

Supporting information for

Different behavior of 2-substituted 3-nitro-2*H*-chromenes in the reaction with stabilized azomethine ylides generated from α -iminoesters

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Vladislav Y. Korotaev* and Vyacheslav Y. Sosnovskikh*

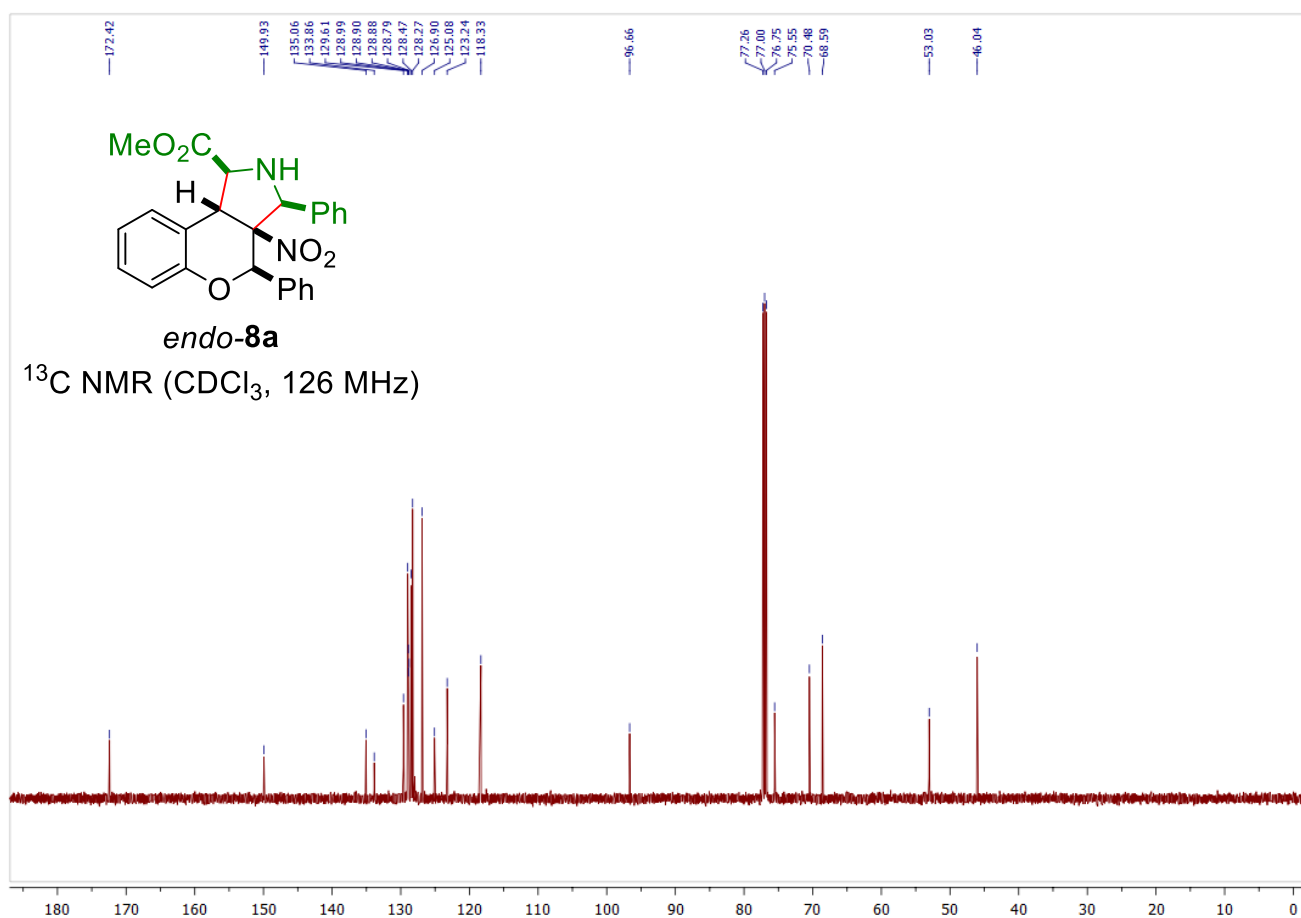
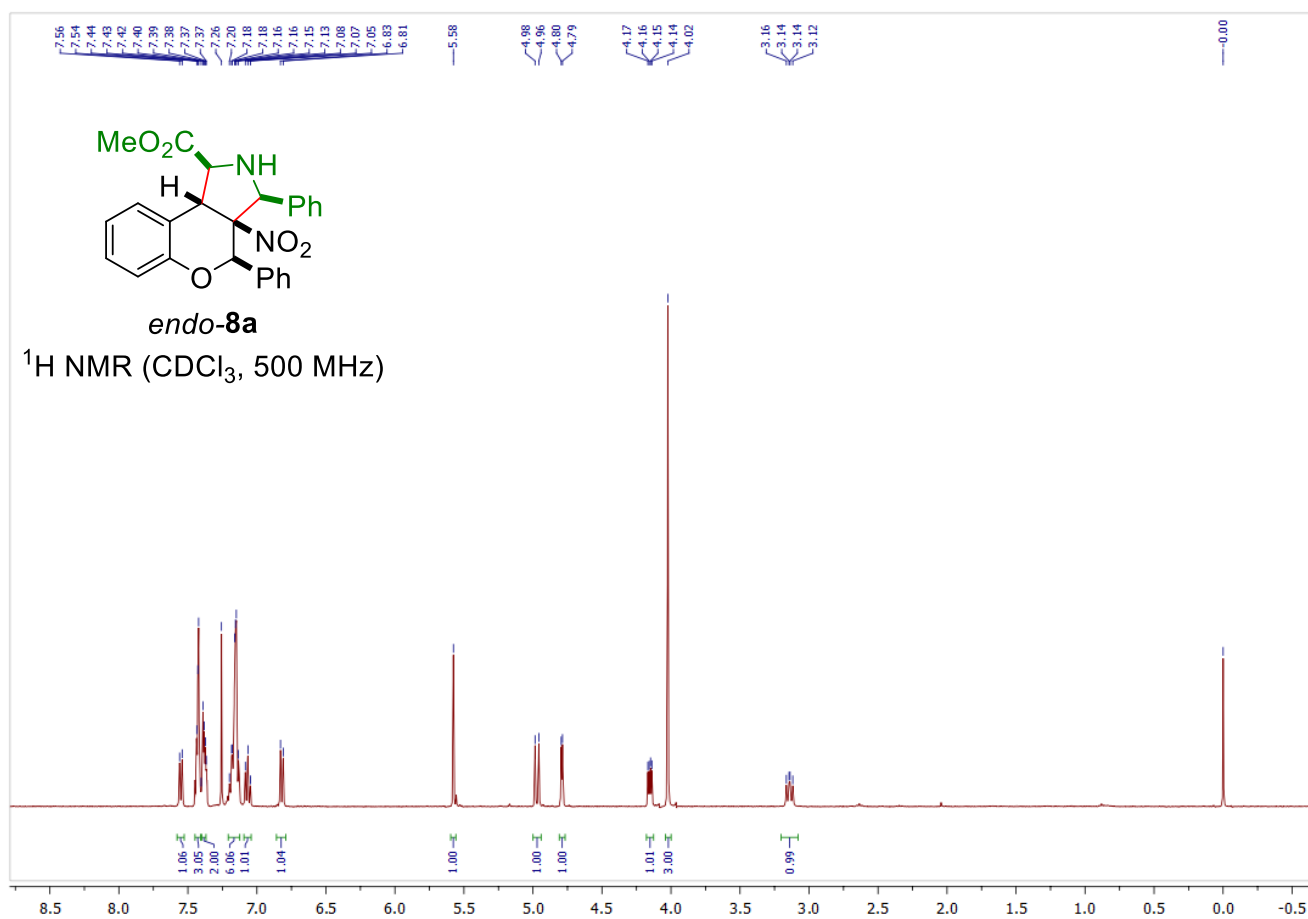
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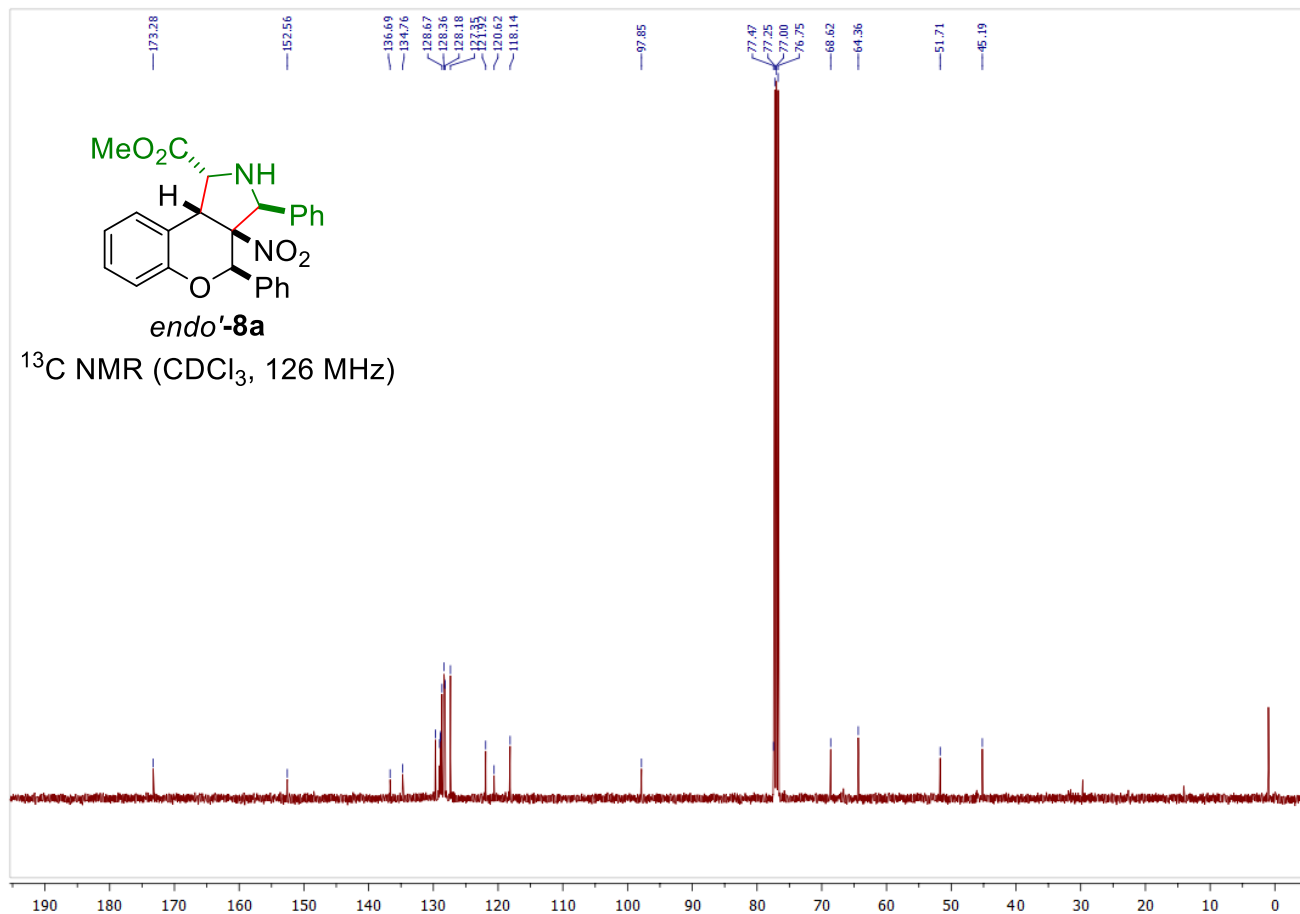
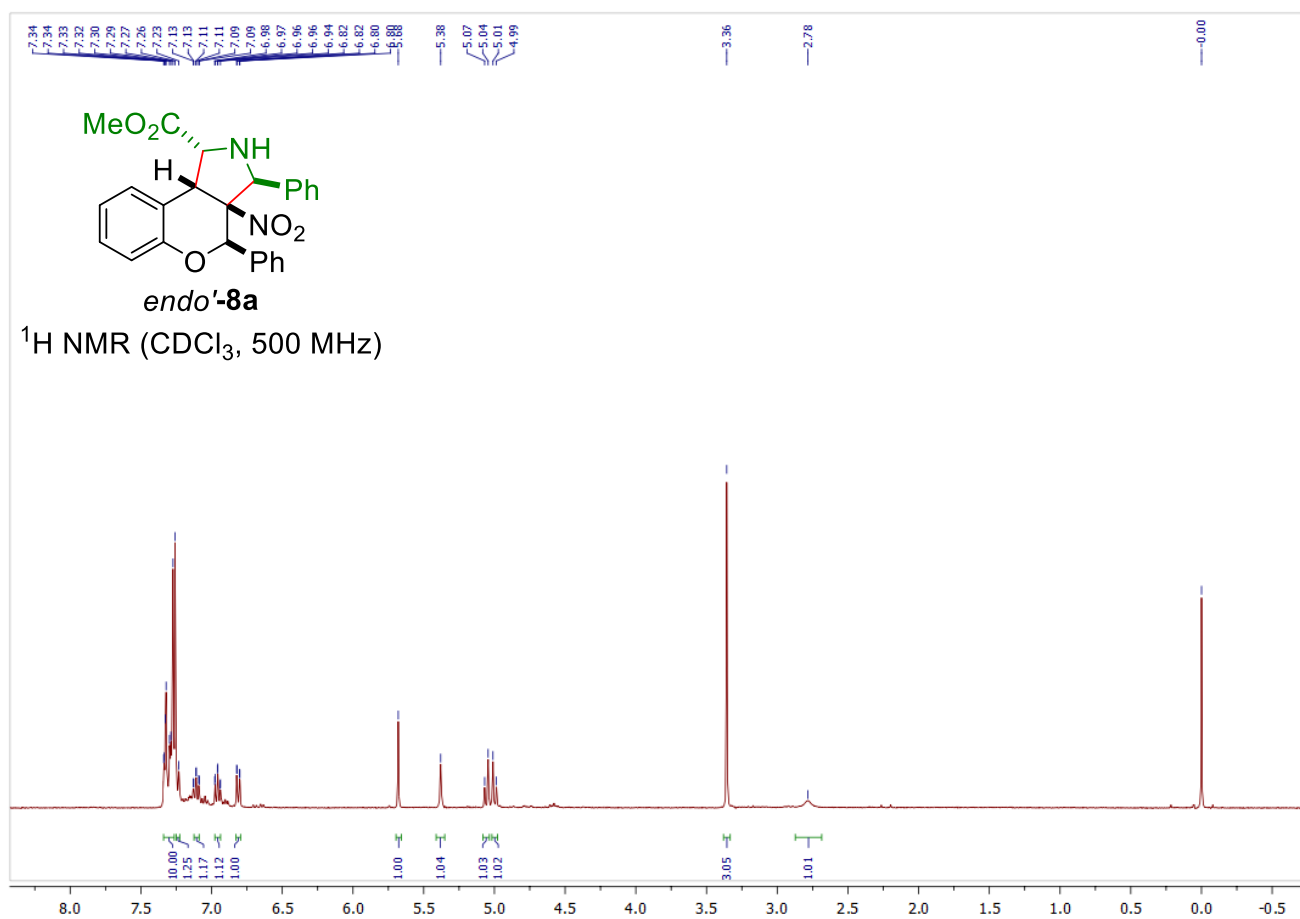
