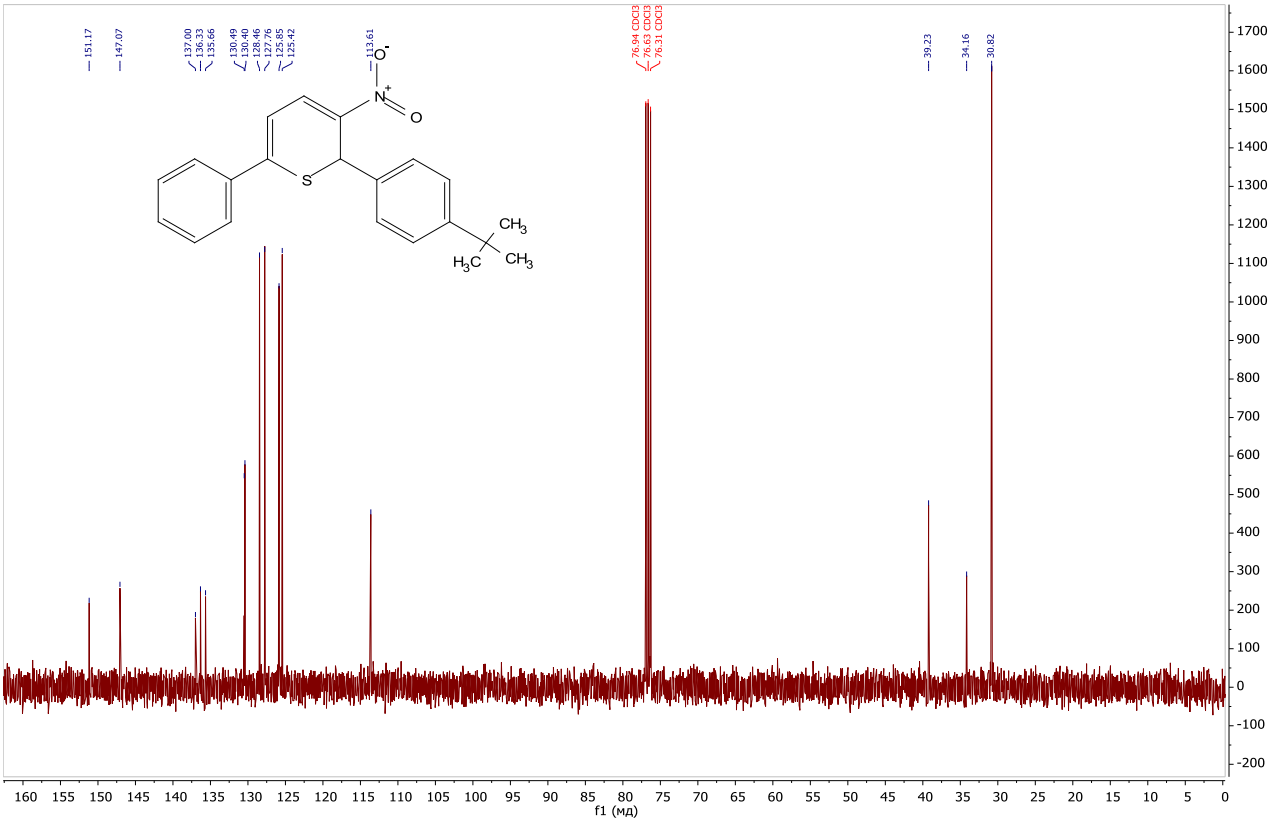
^{1}H and ^{13}C NMR spectra of compound **5b** ($CDCl_3$)





^{1}H and ^{13}C NMR spectra of compound **5c** ($CDCl_3$)





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