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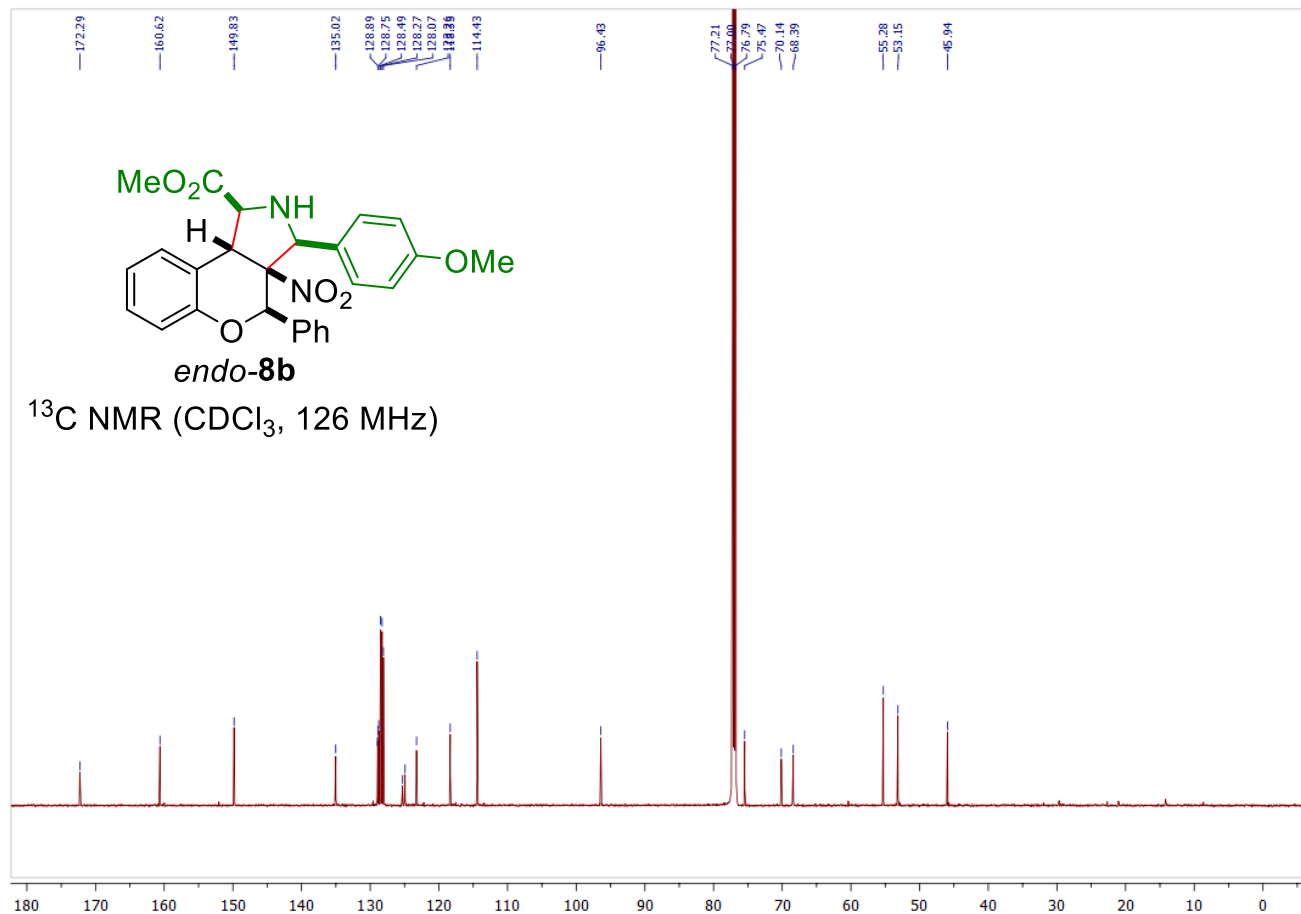
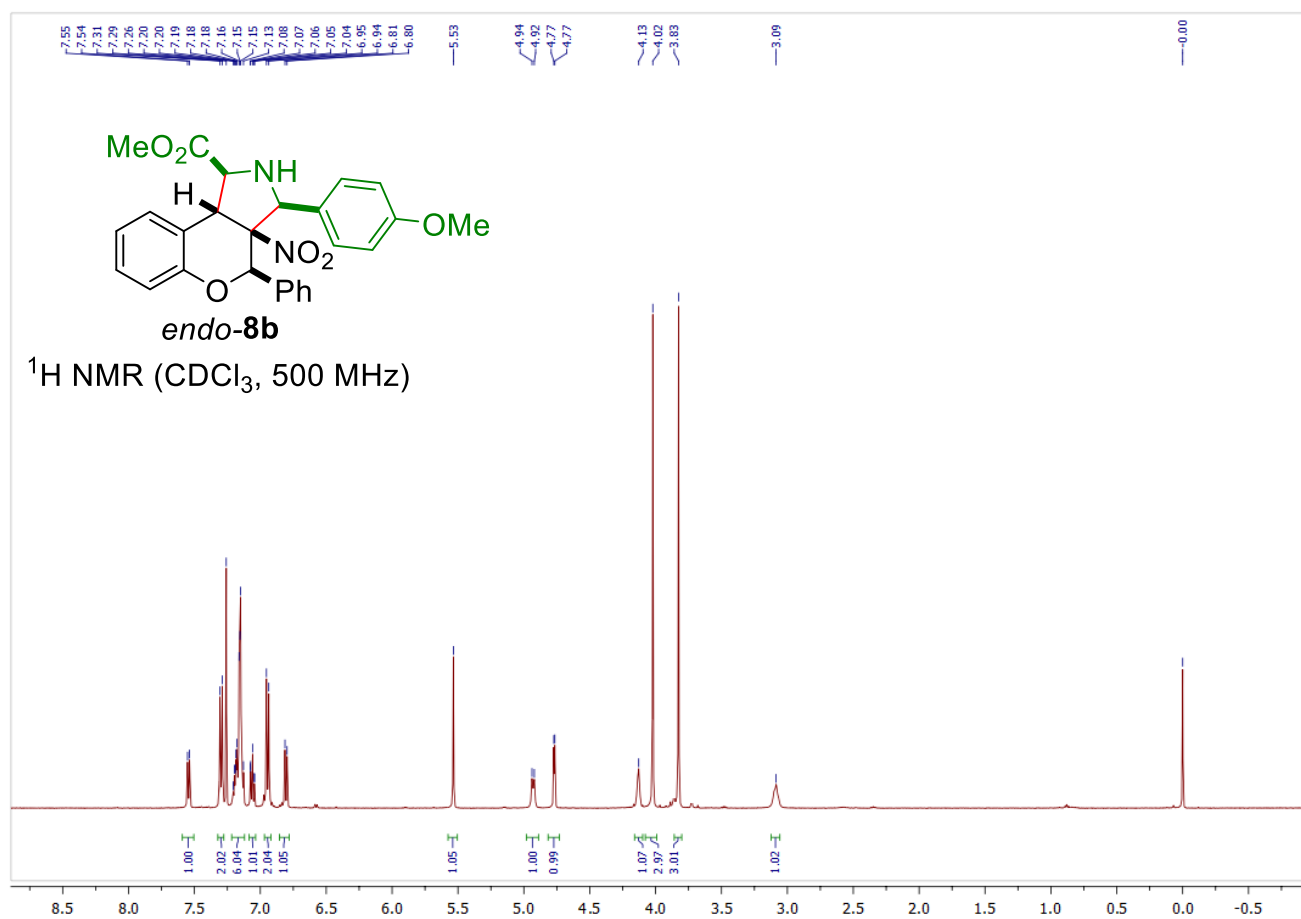
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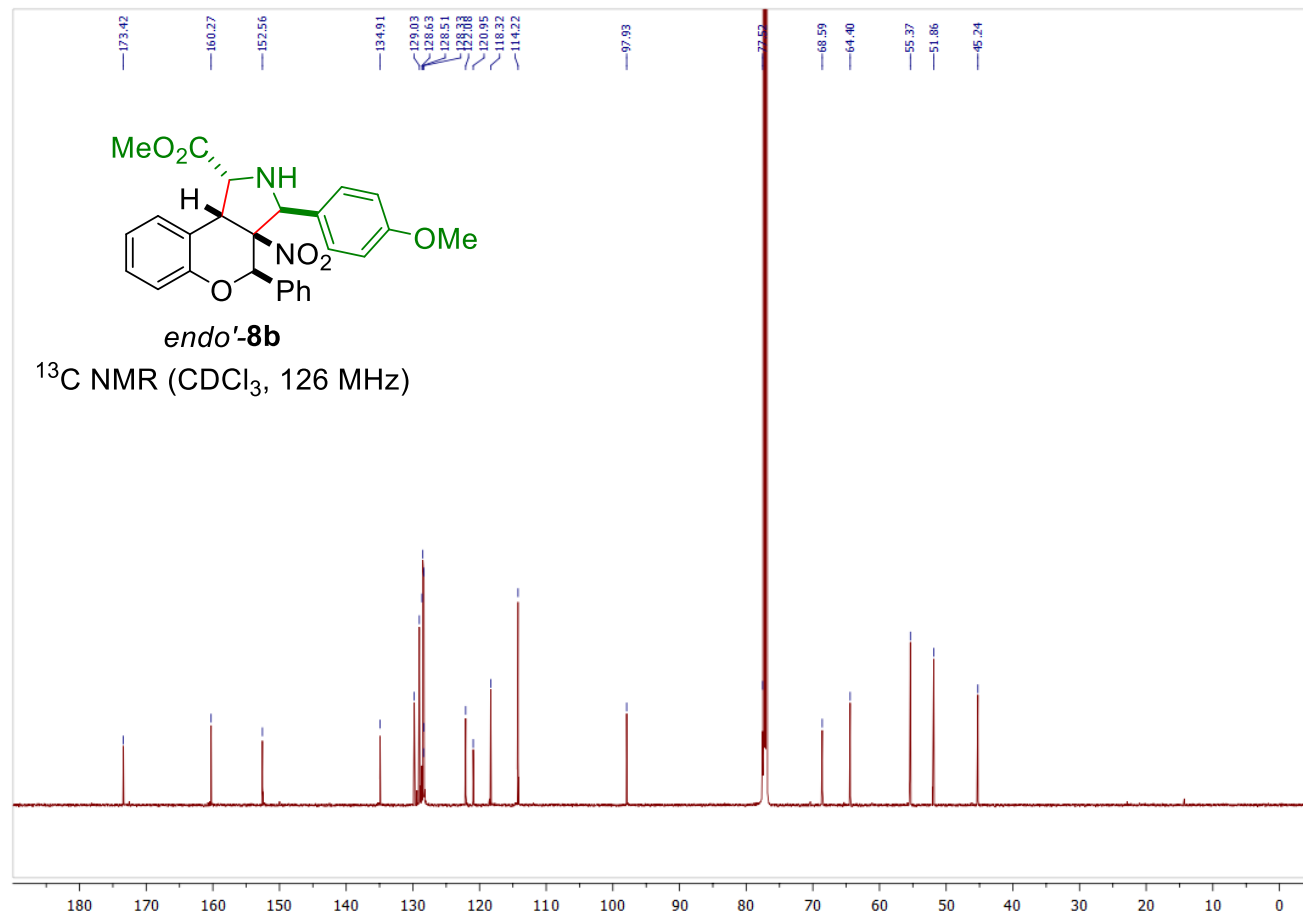
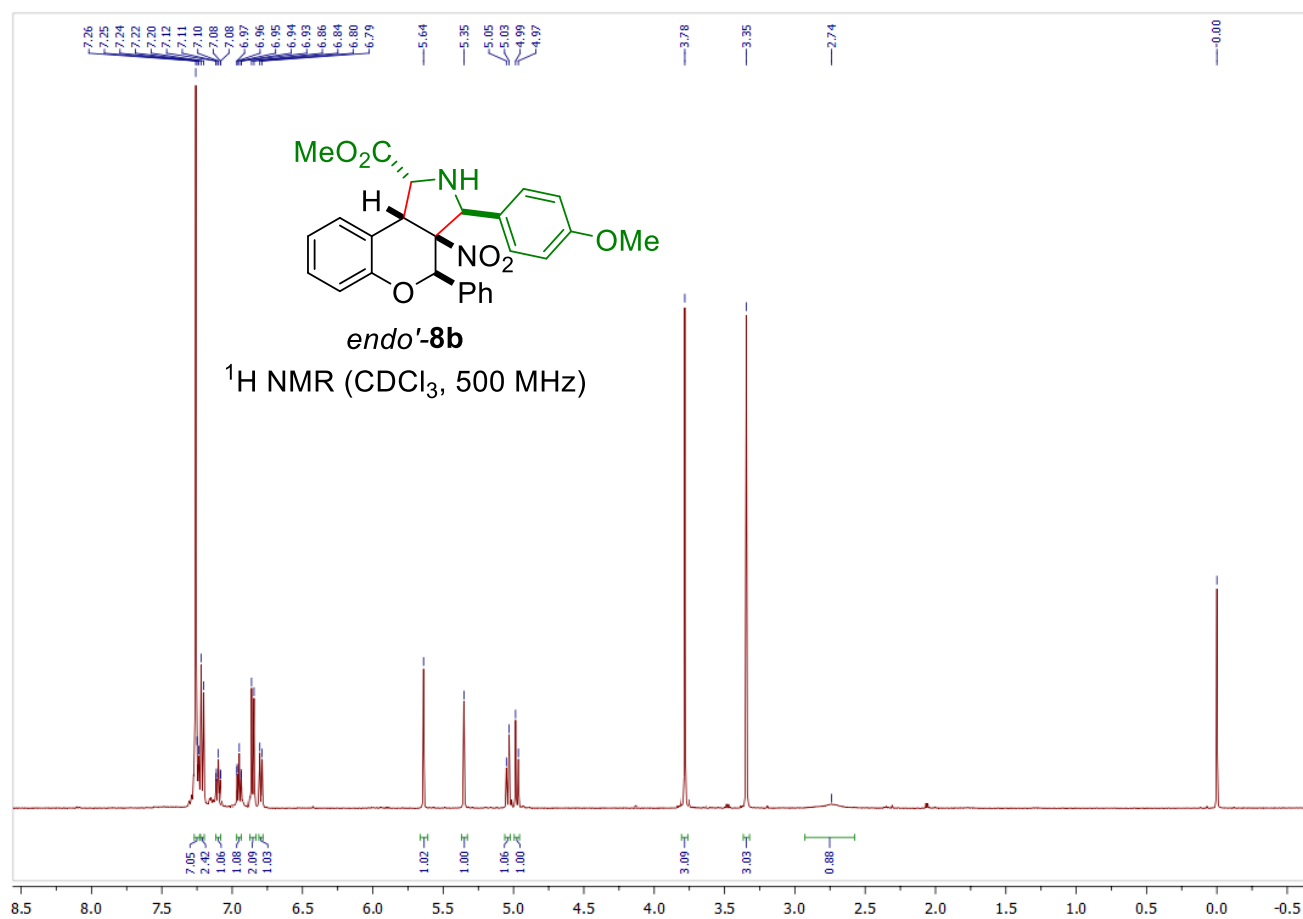
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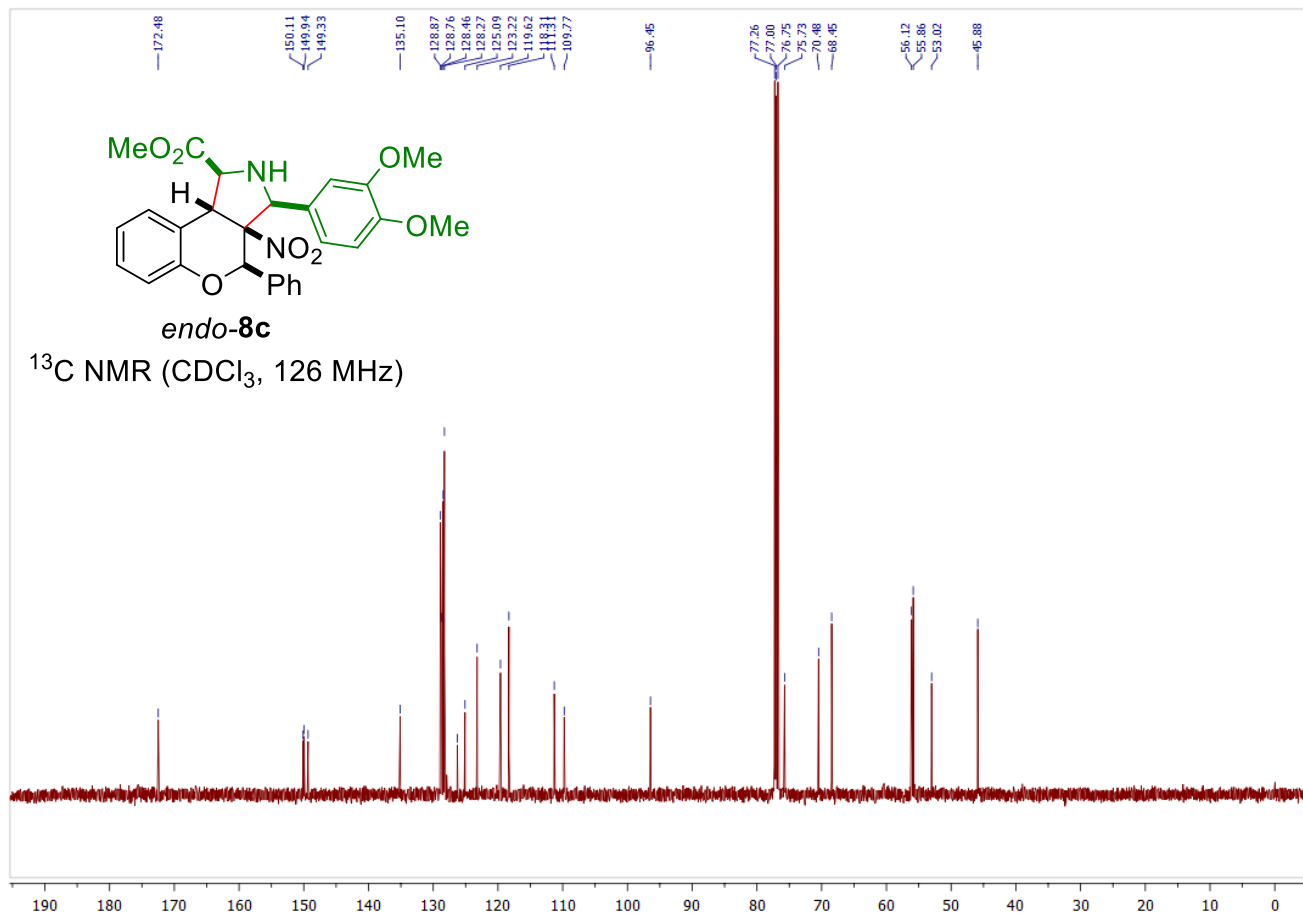
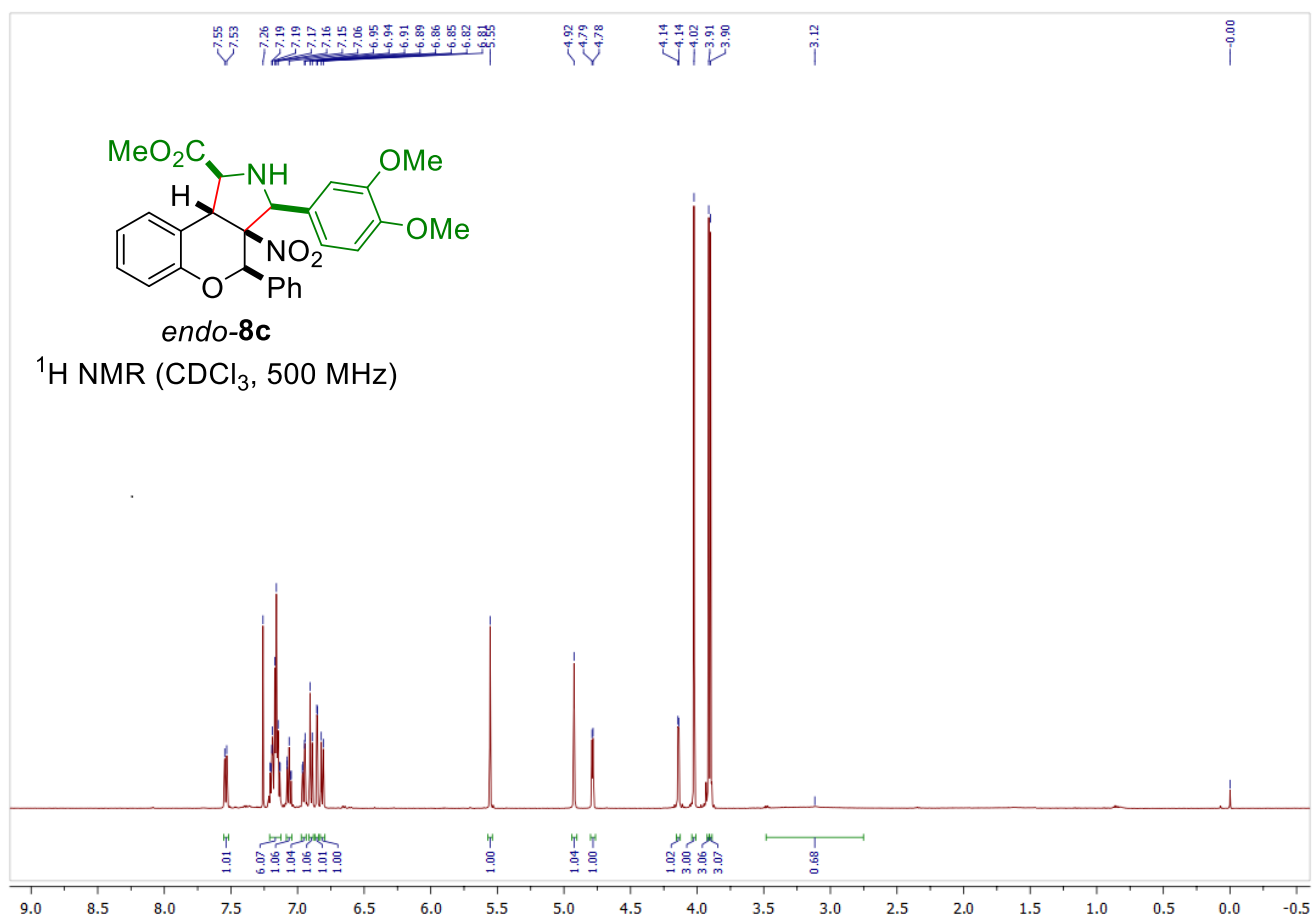
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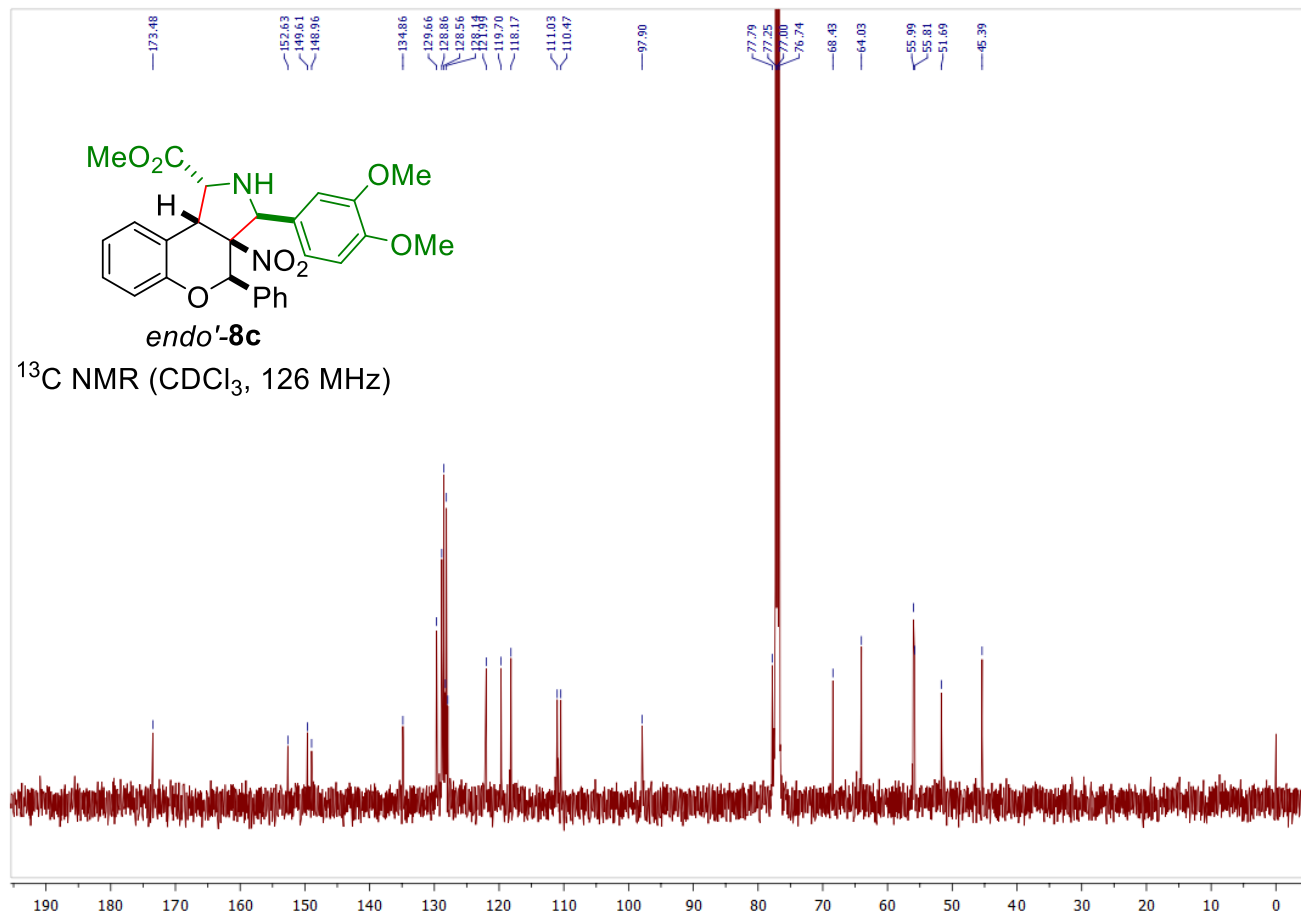
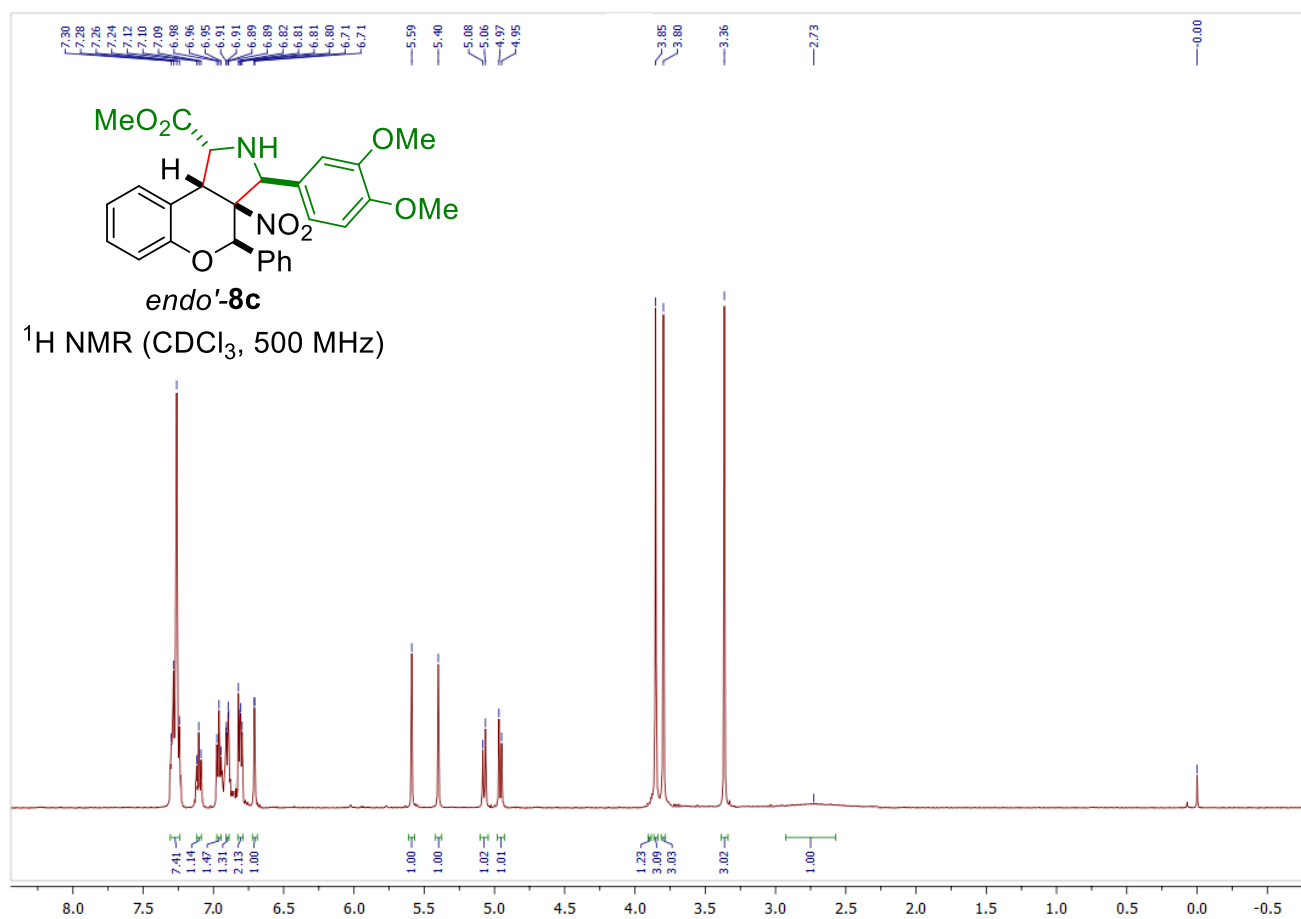


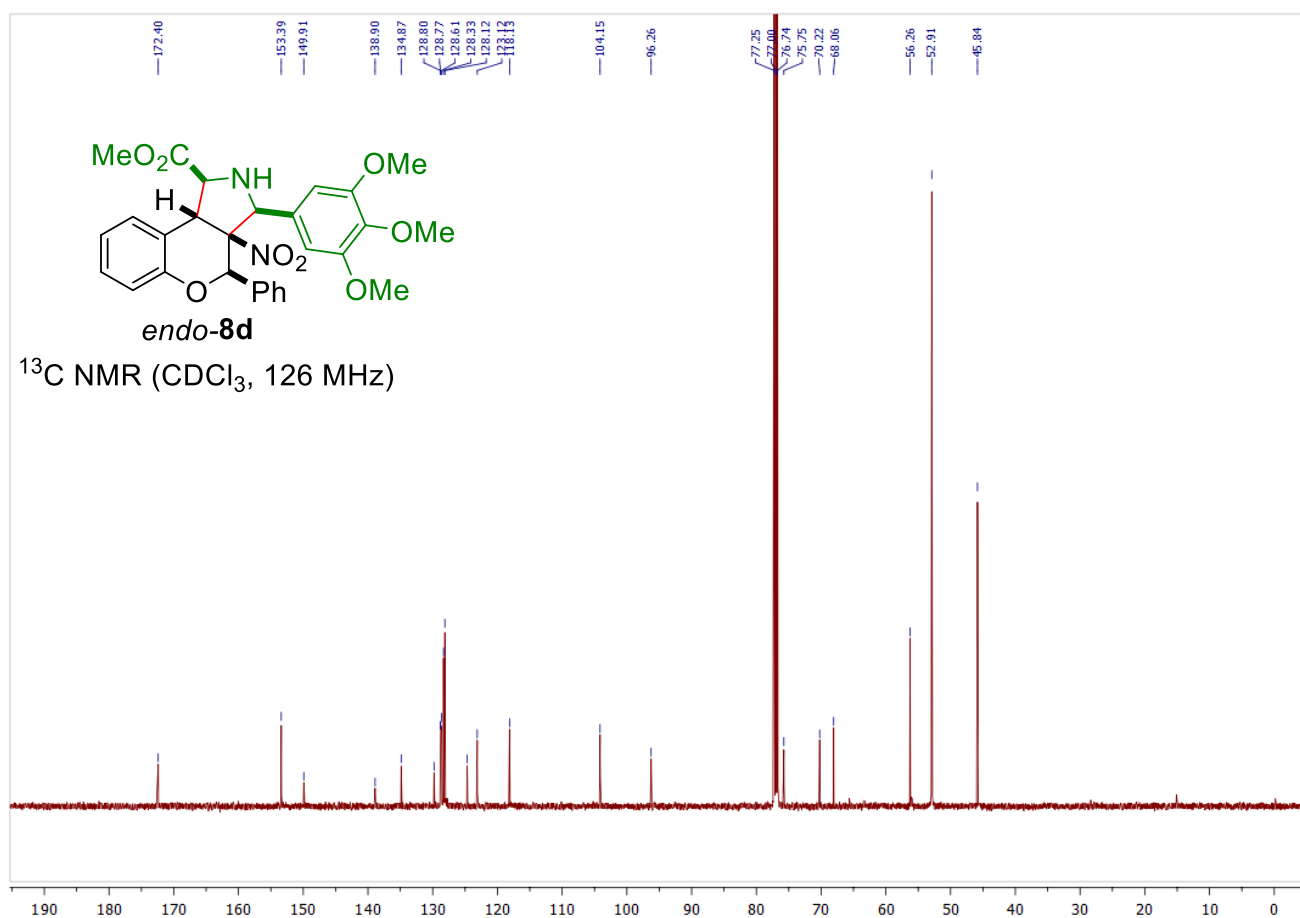
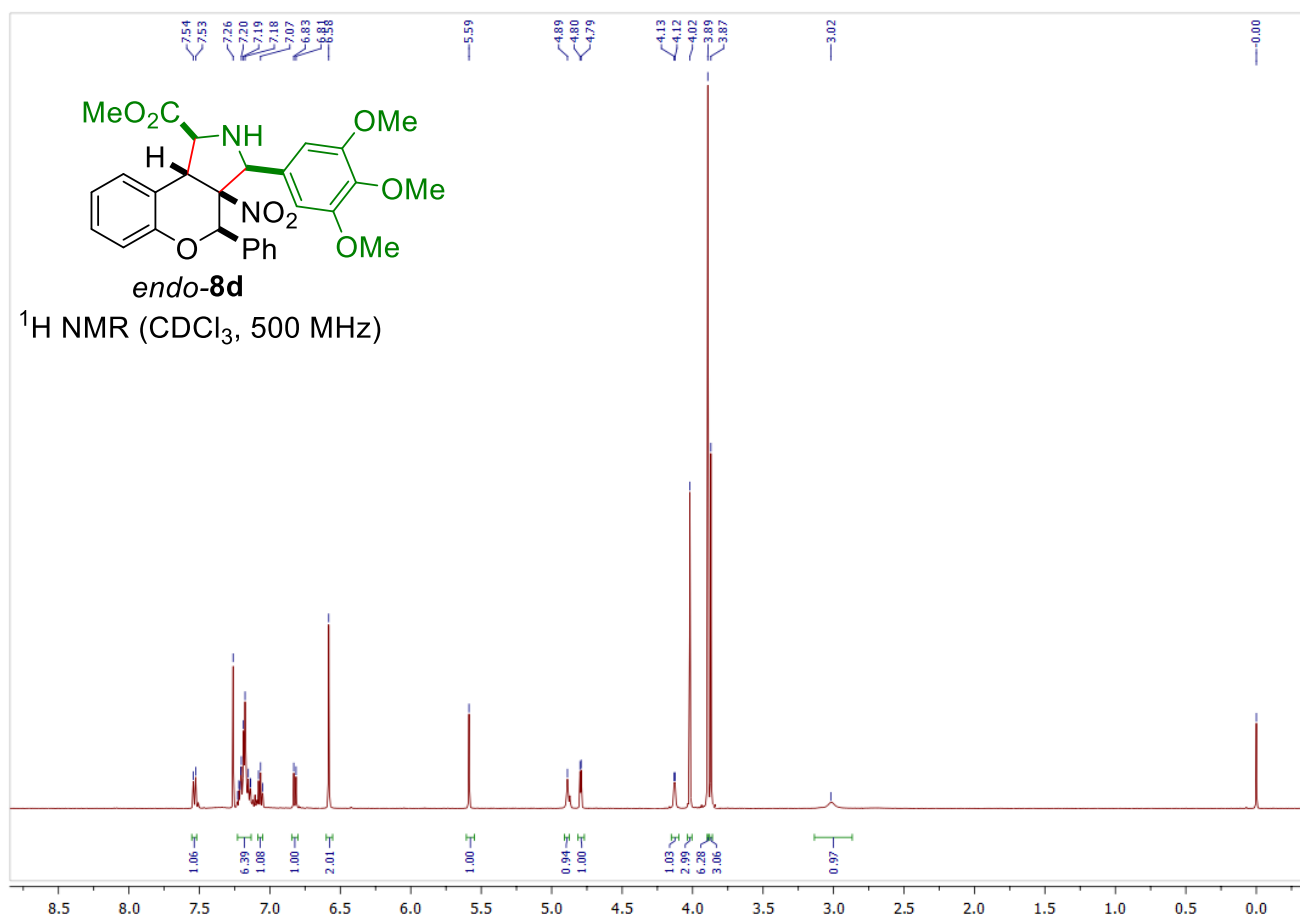