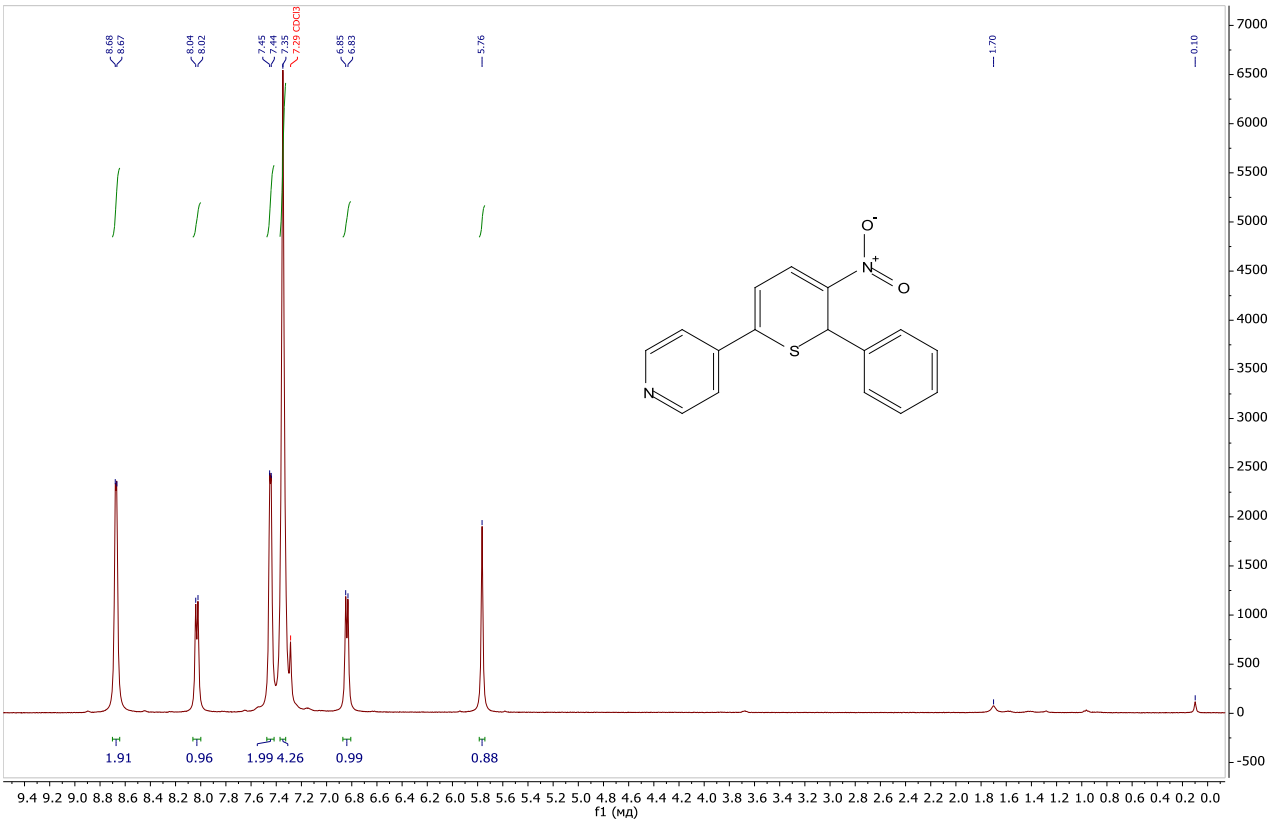
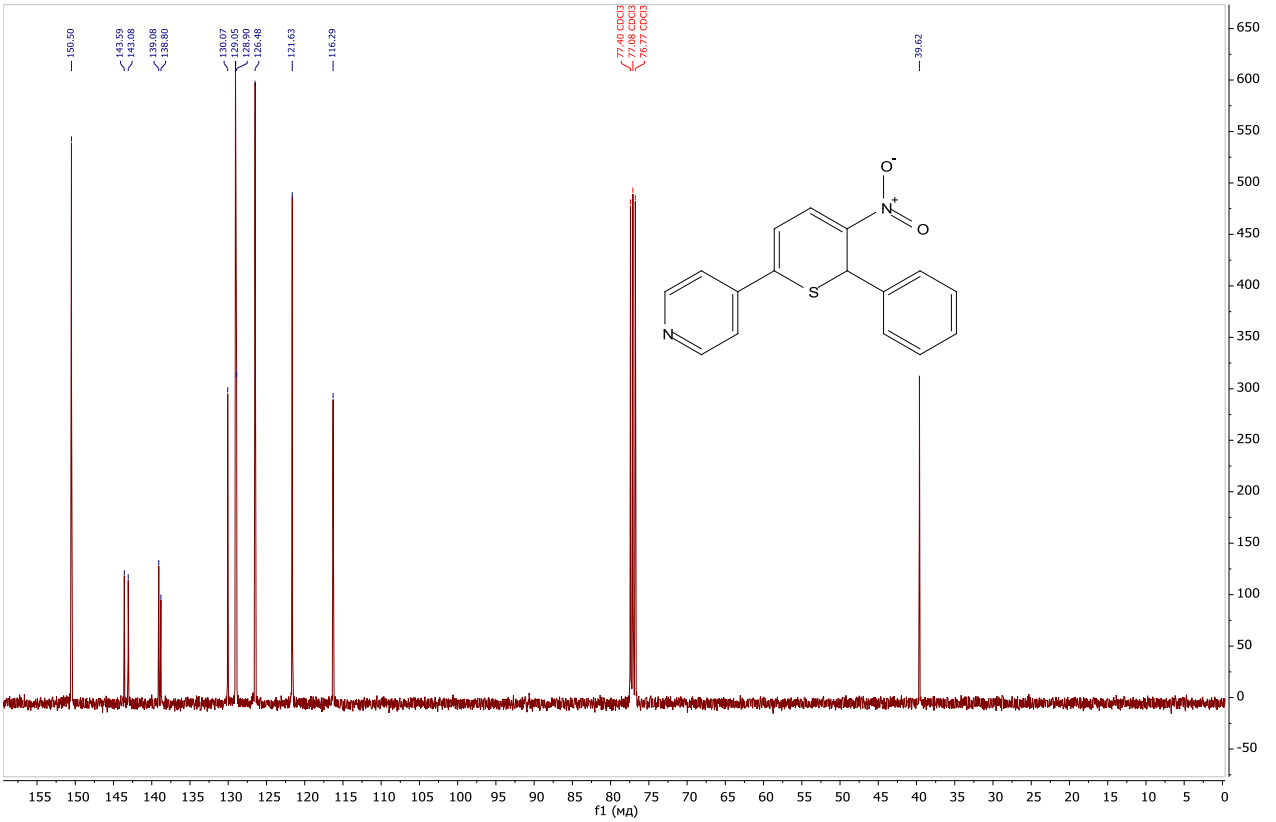
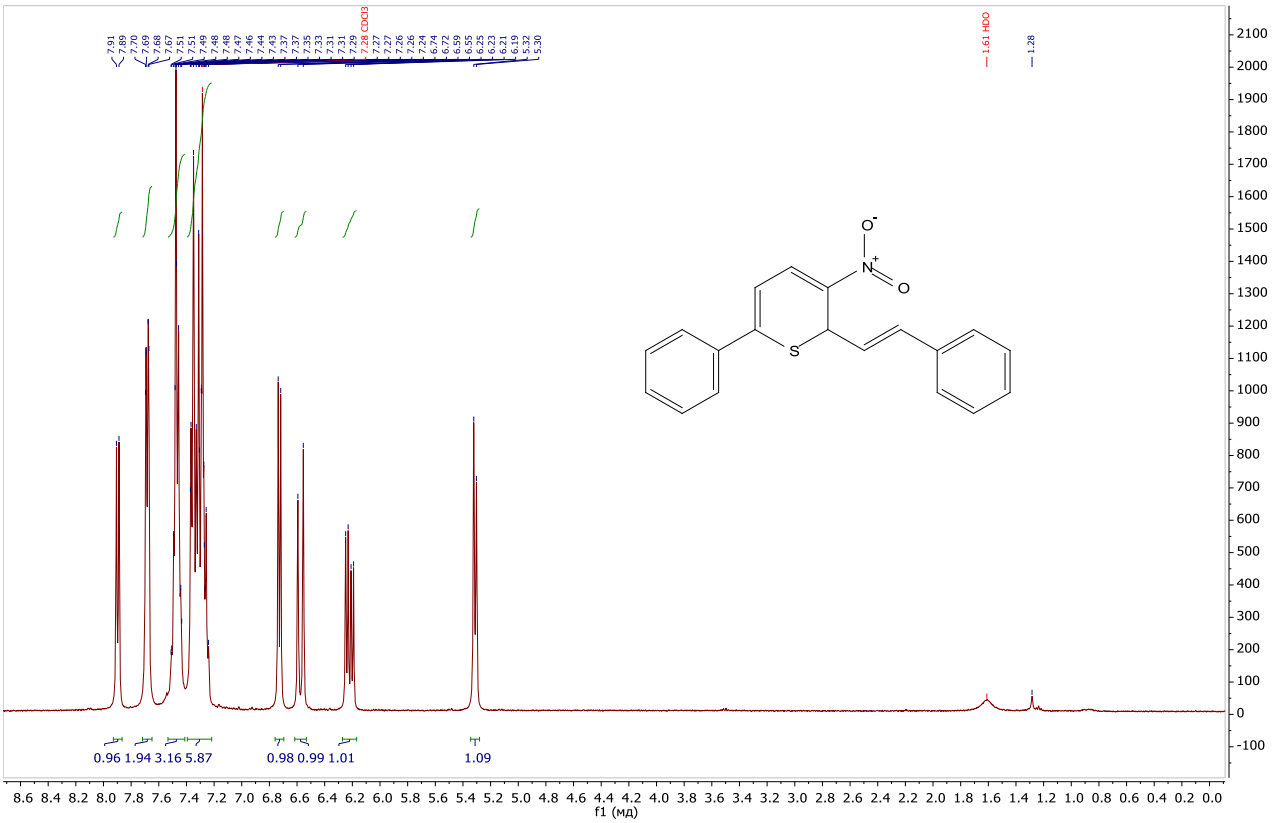
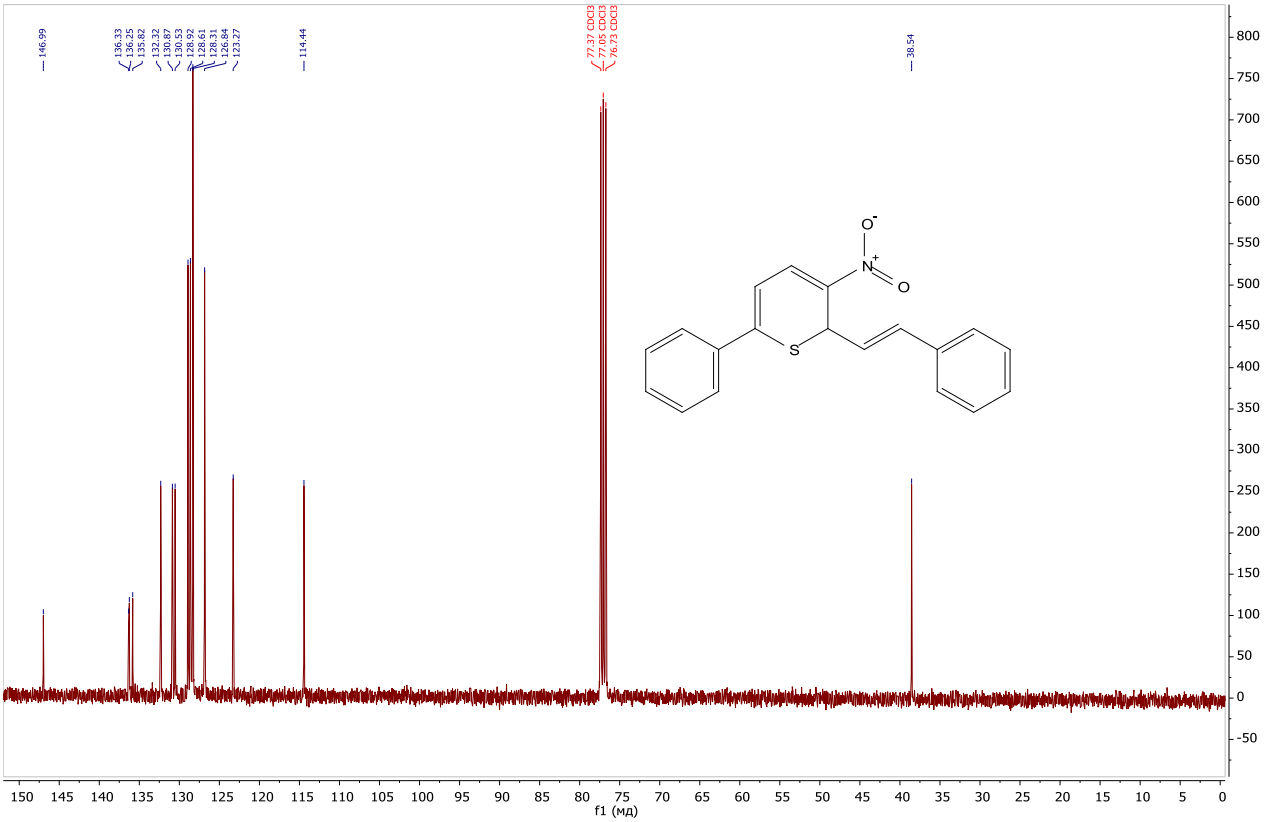
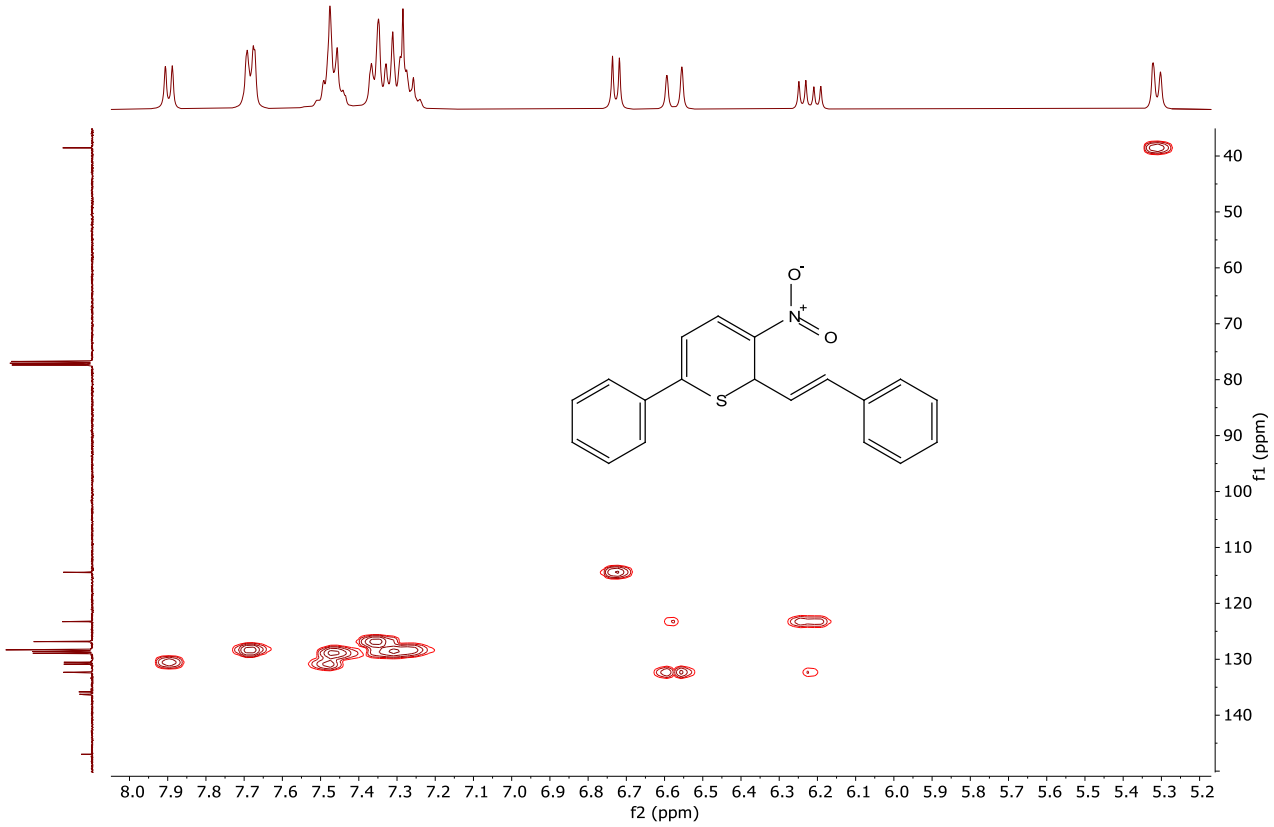