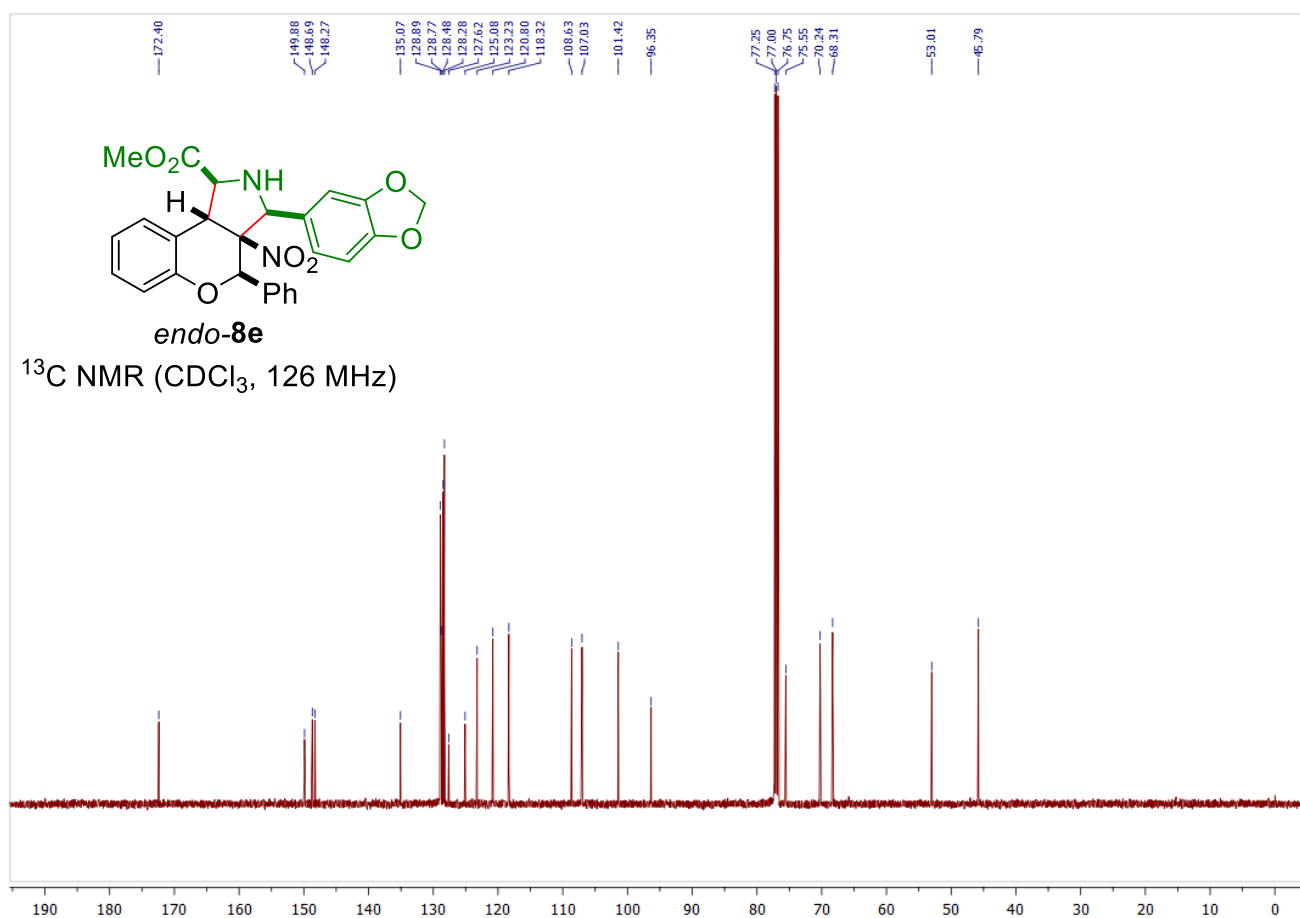
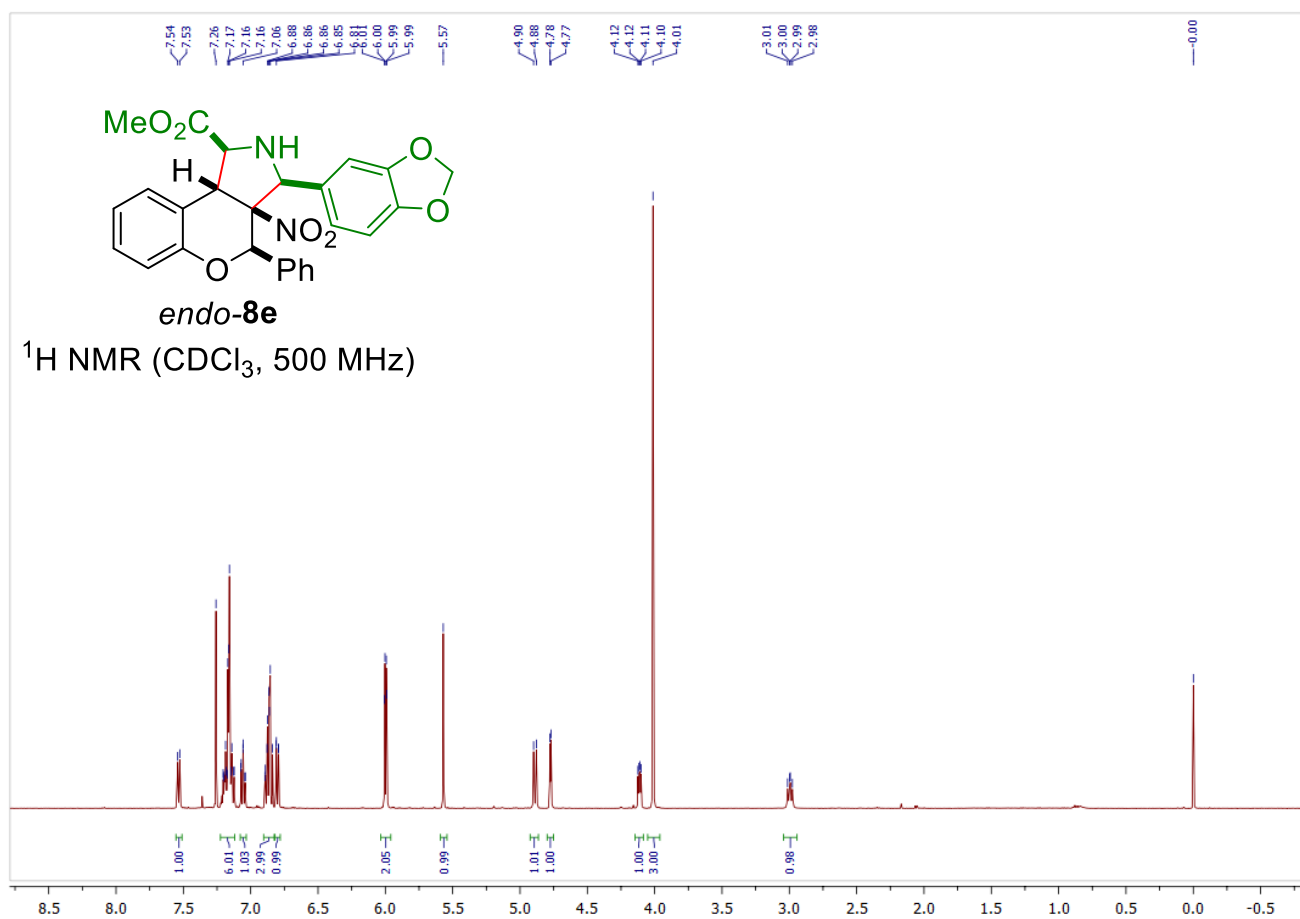


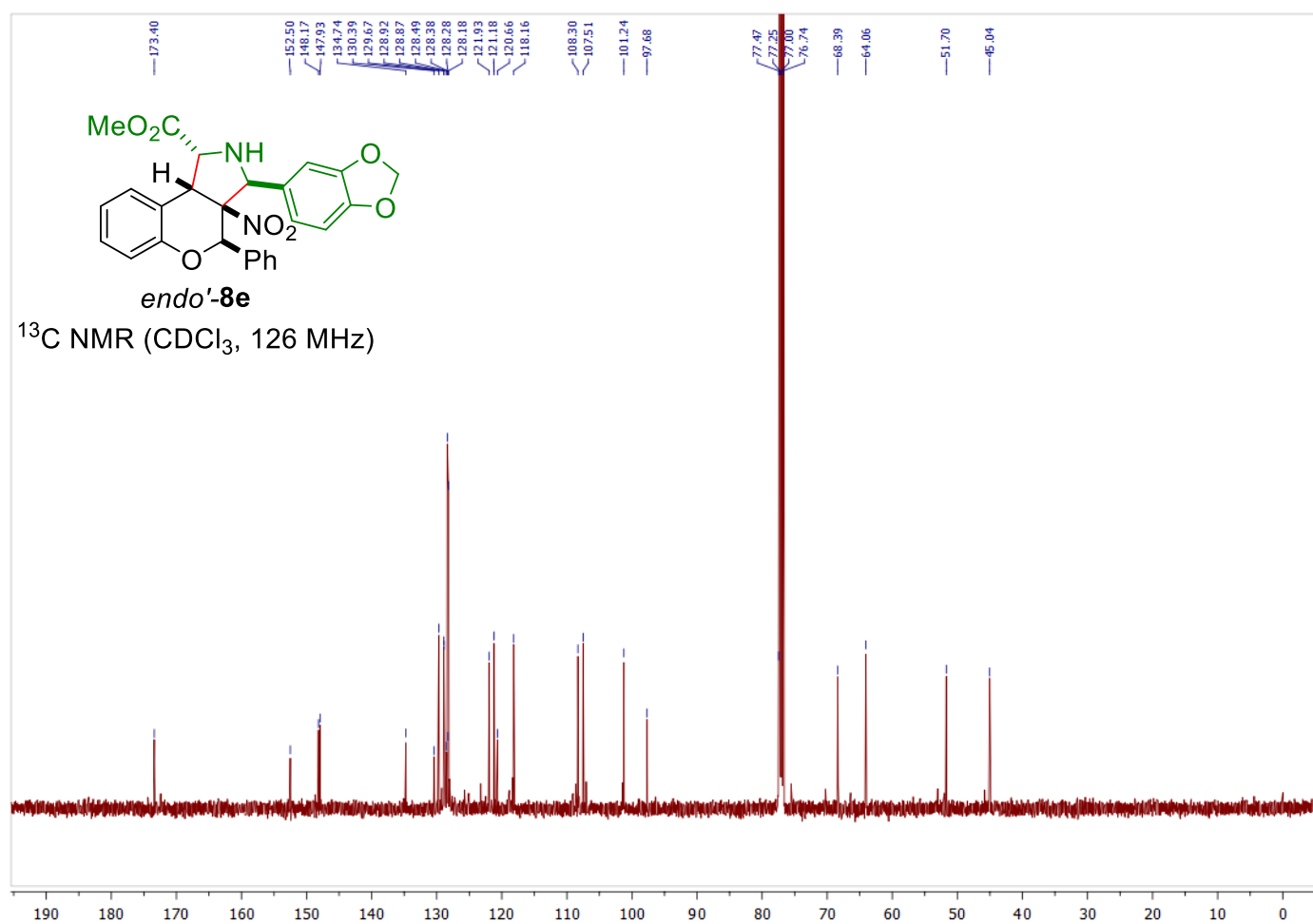
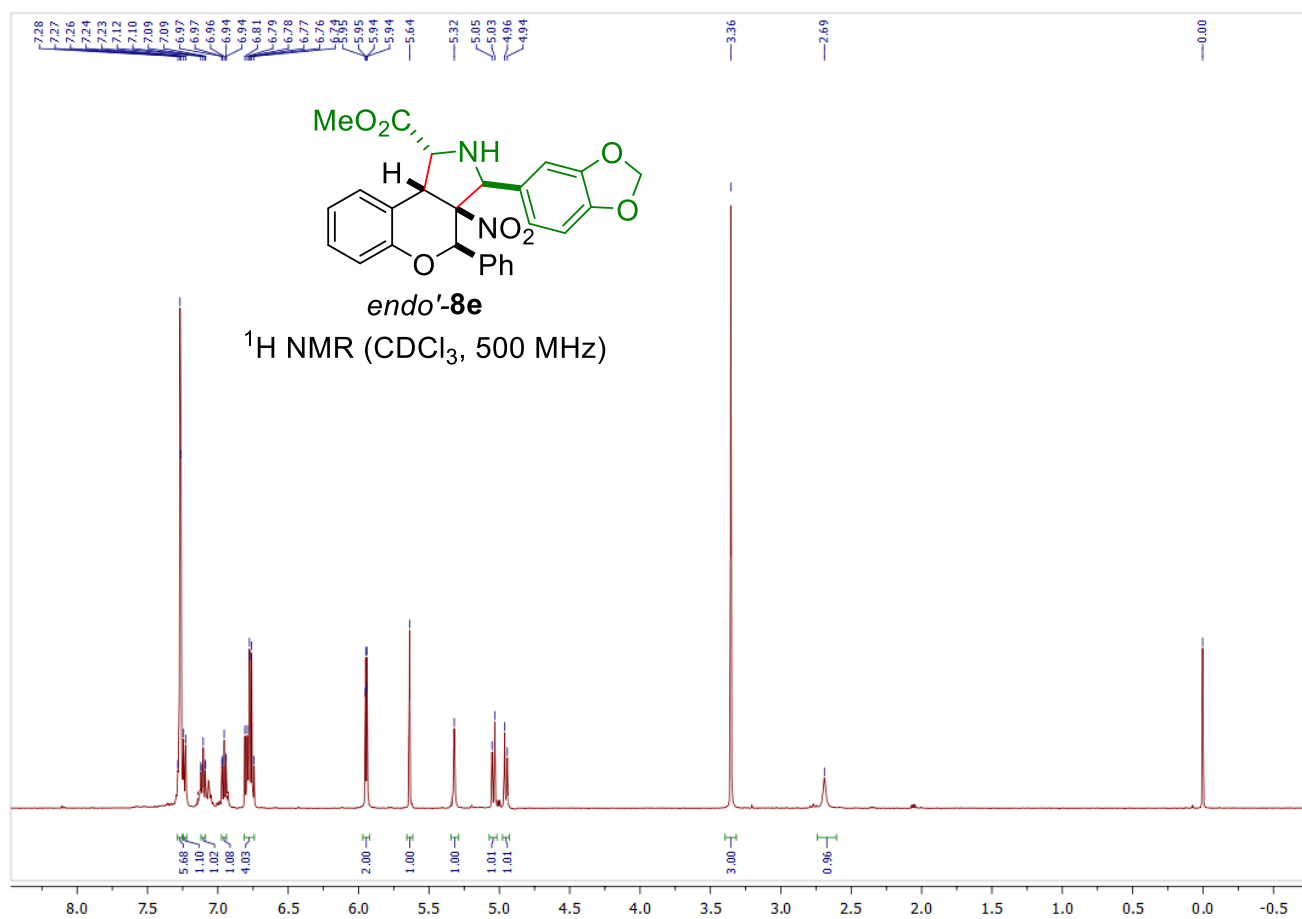