^{1}H,^{13}C, ^{1}H-^{13}C-HSQC and ^{1}H-^{13}C-HMBC NMR spectra of compound **7** ($CDCl_3$)



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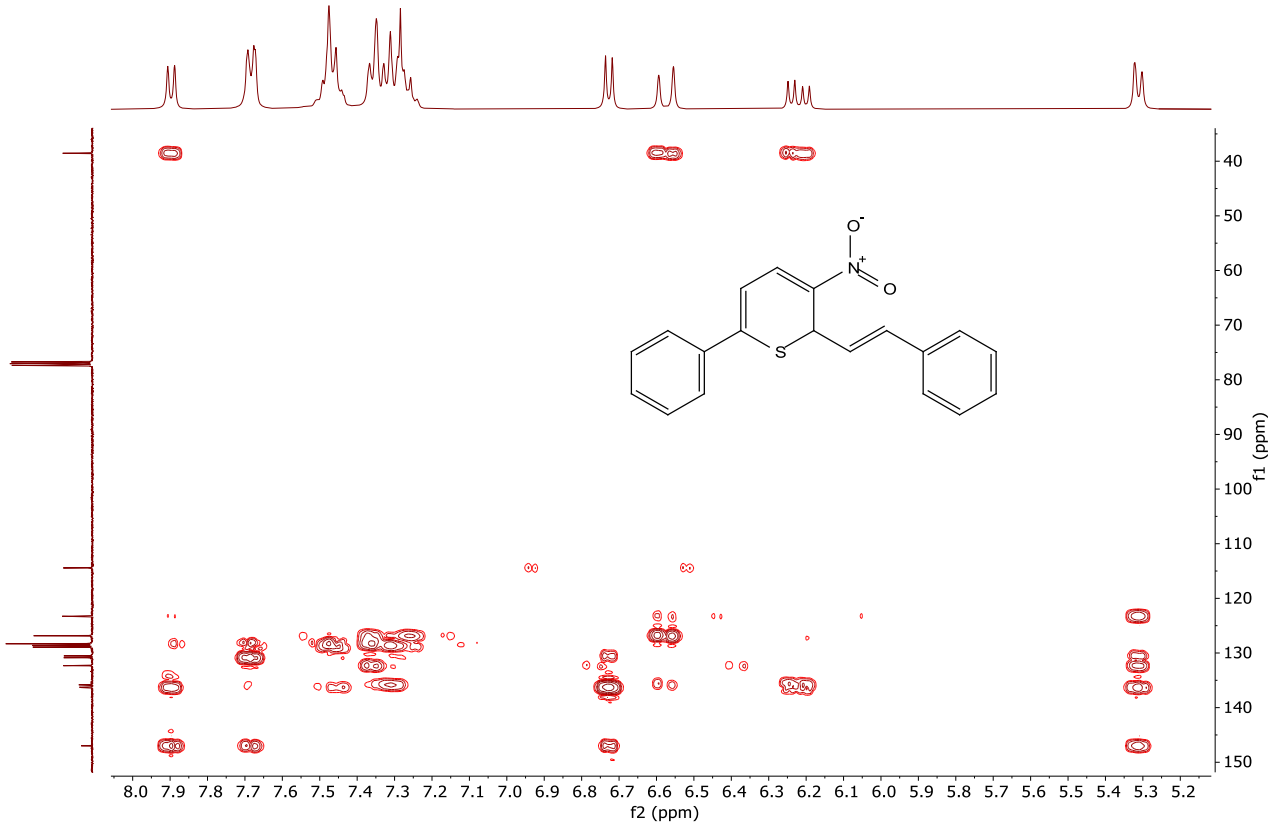
f2 (ppm)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f1 (ppm)
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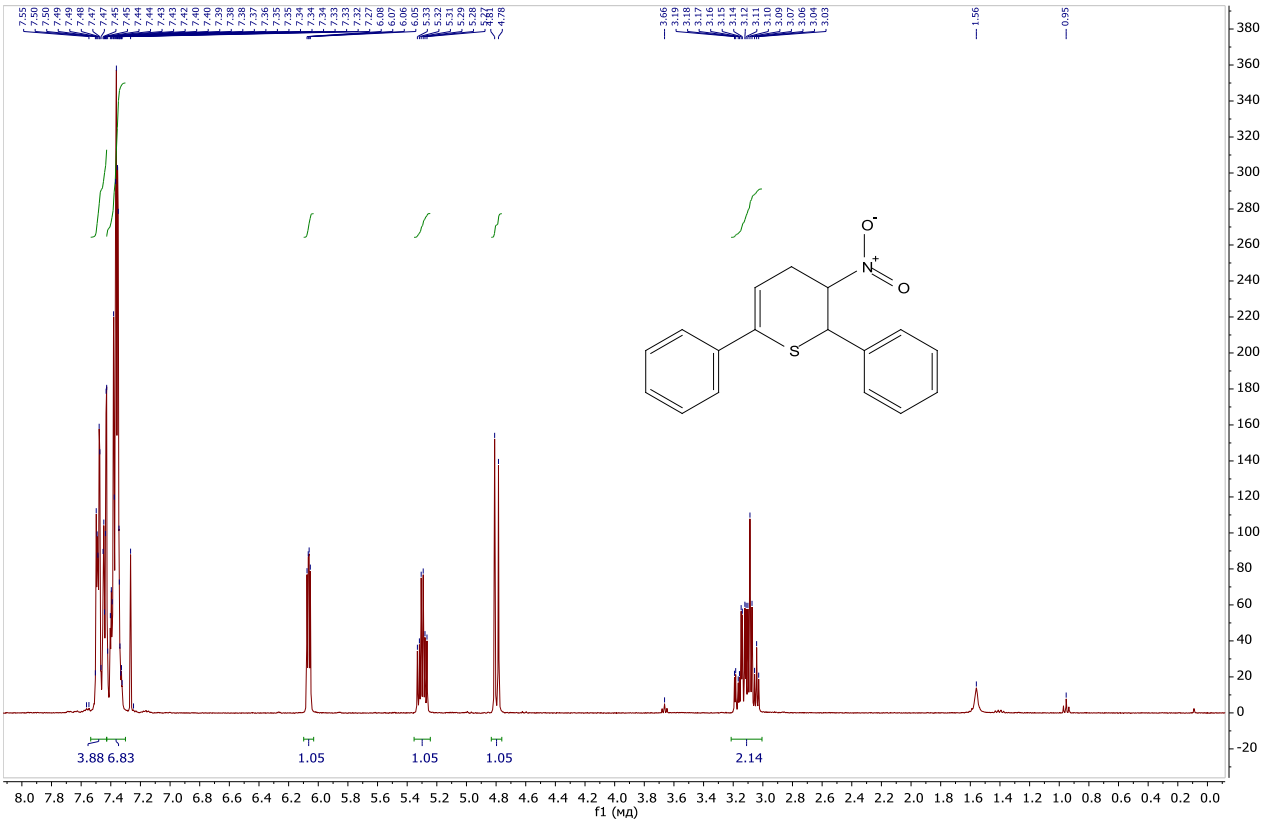
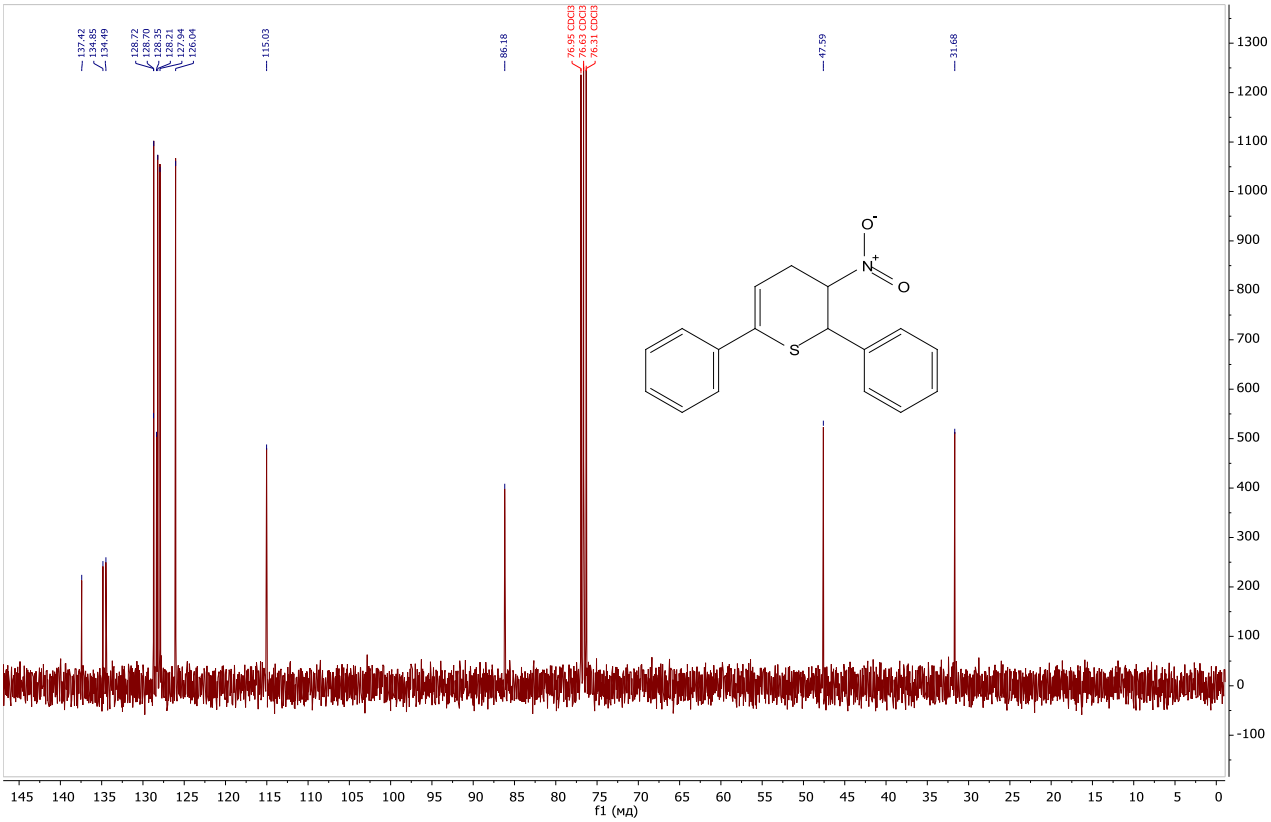
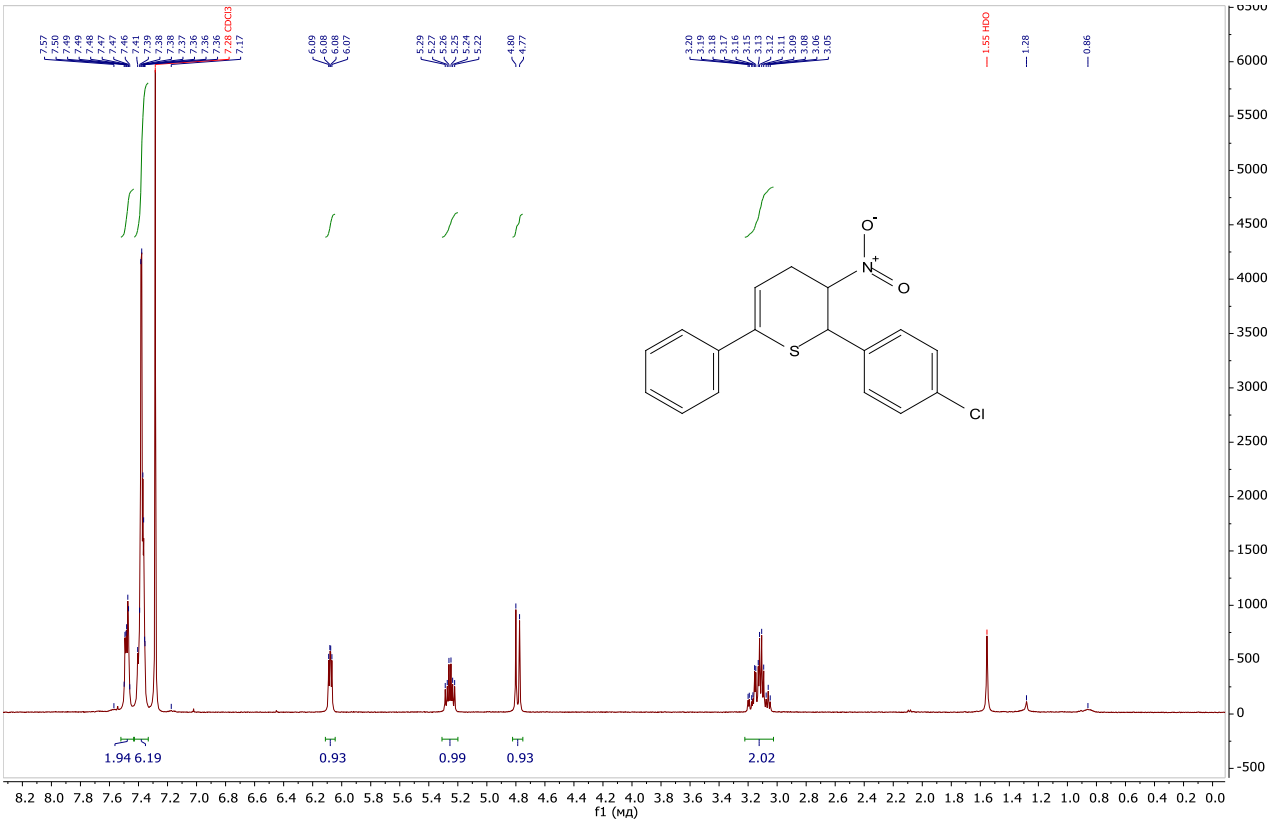
f2 (ppm)

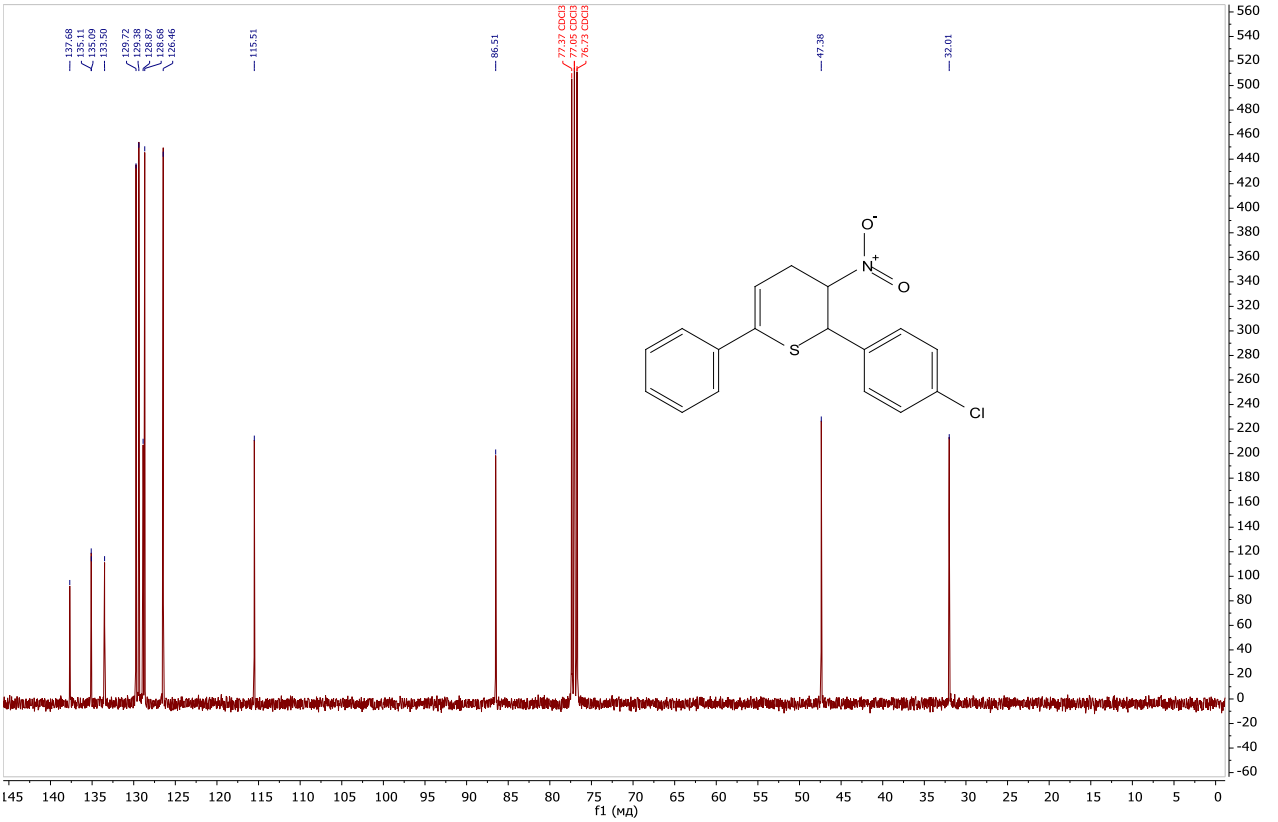
^{1}H and ^{13}C NMR spectra of compound **9a** ($CDCl_3$)





^{1}H and ^{13}C NMR spectra of compound **9b** ($CDCl_3$)





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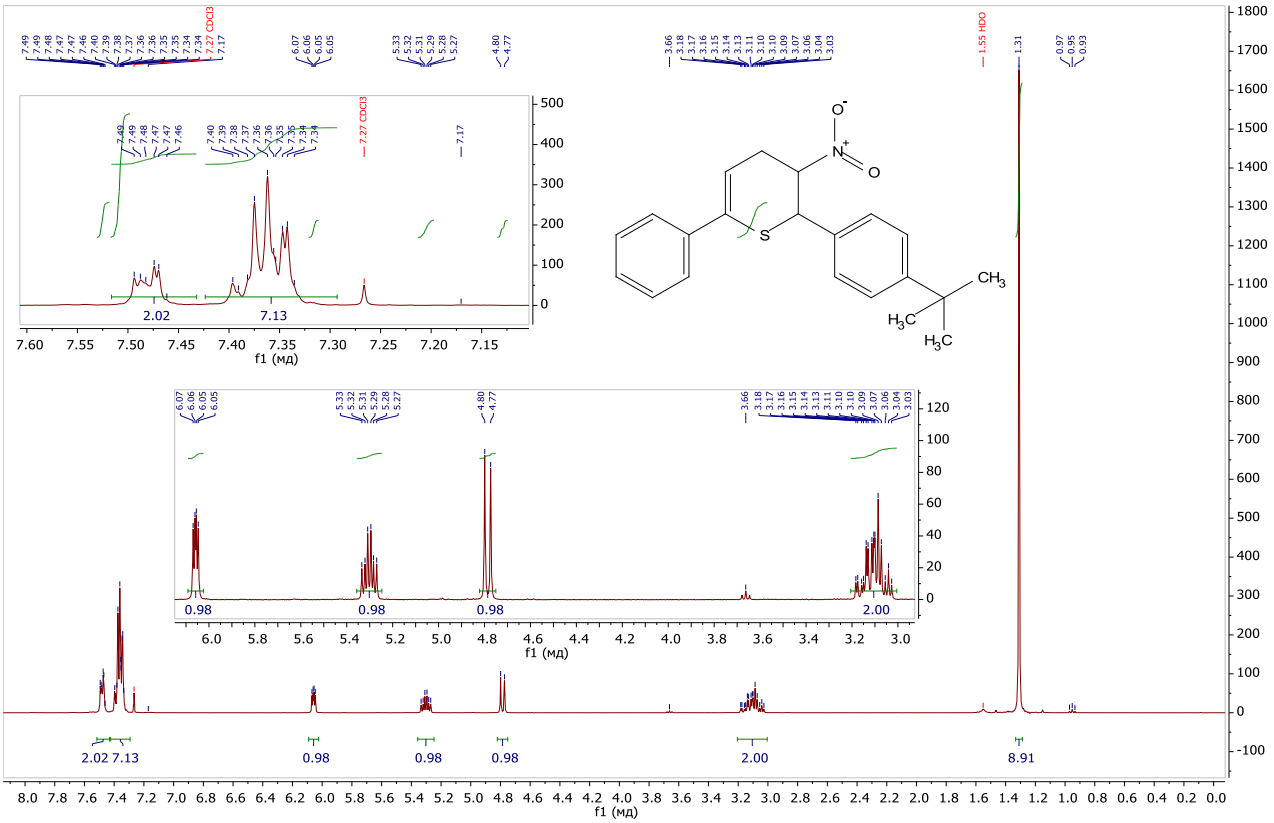

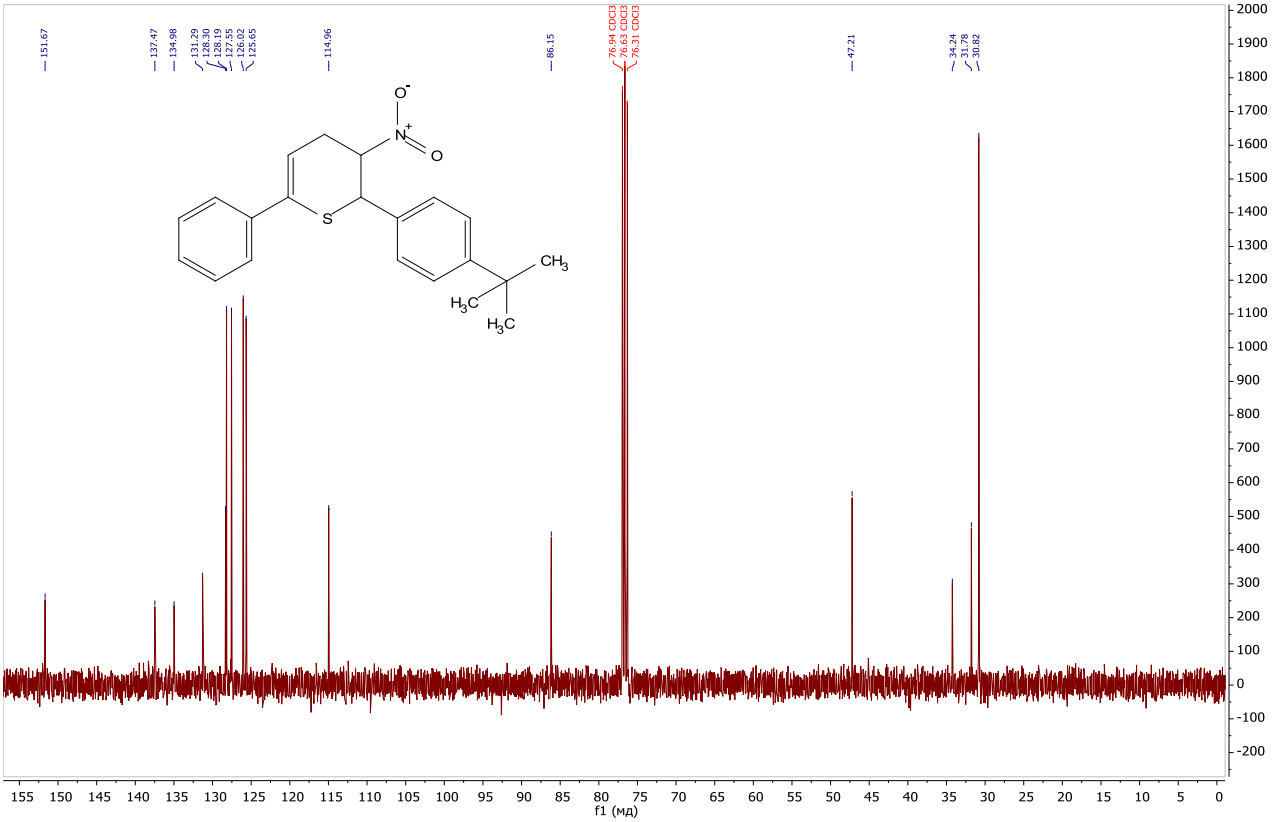

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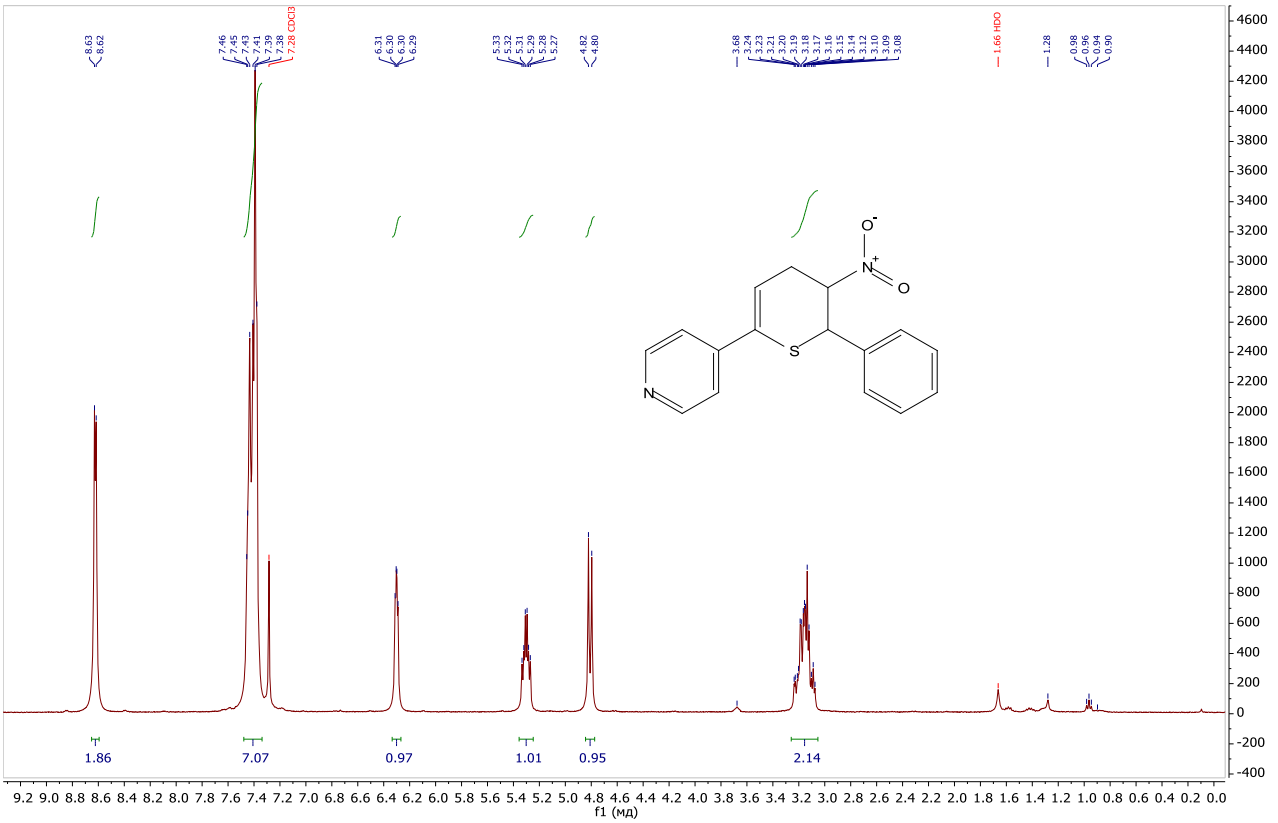