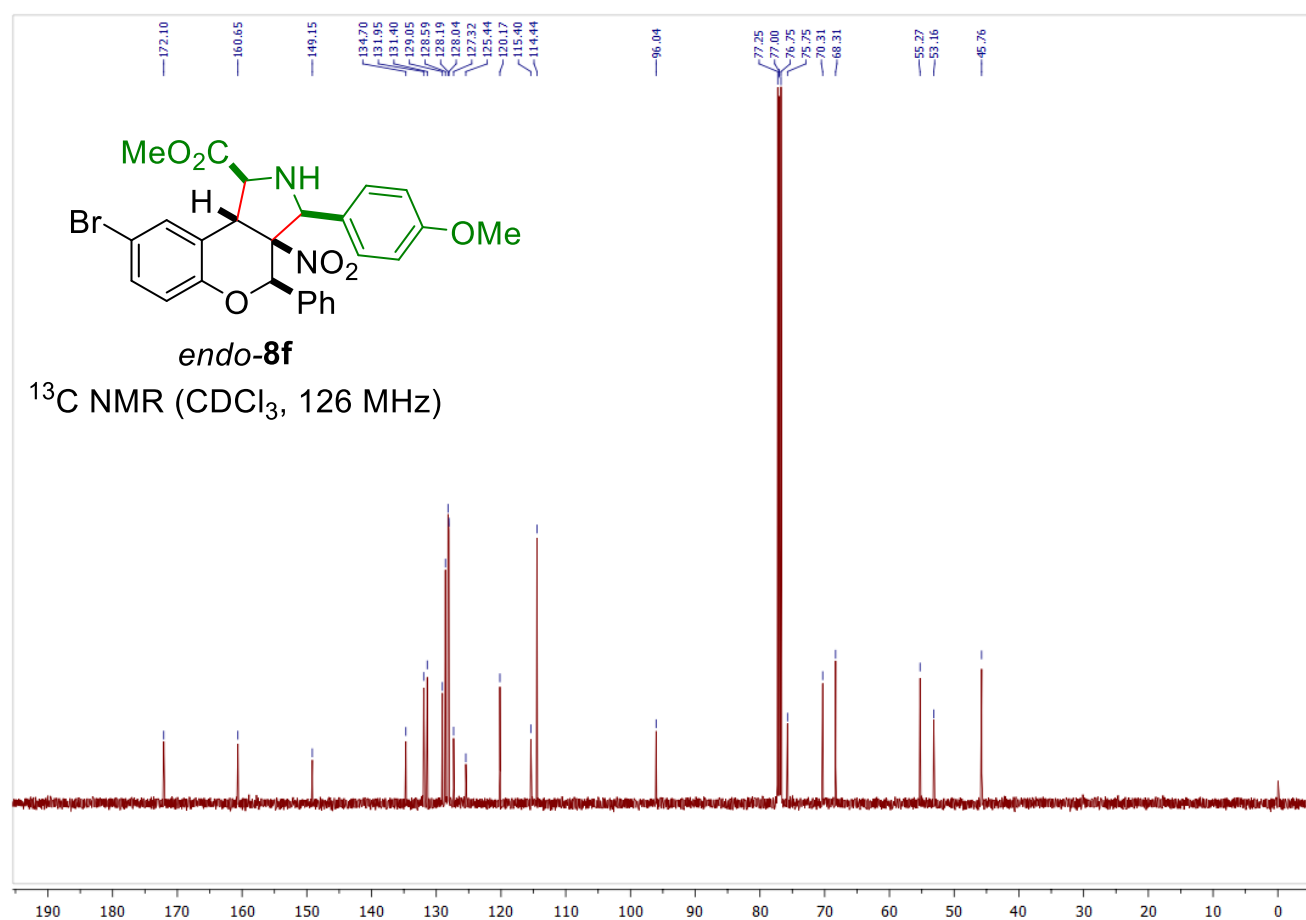
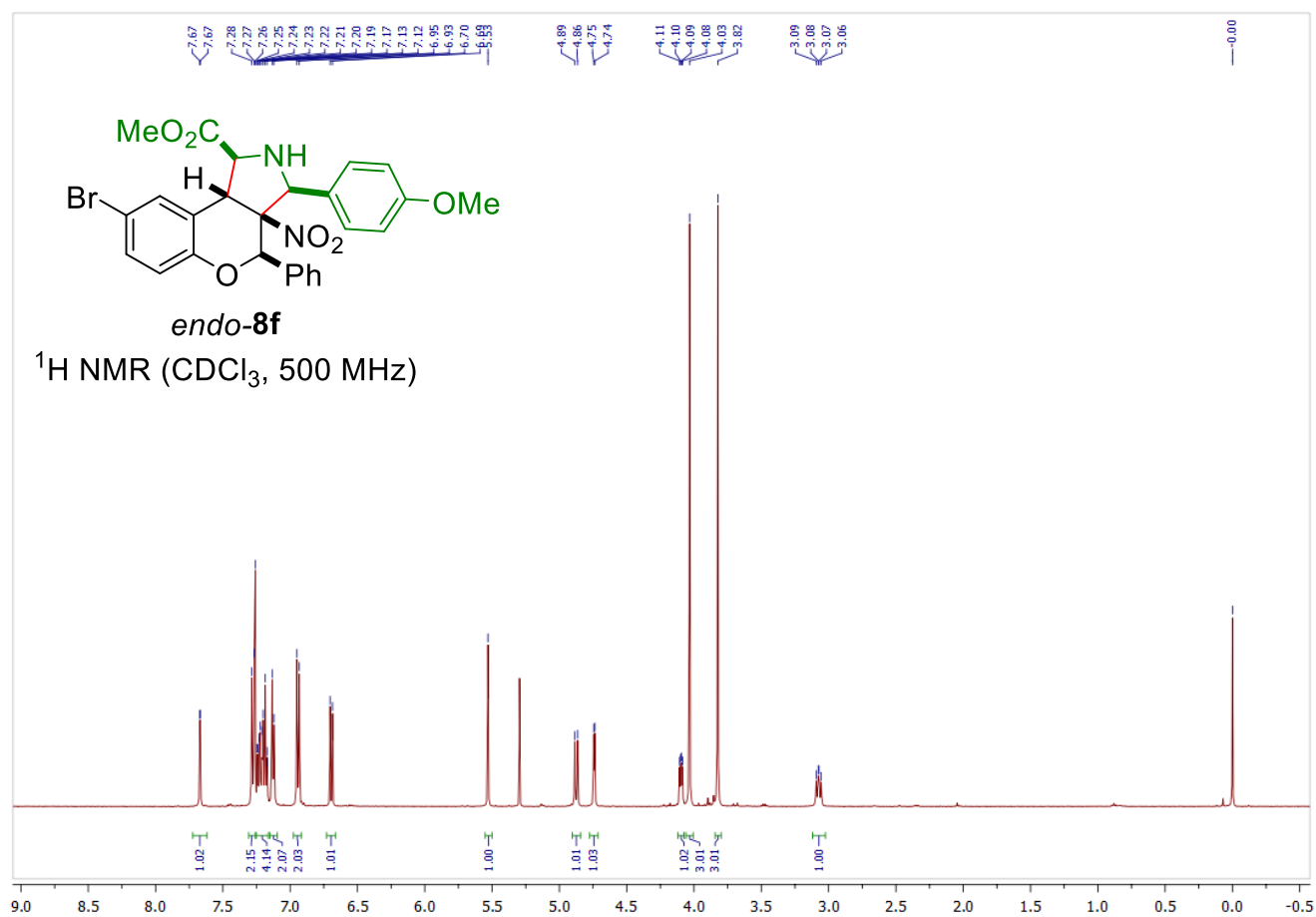


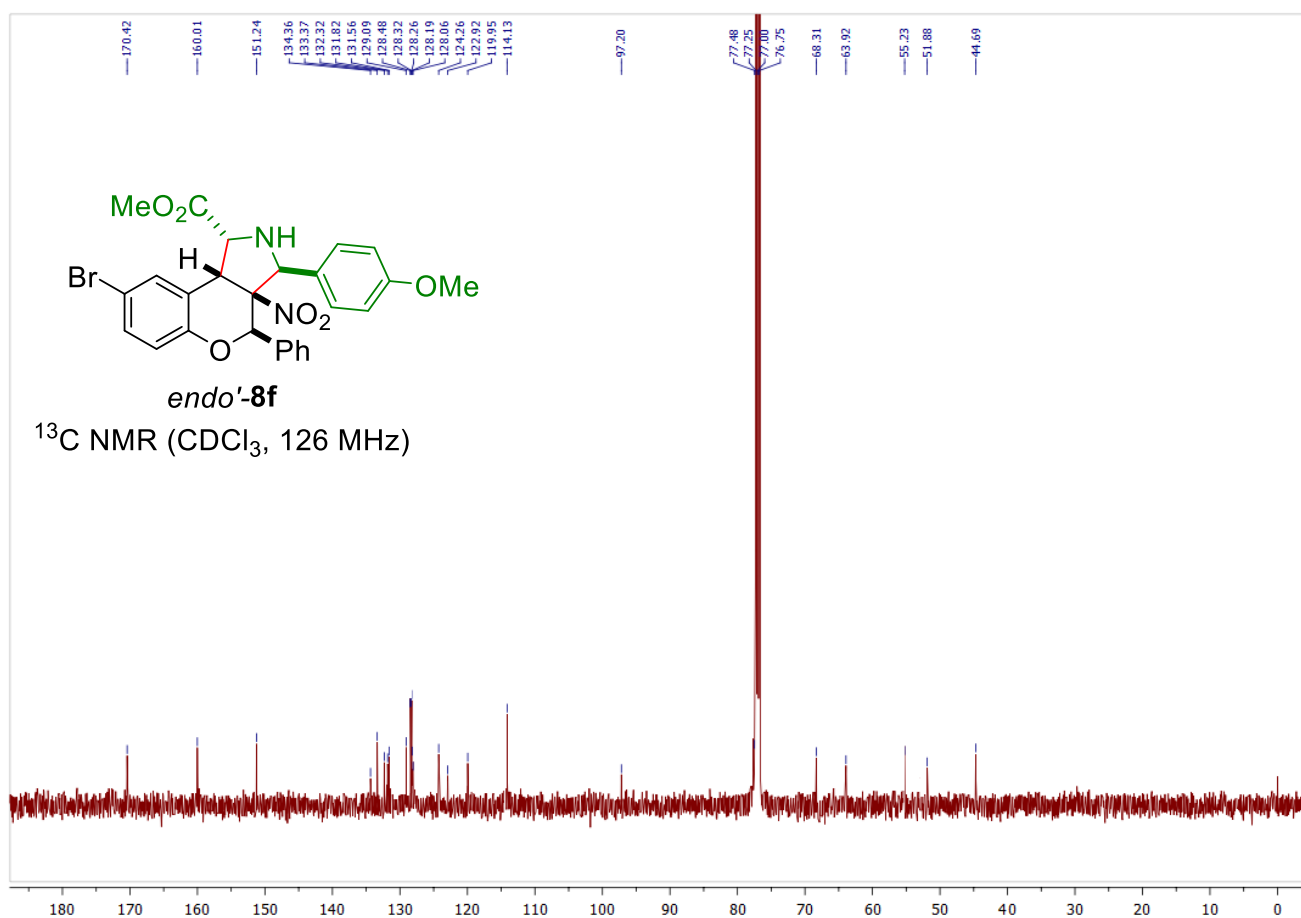
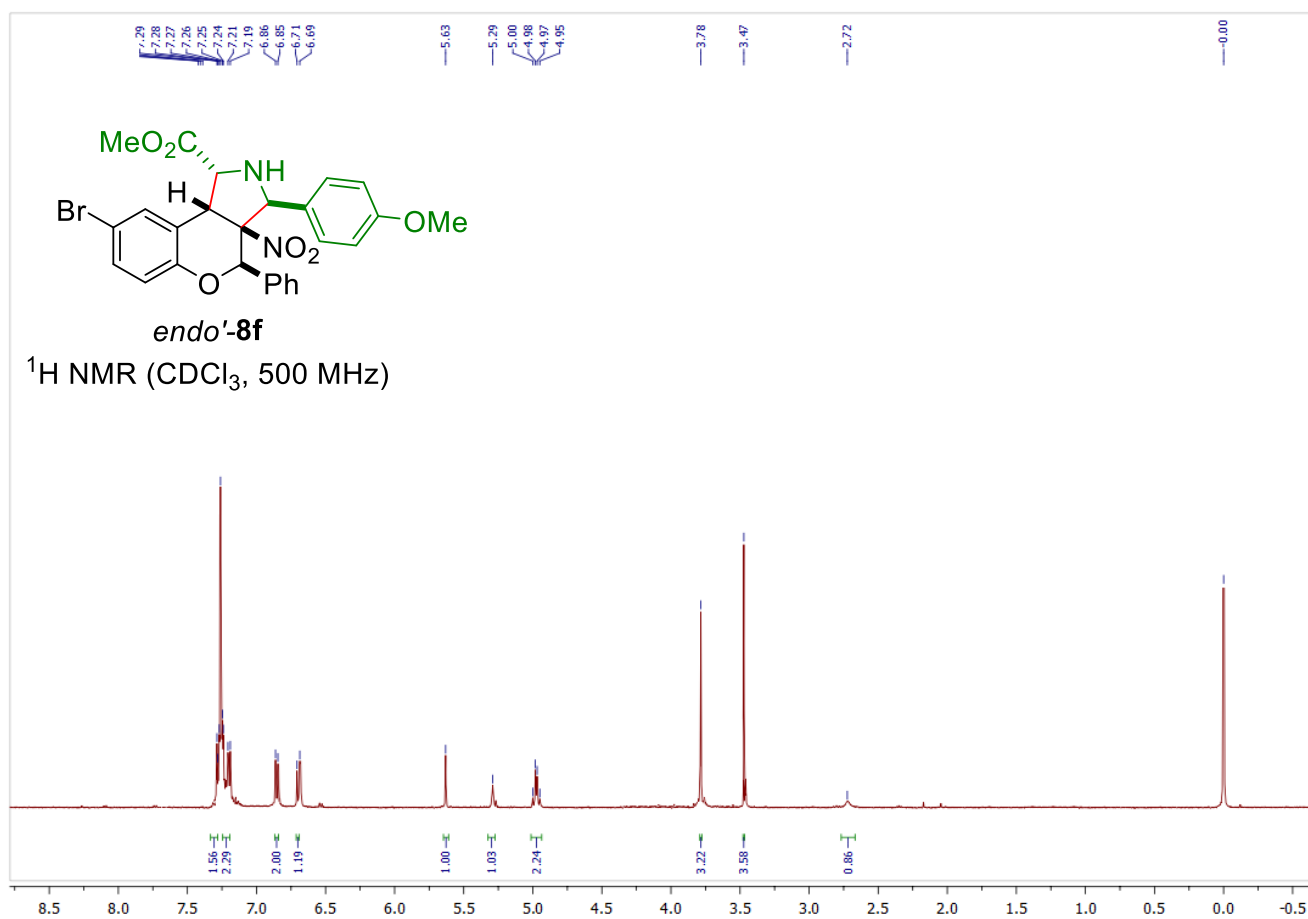