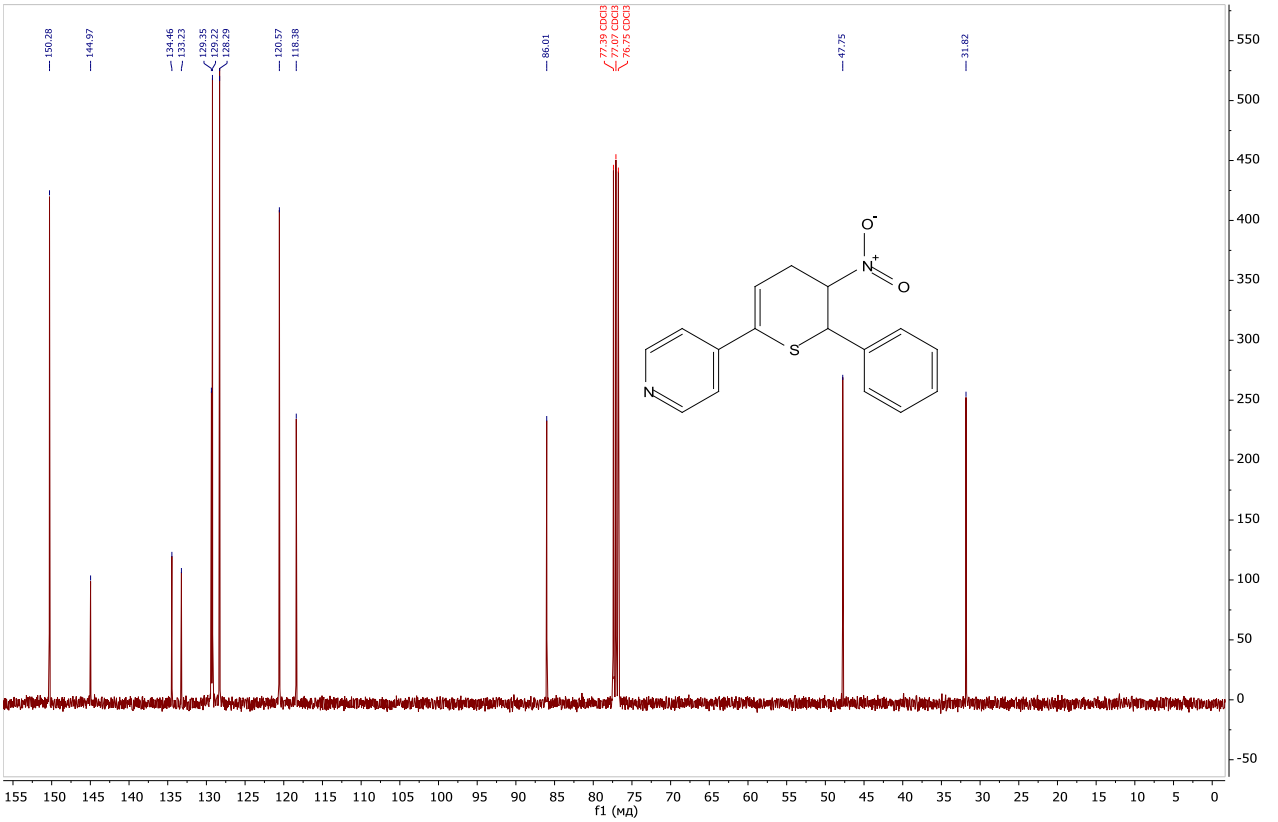

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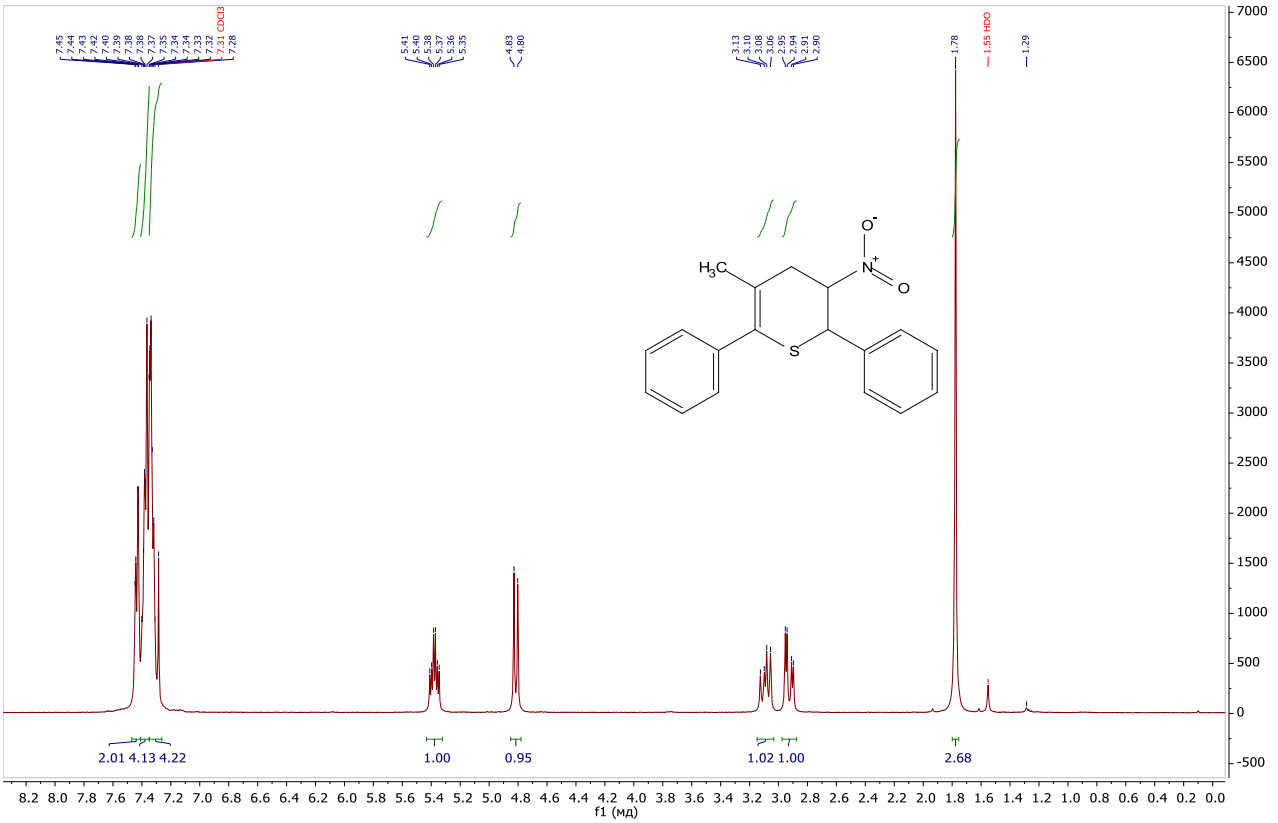
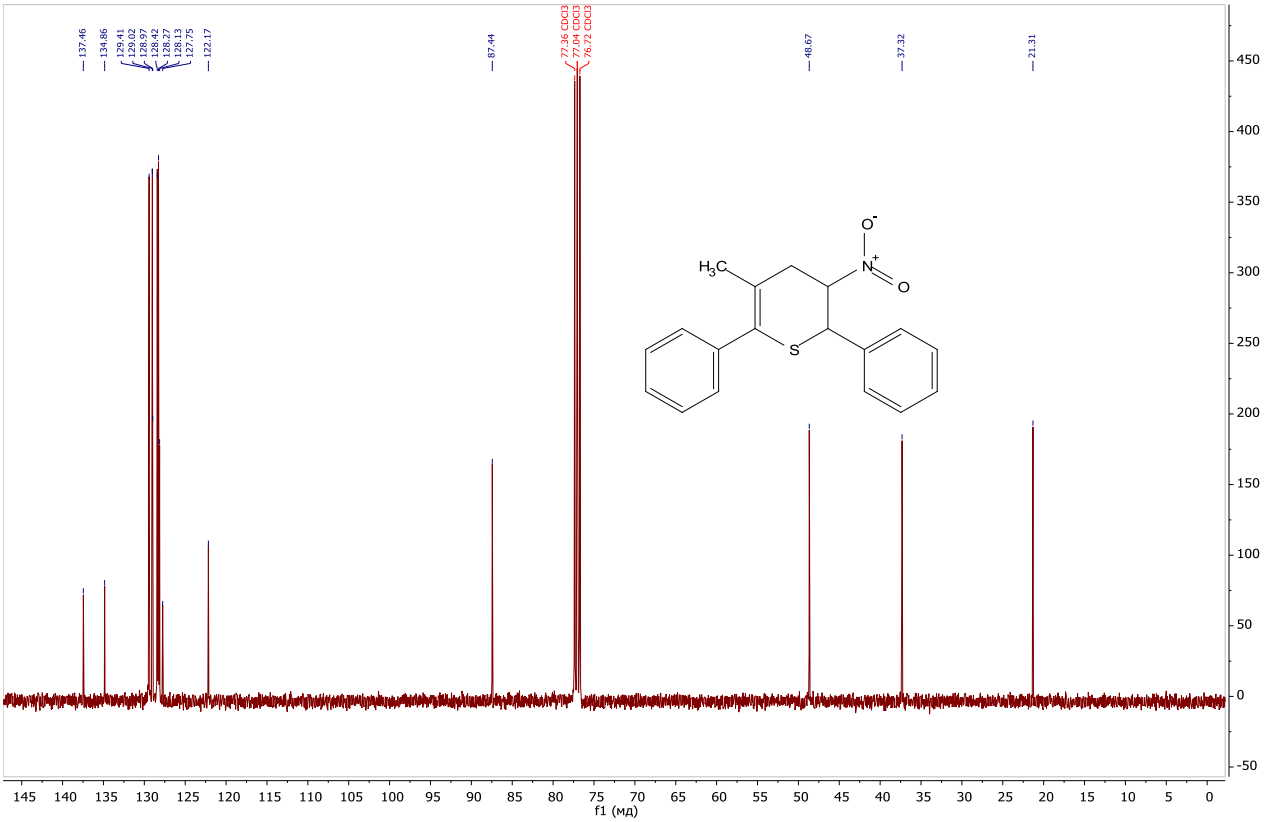
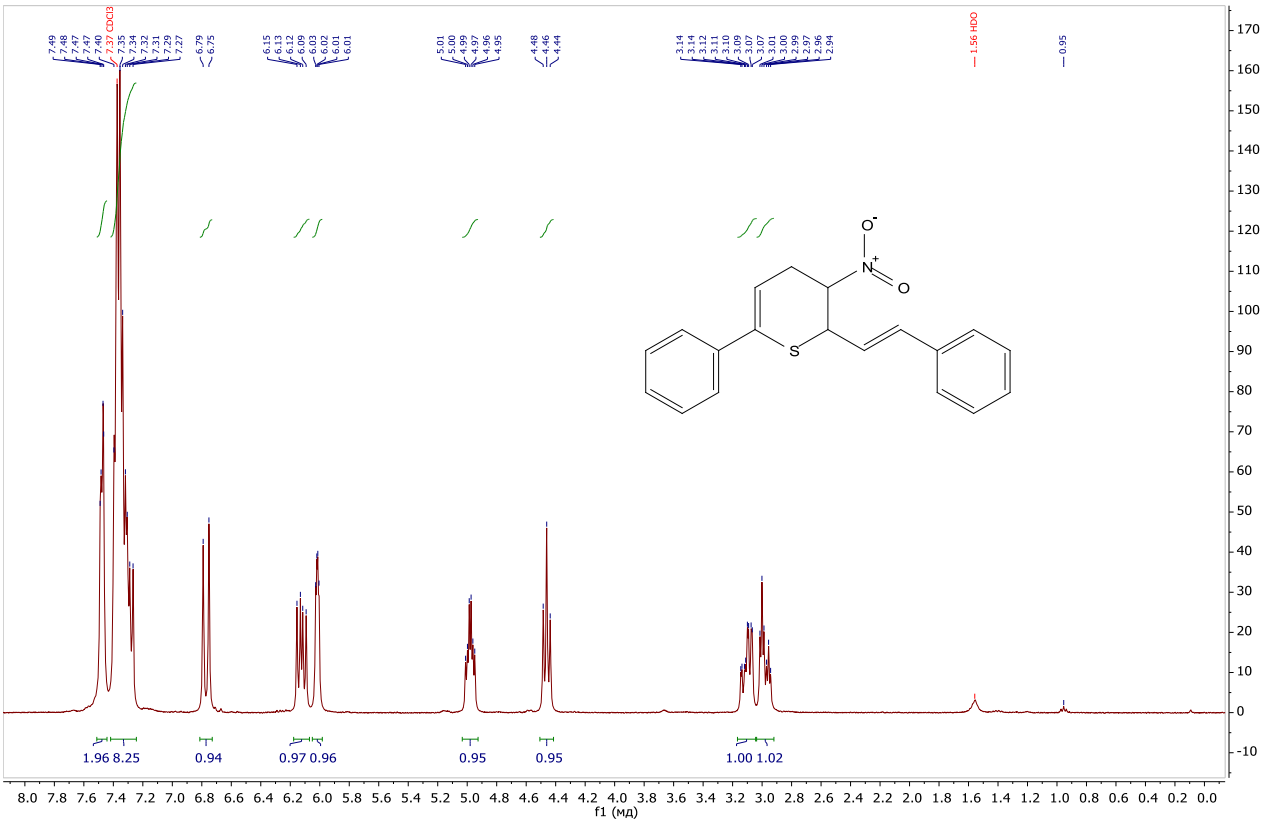
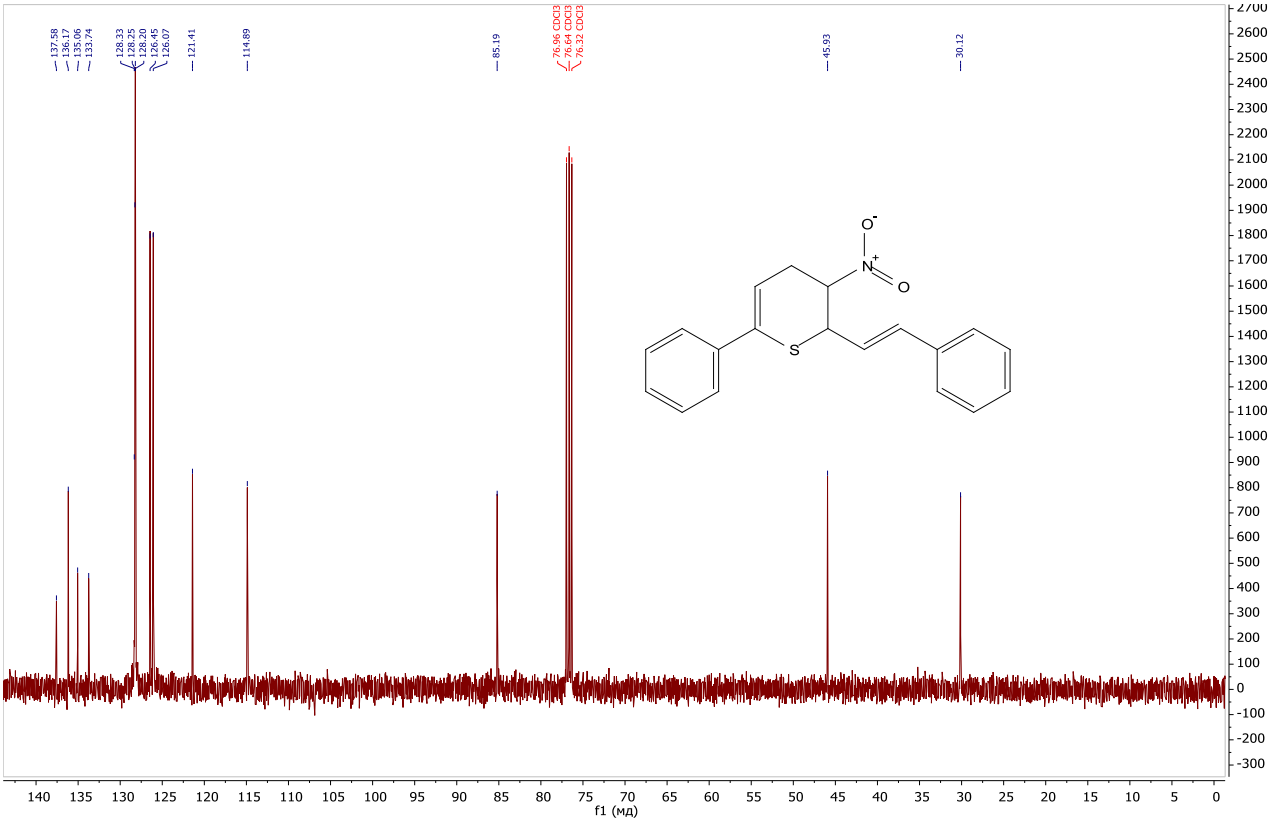




^{1}H,^{13}C, ^{1}H-^{13}C-HSQC and ^{1}H-^{13}C-HMBC NMR spectra of compound **10** ($CDCl_3$)



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f1 (ppm)
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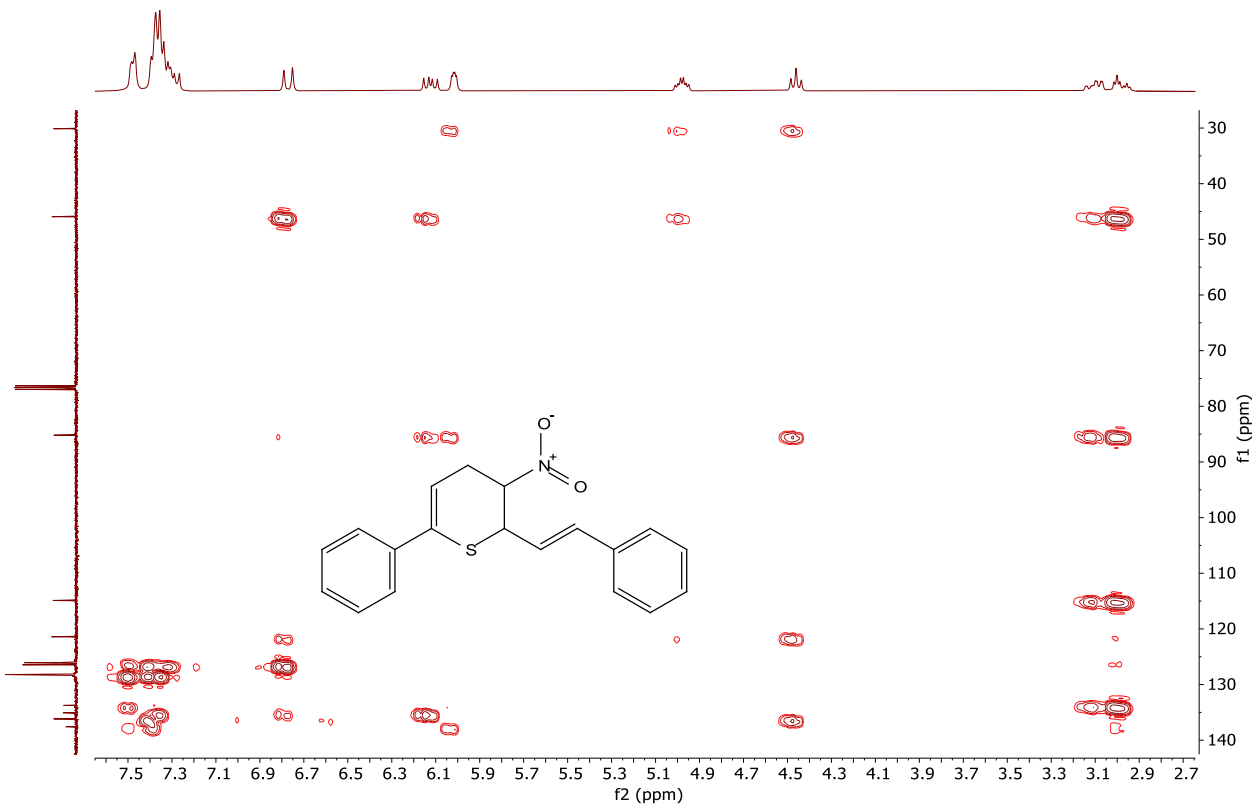
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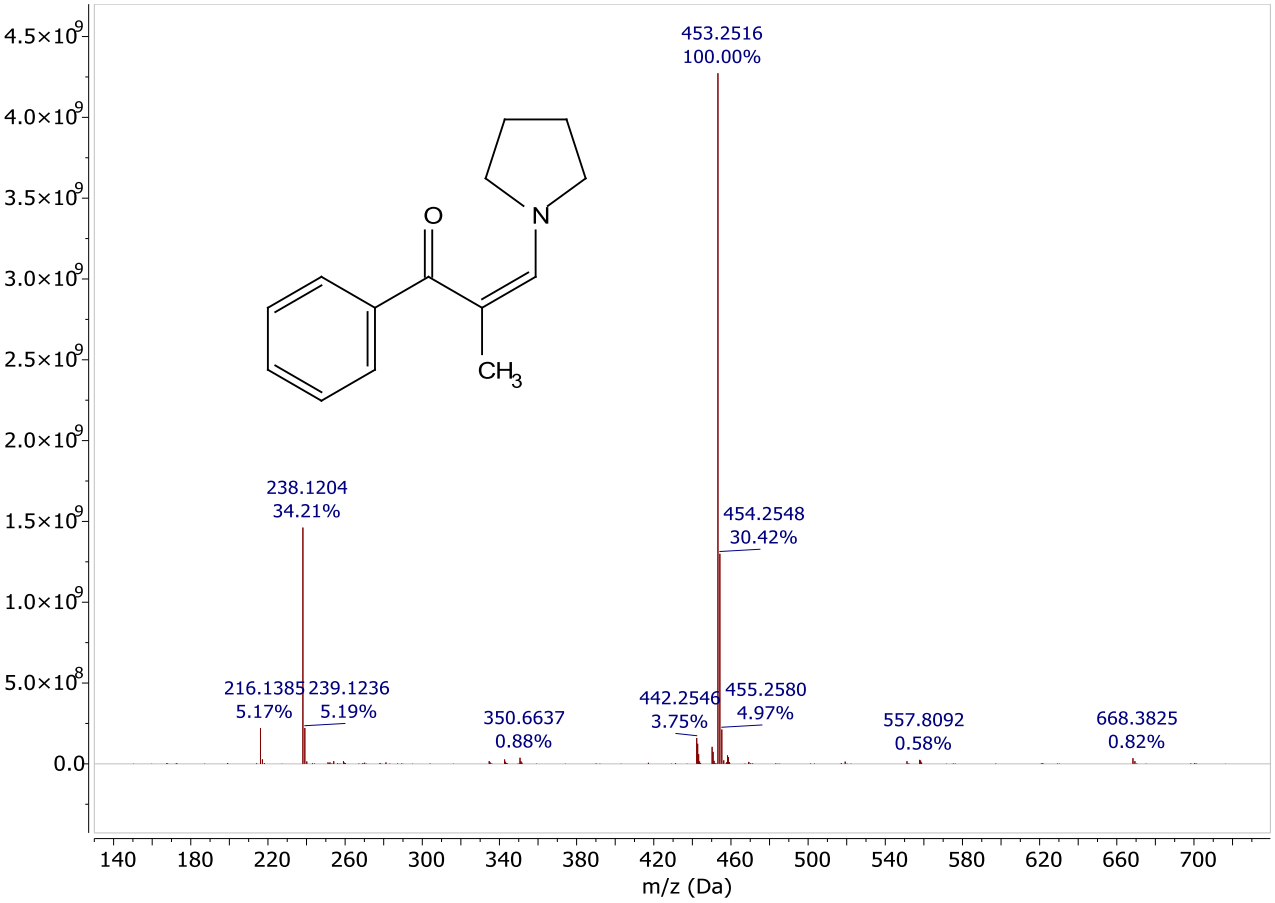
f2 (ppm)

3. Copies of HRMS spectra

HRMS spectra of compound **1c**



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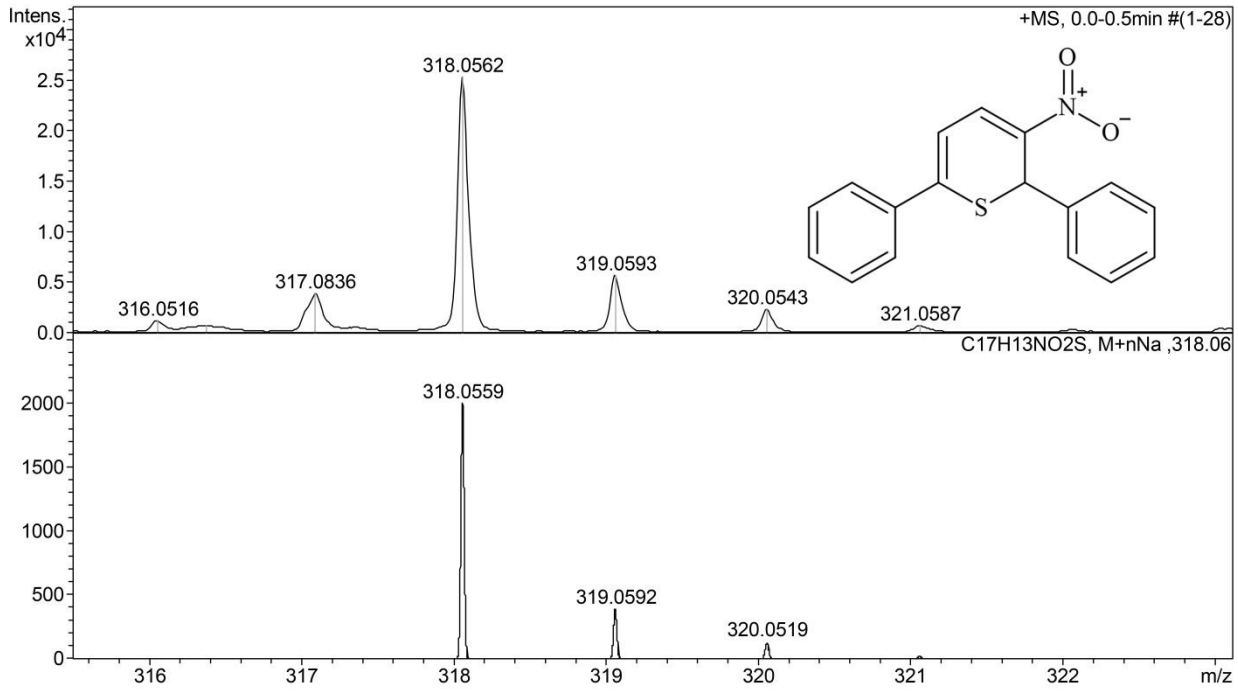

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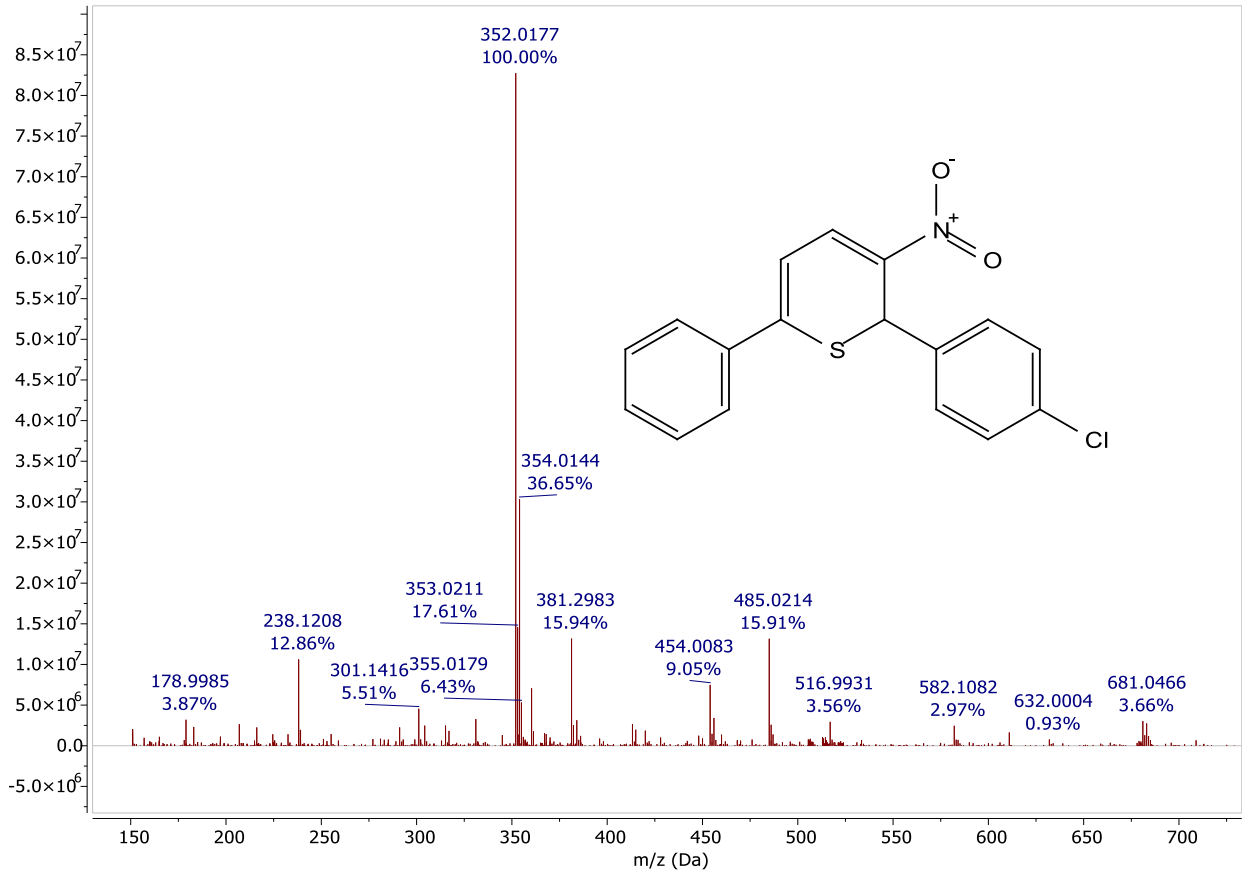