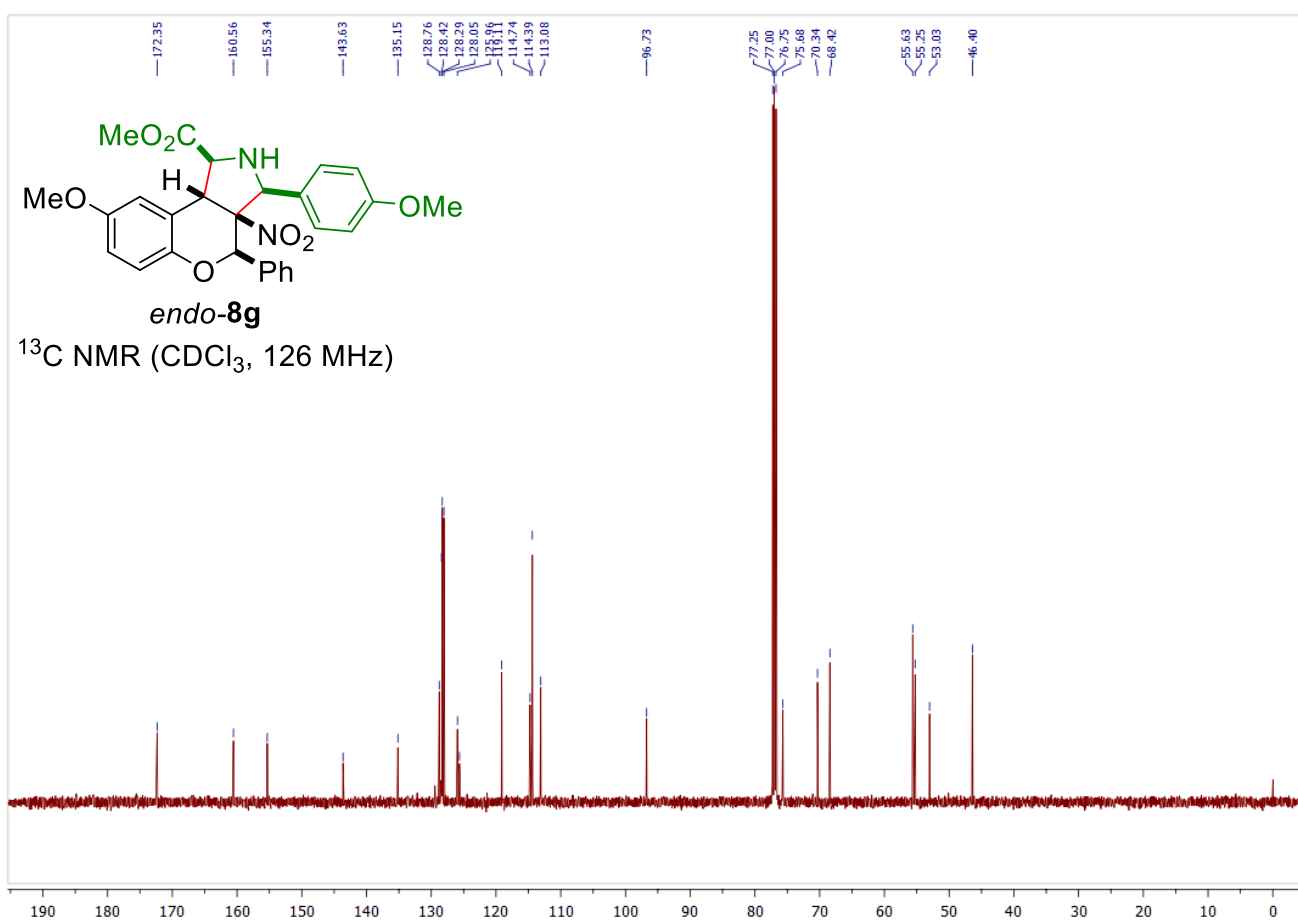
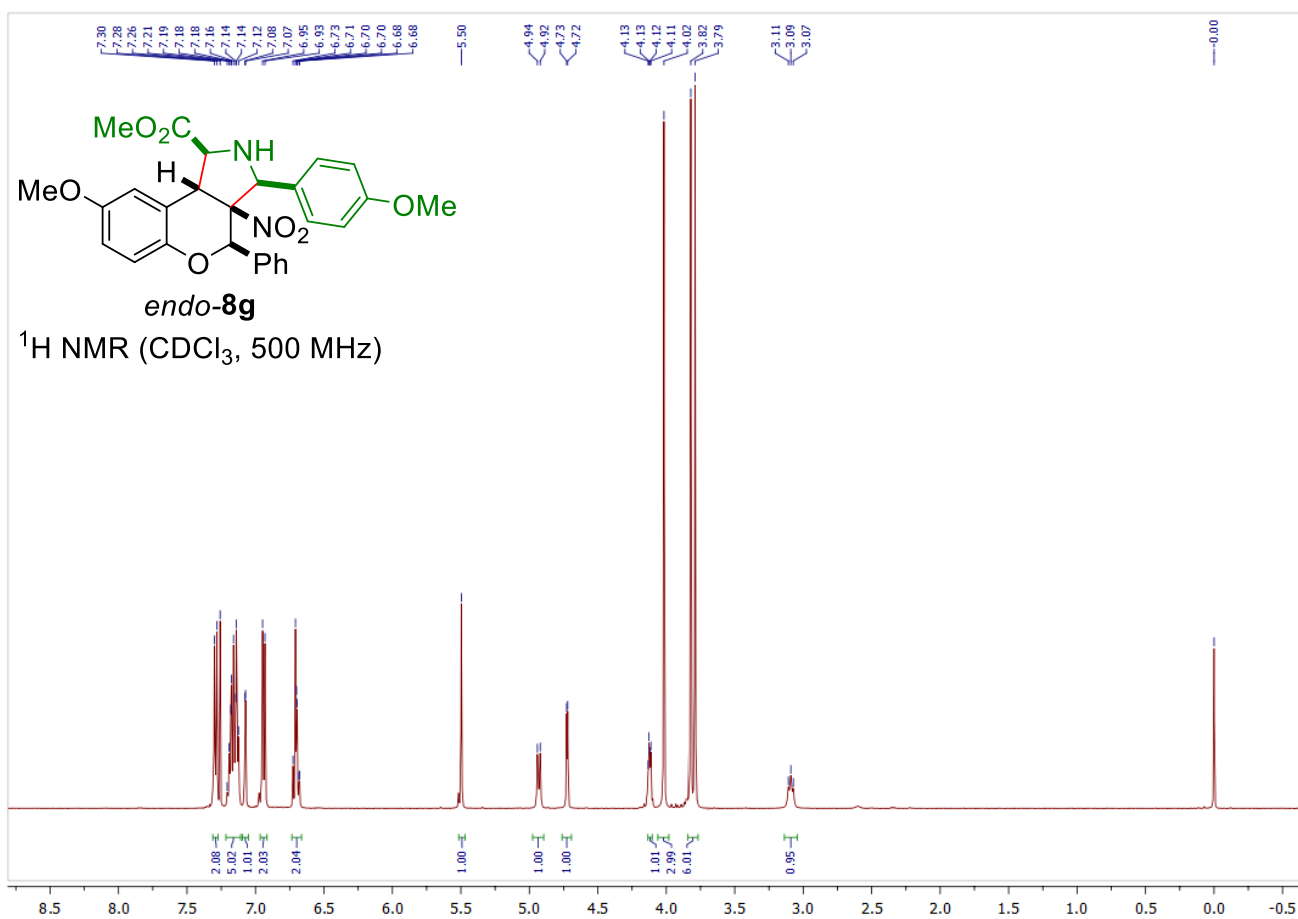


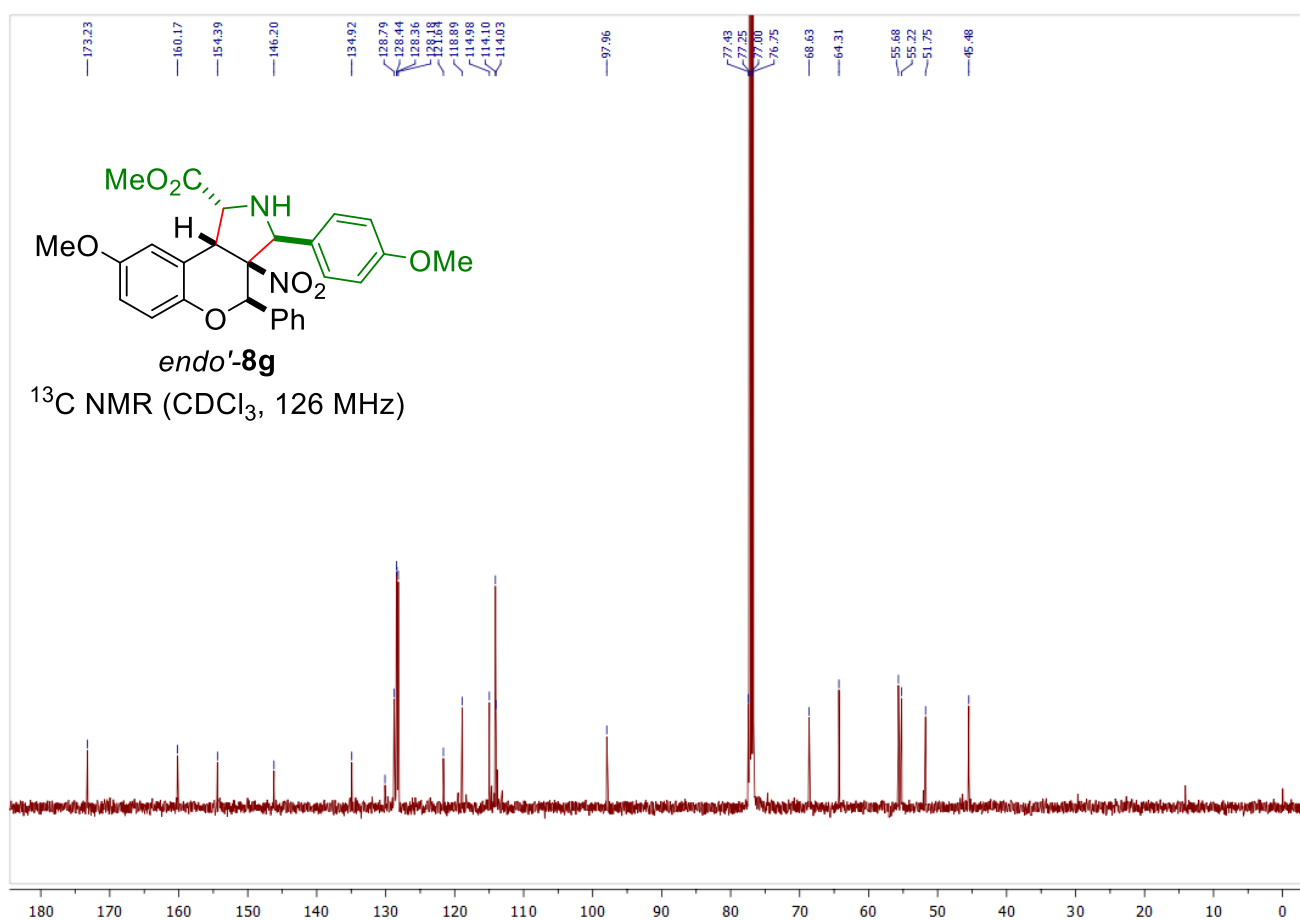
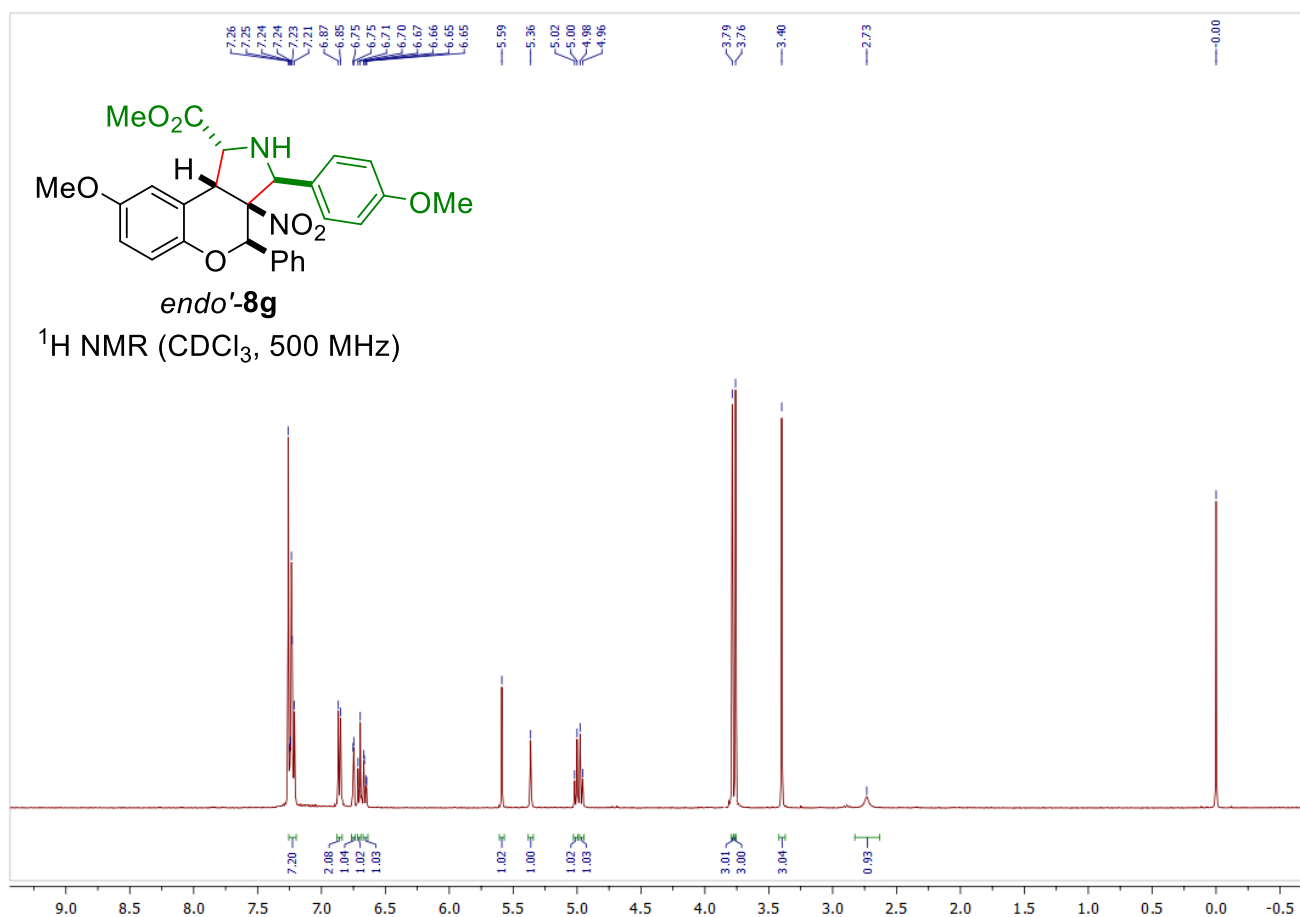