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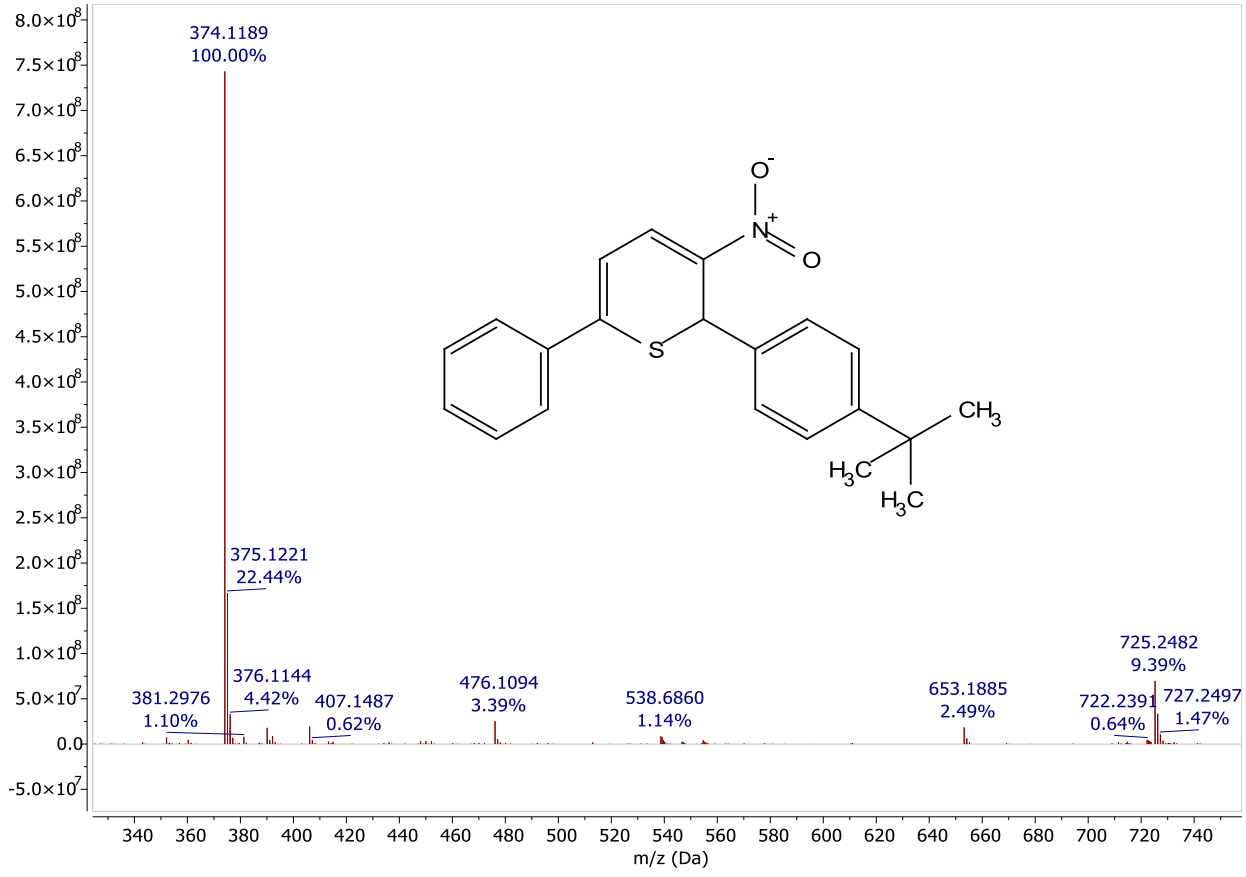

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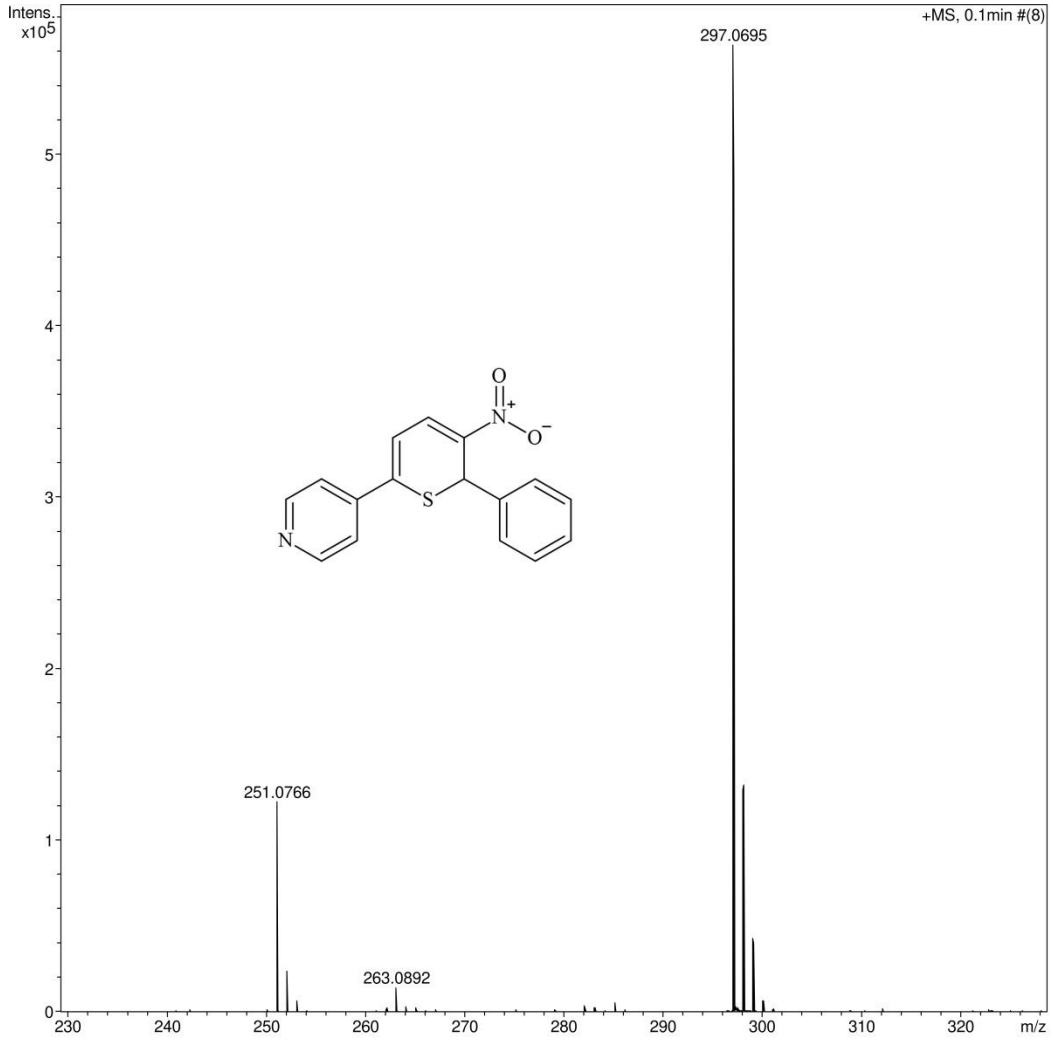

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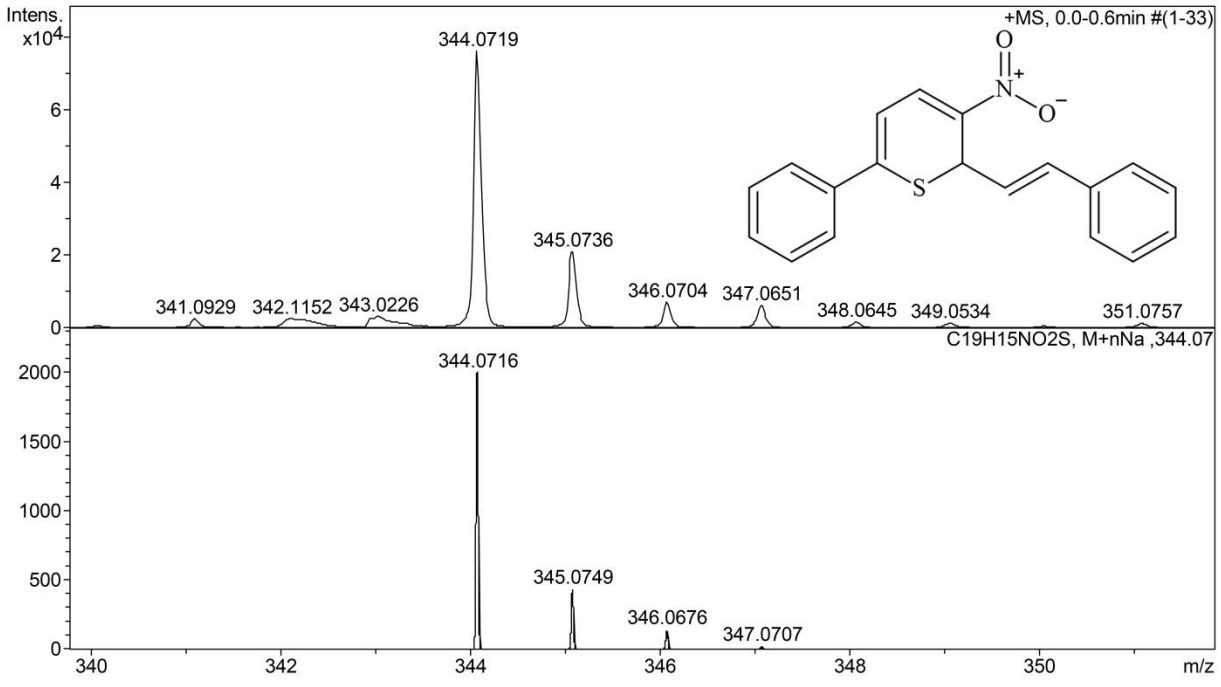

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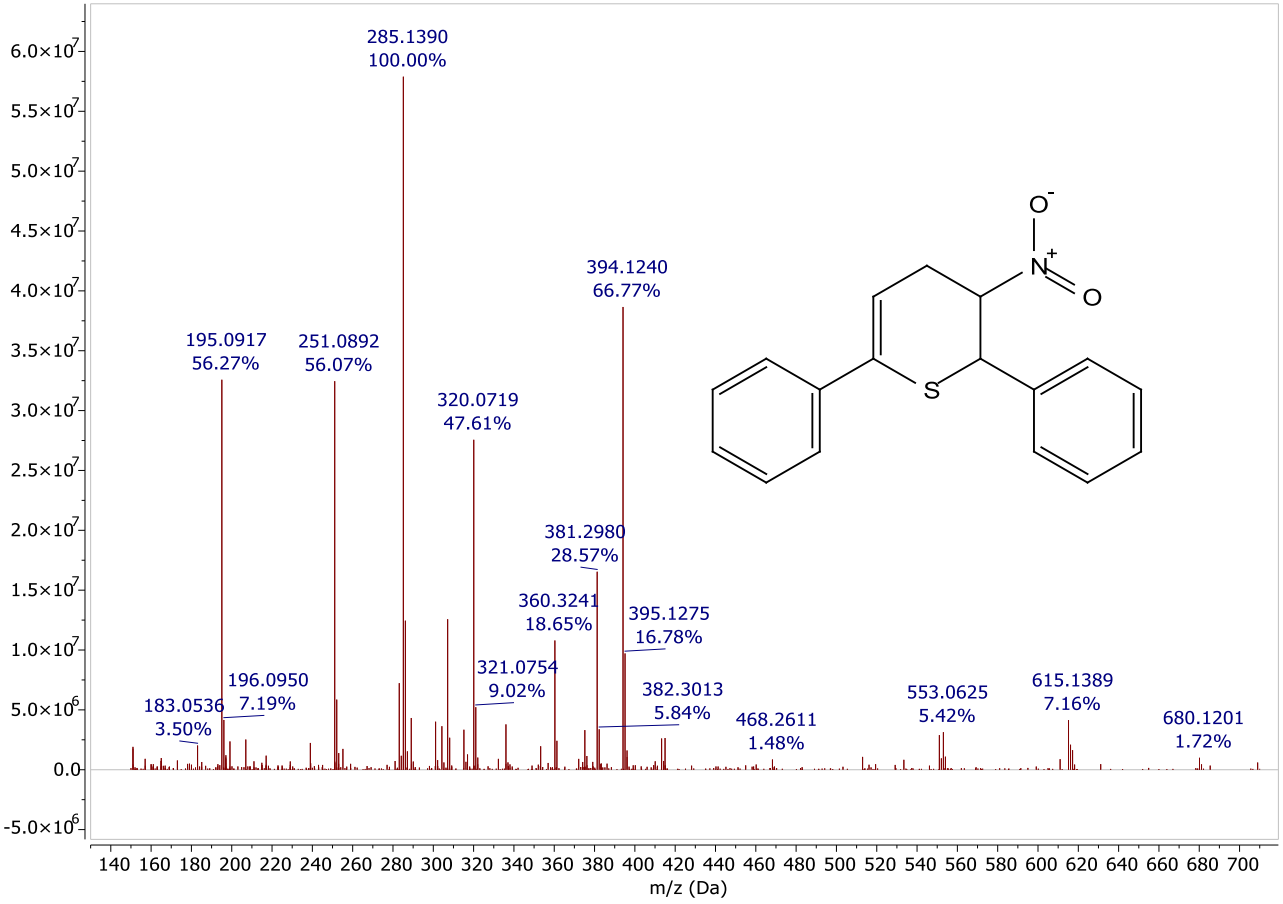