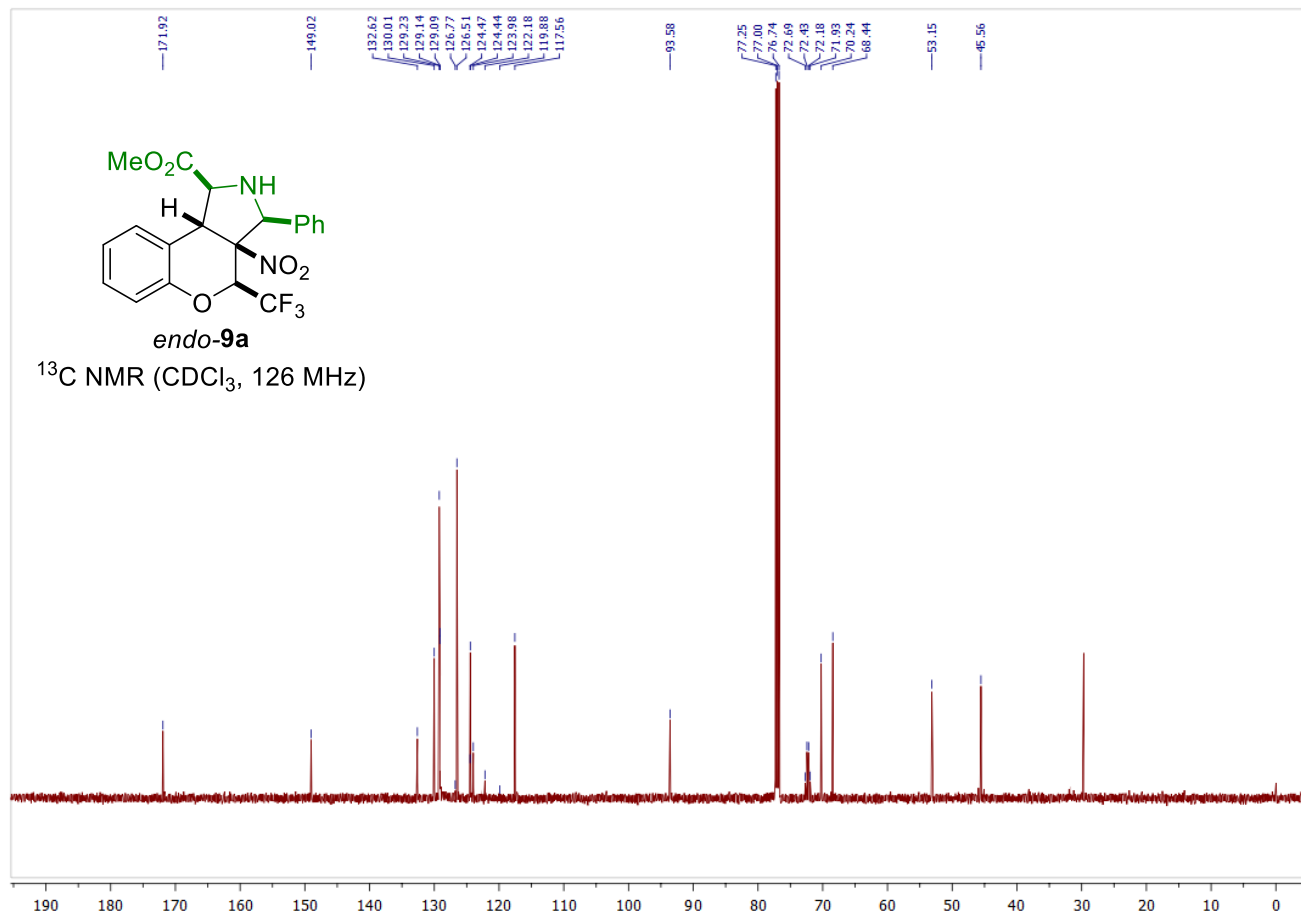
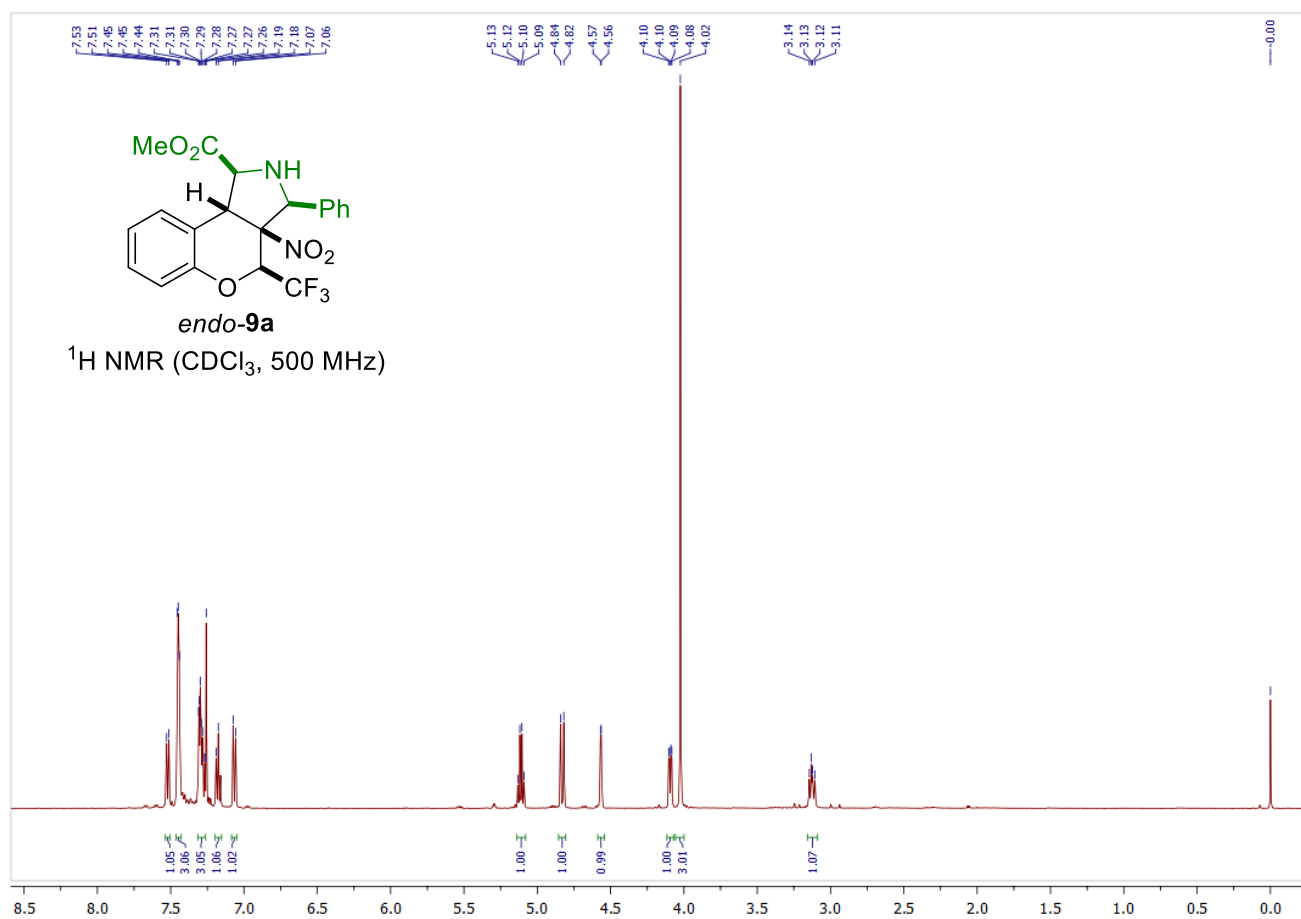


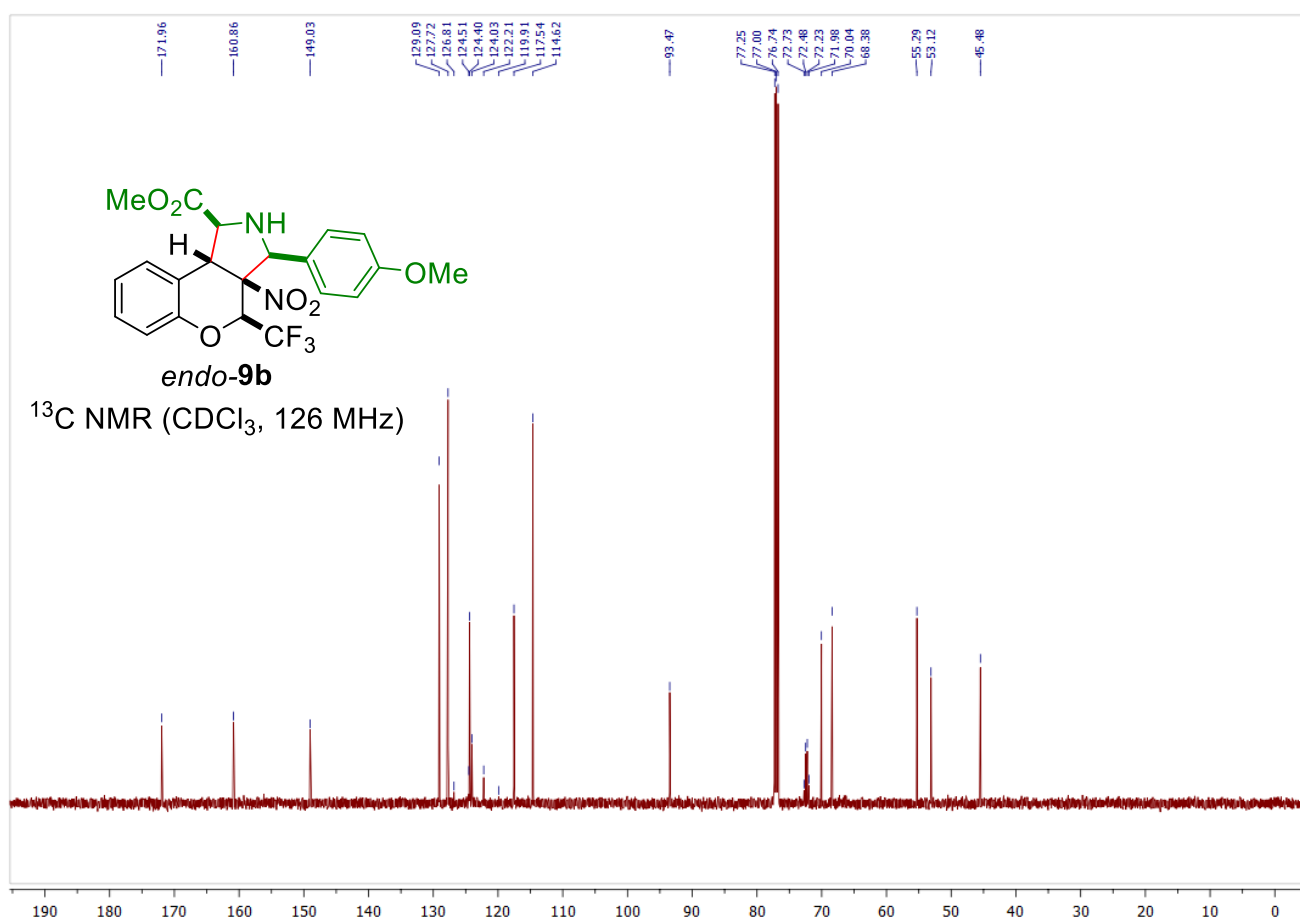
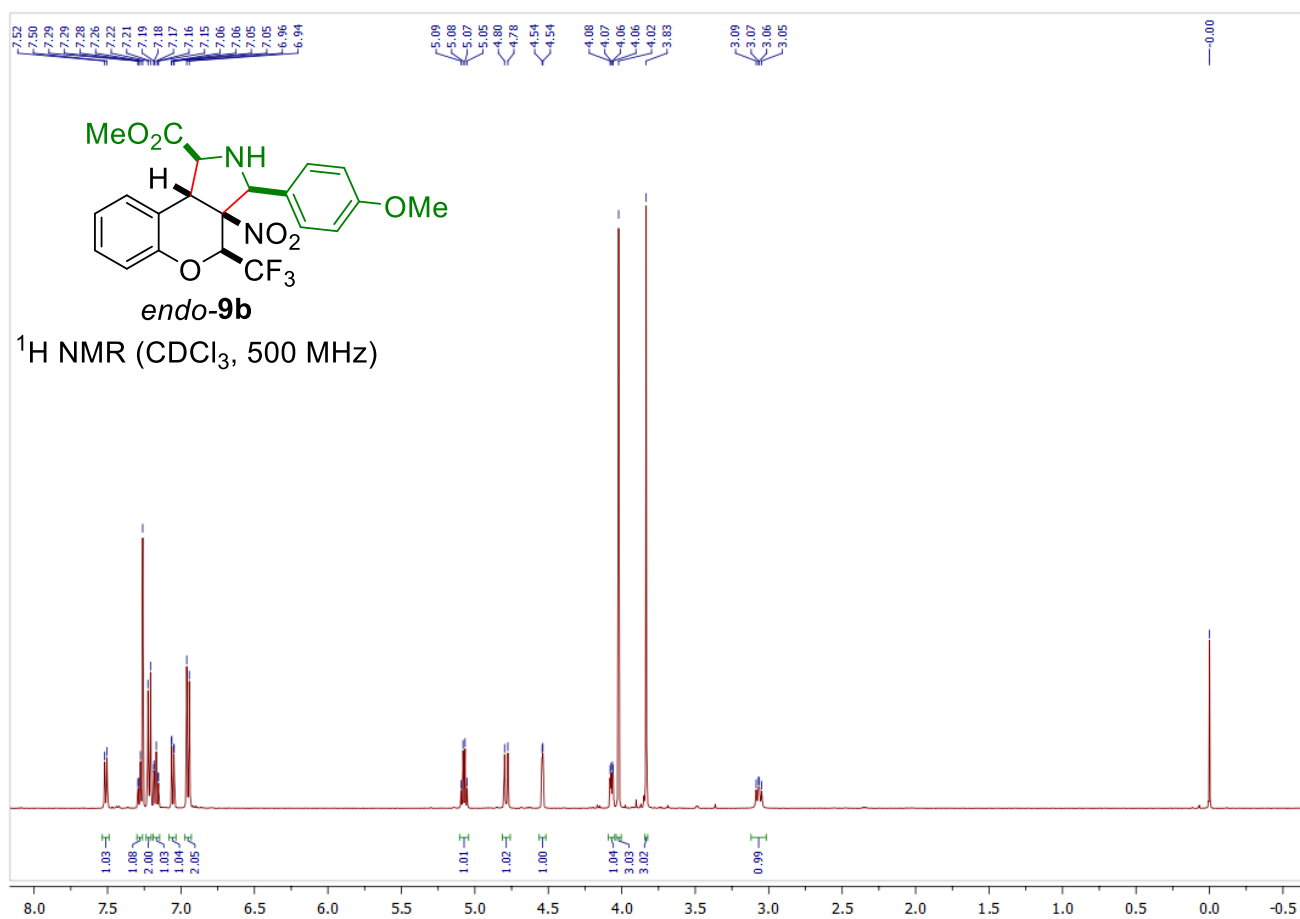


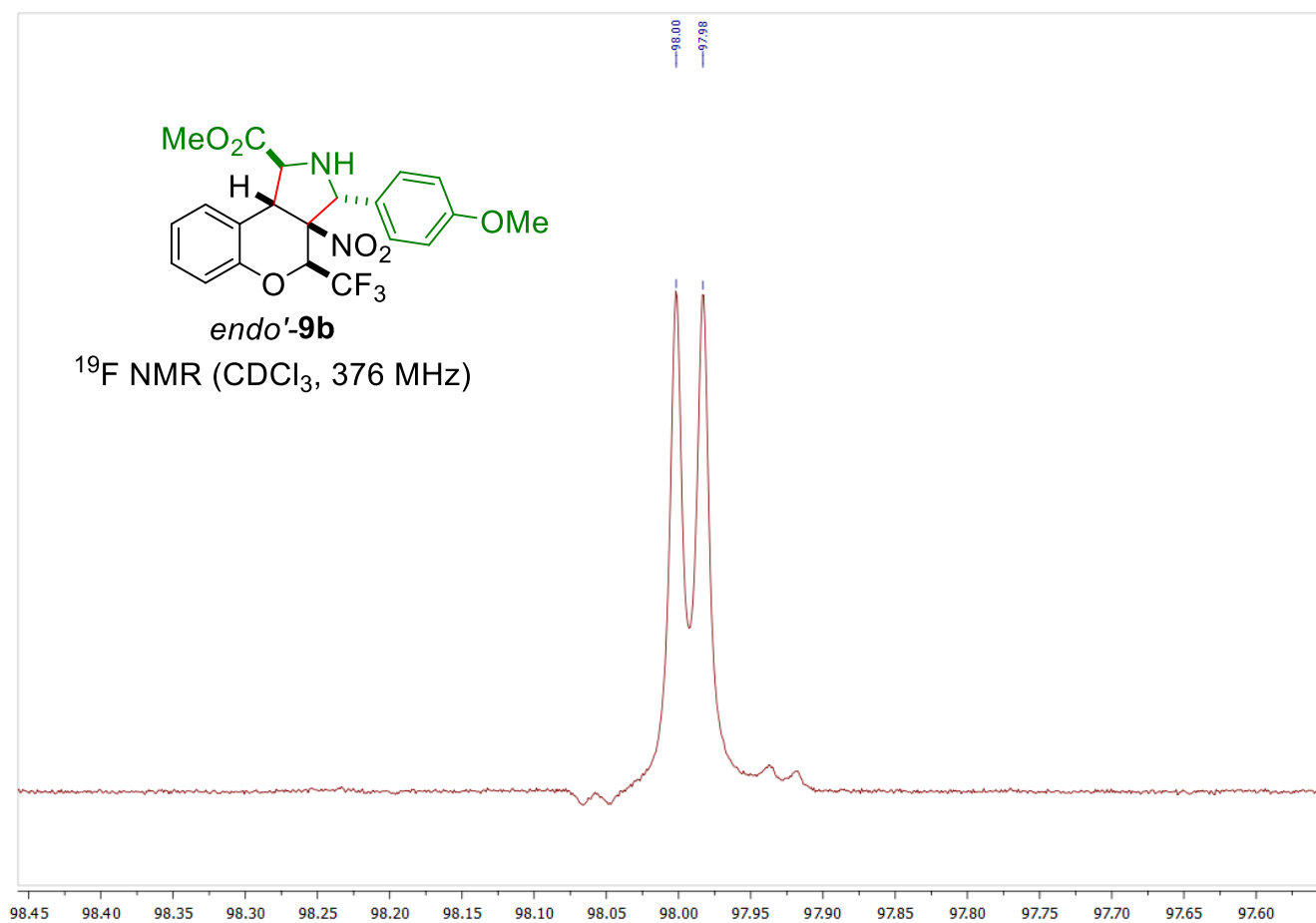
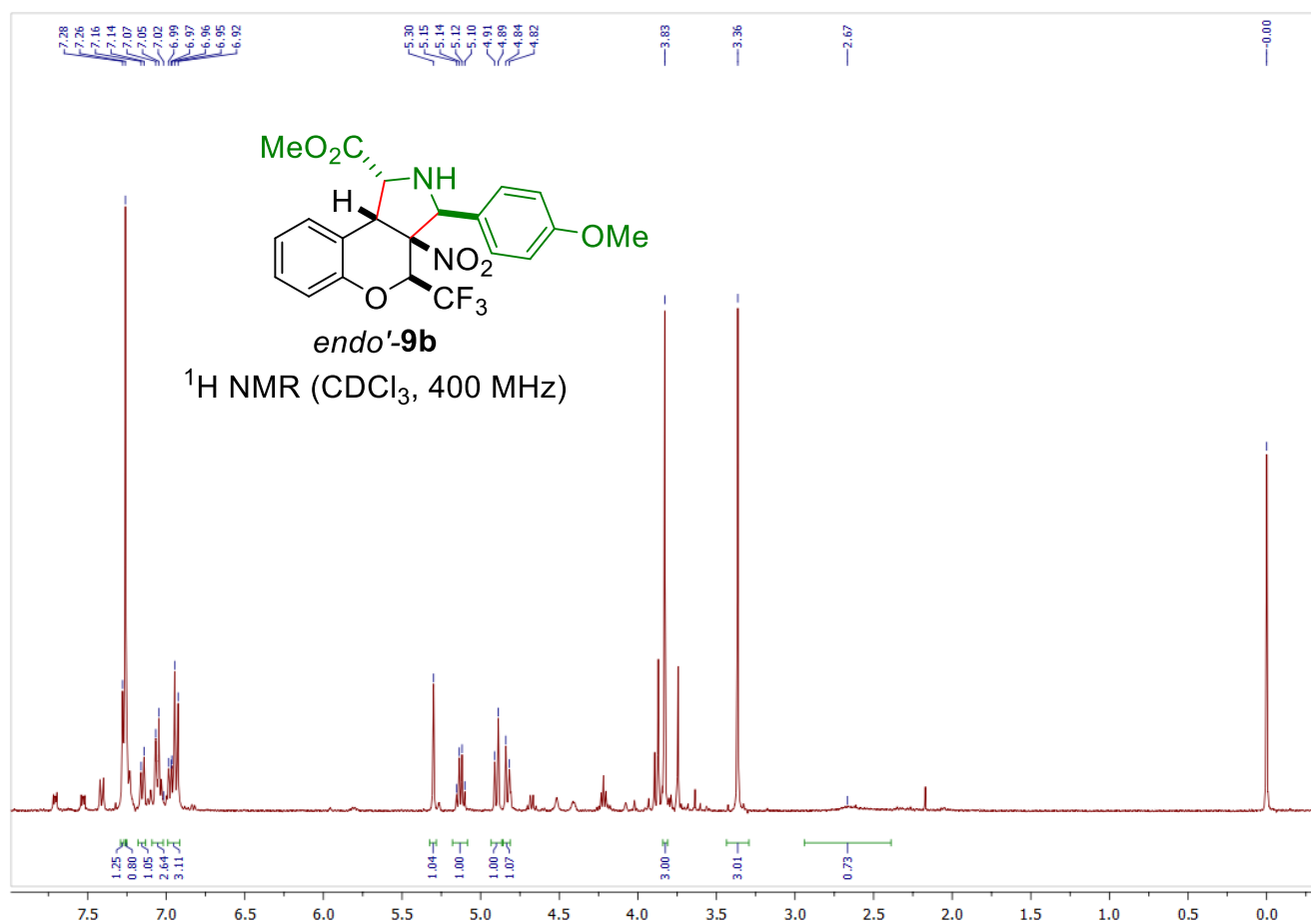