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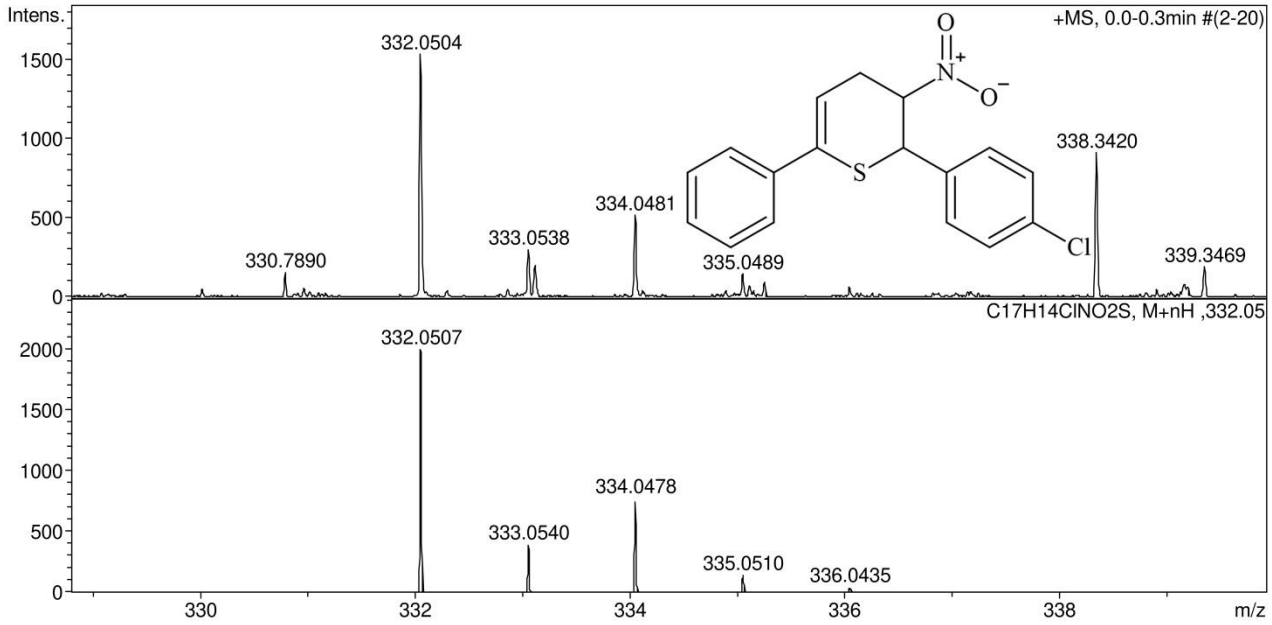

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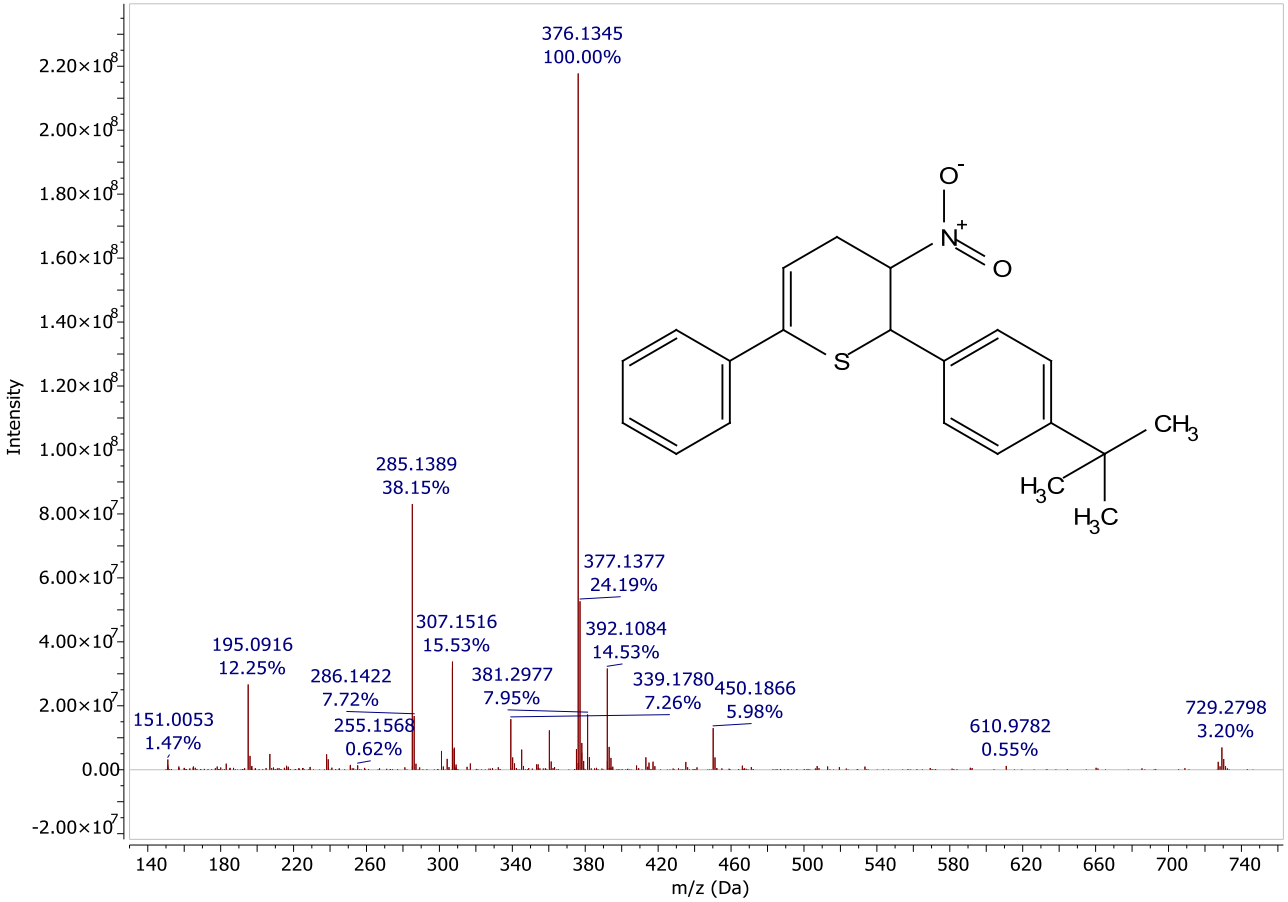

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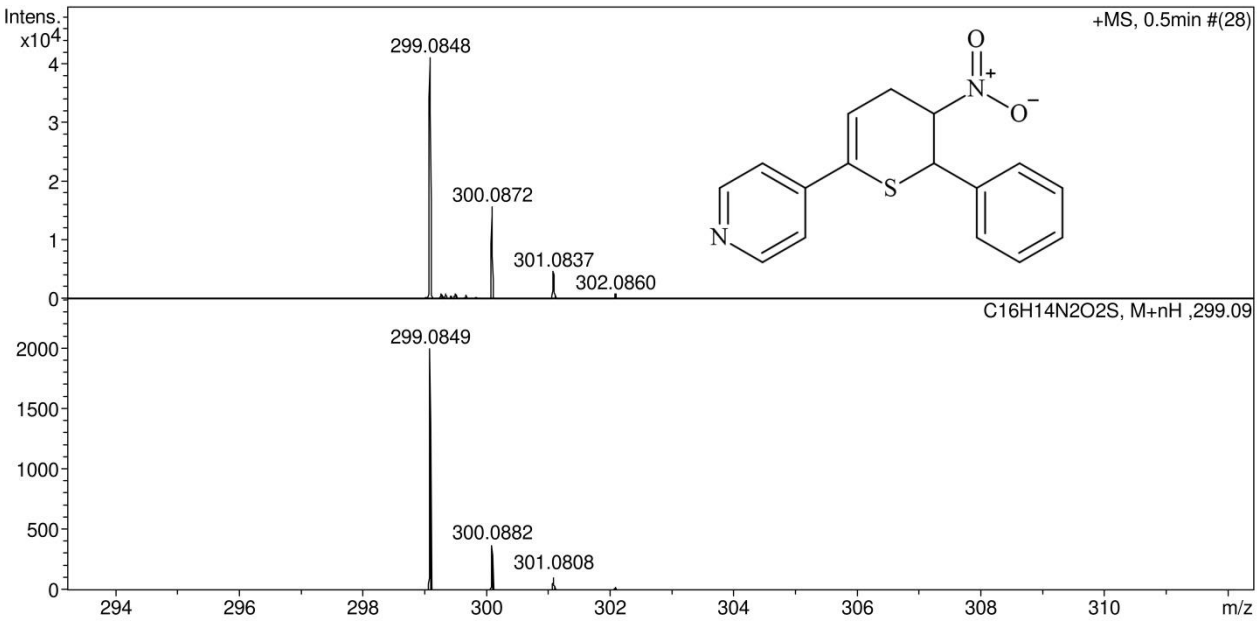

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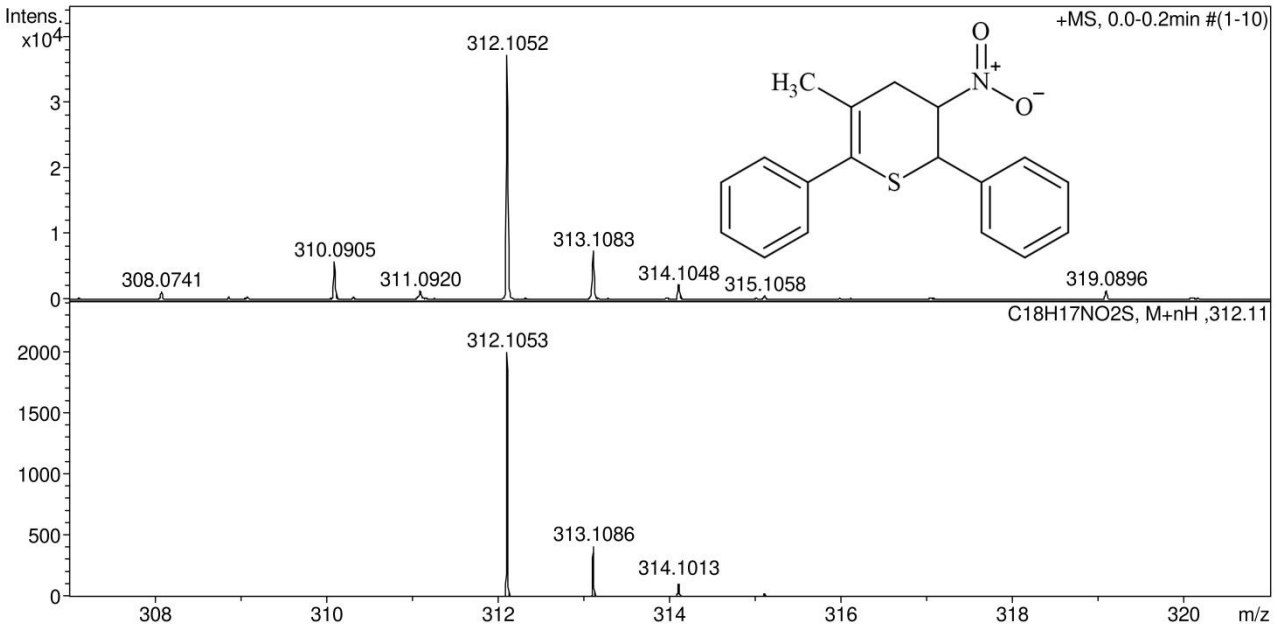

HRMS spectra of compound **10**