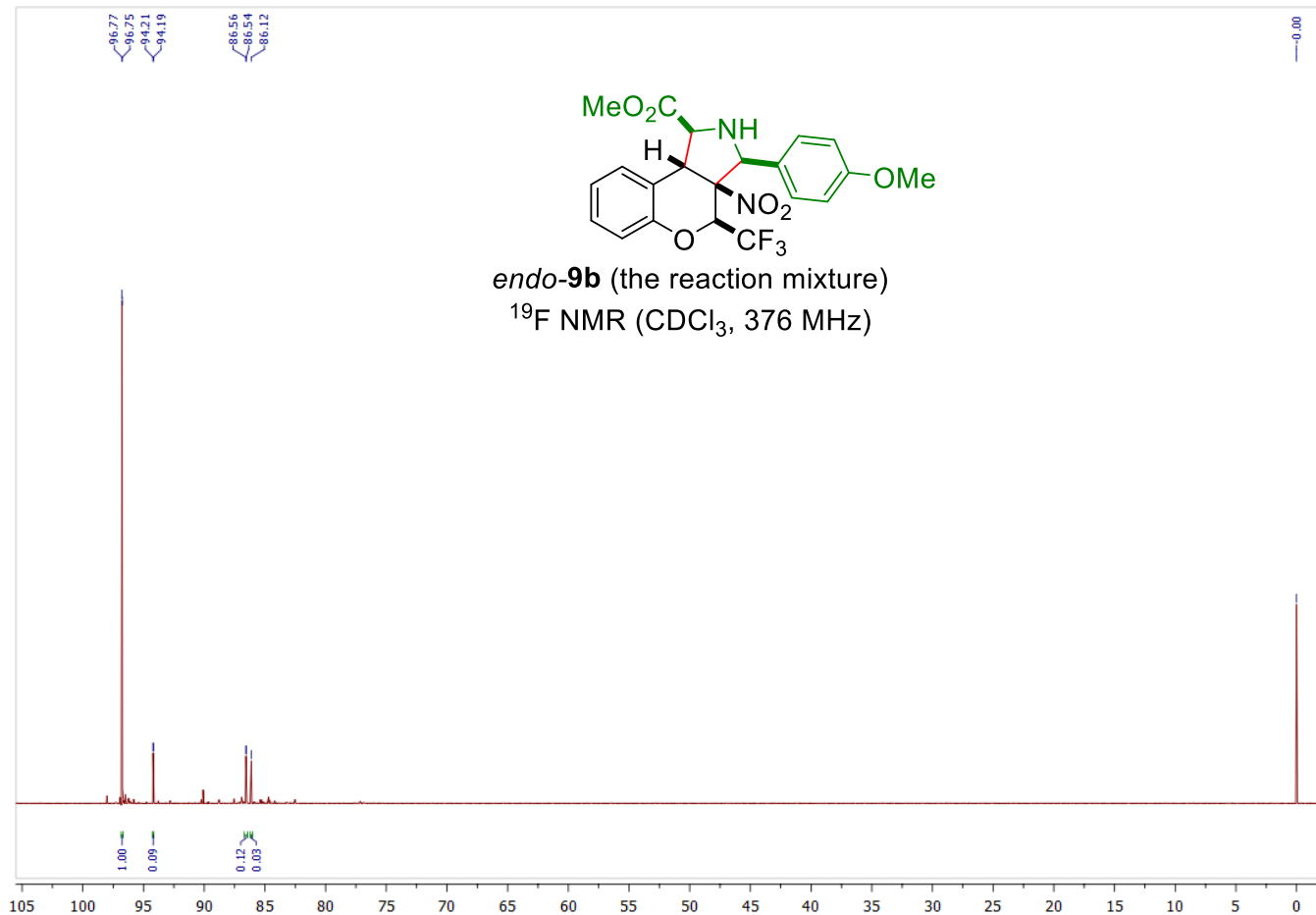
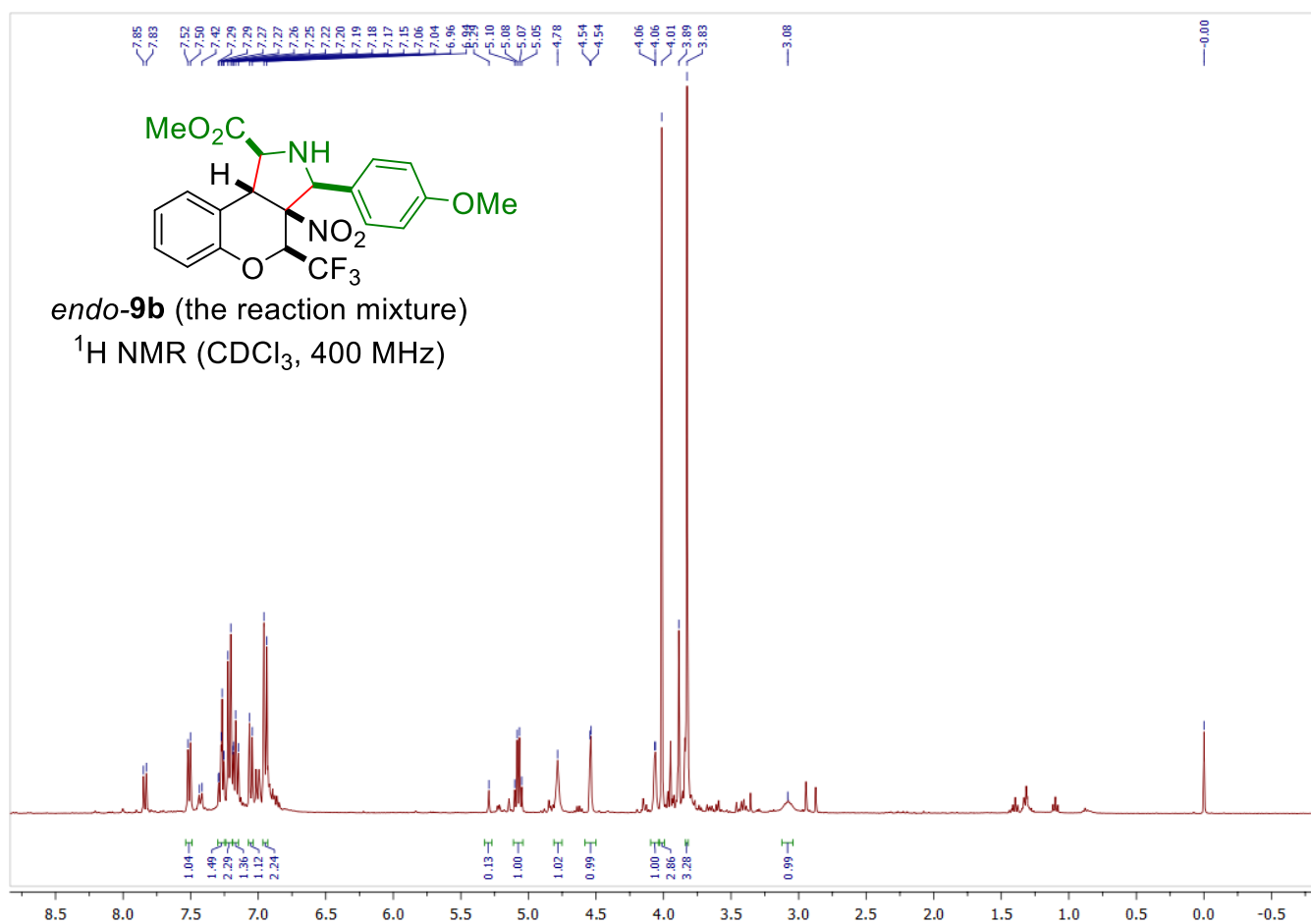


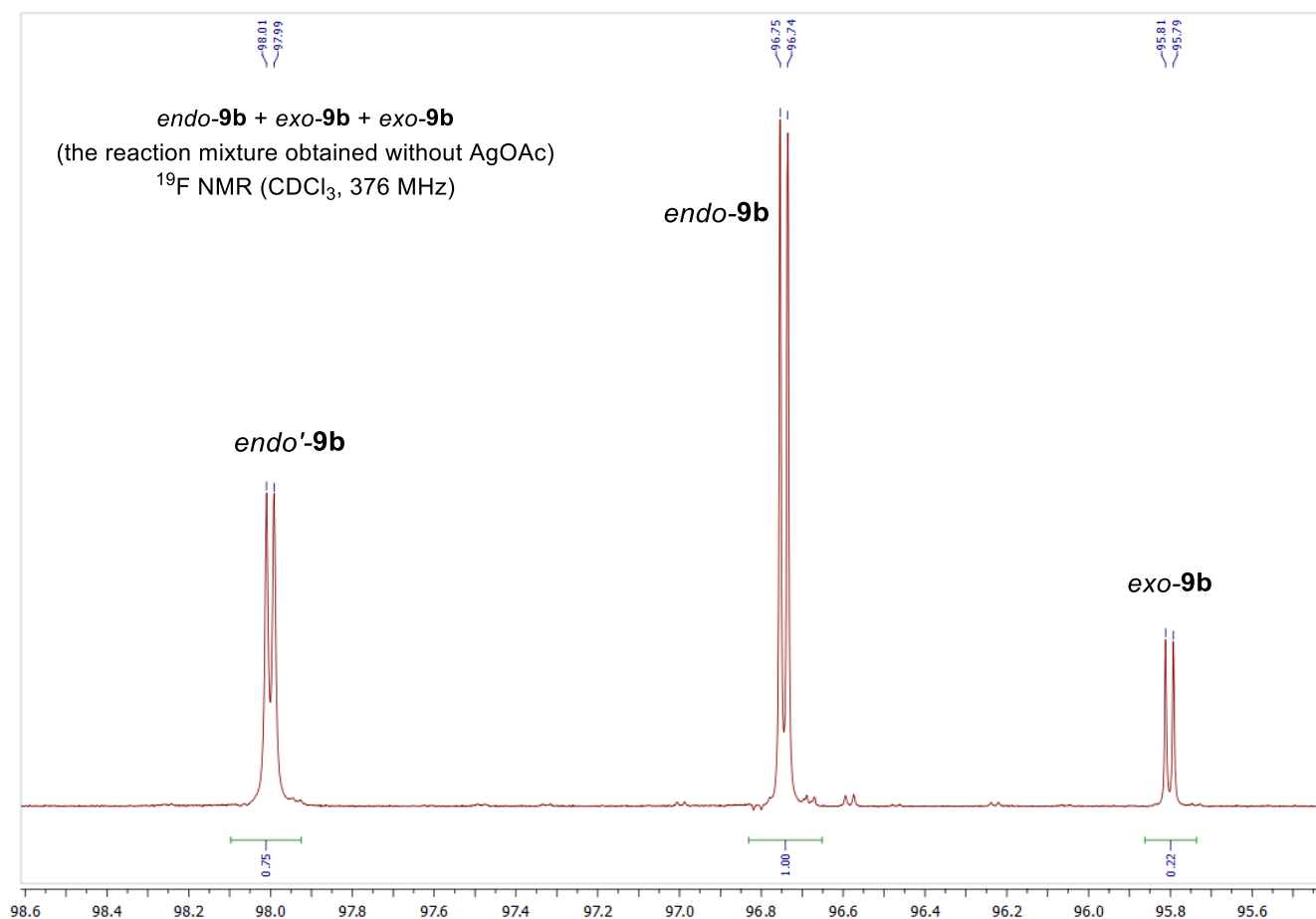
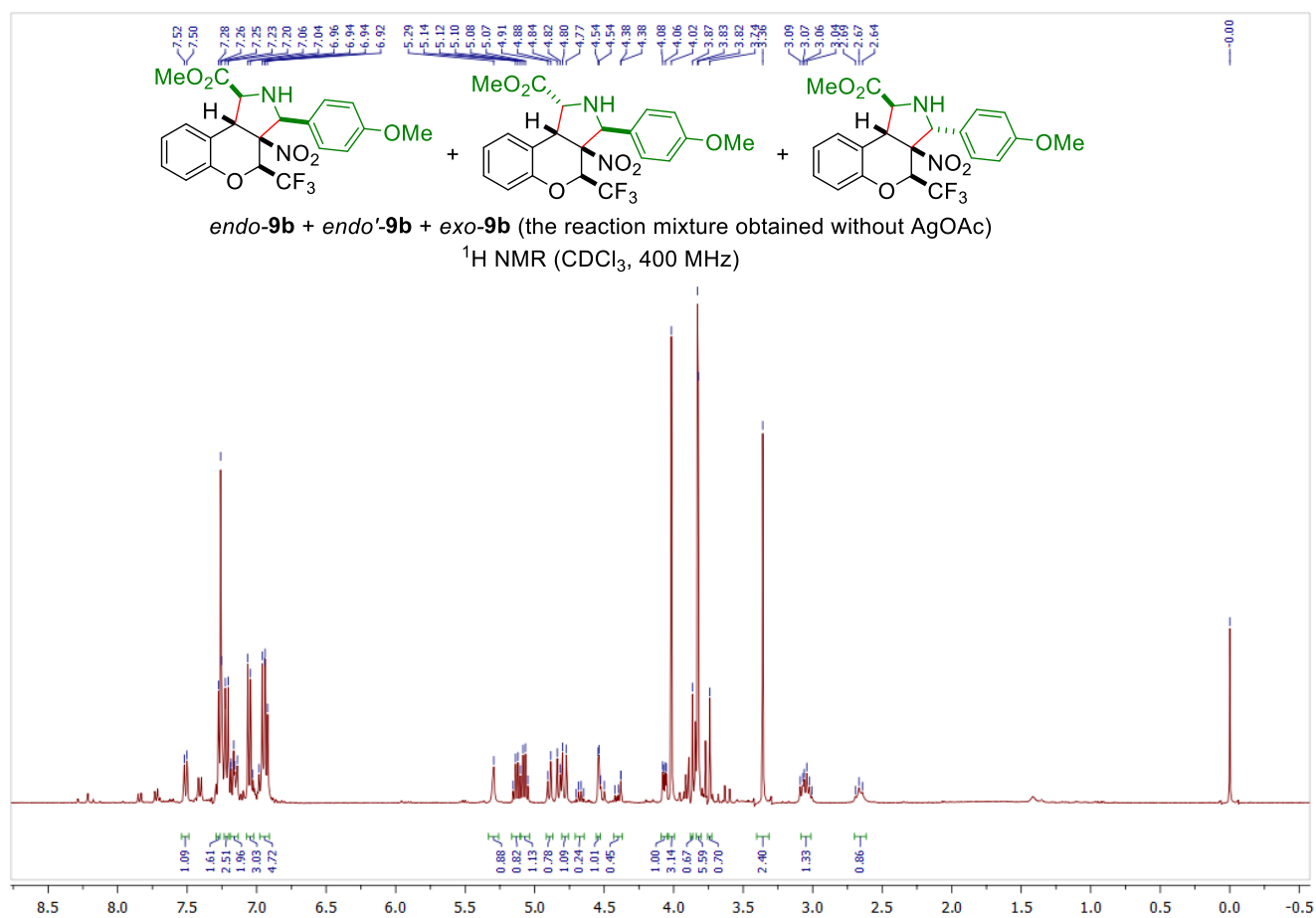


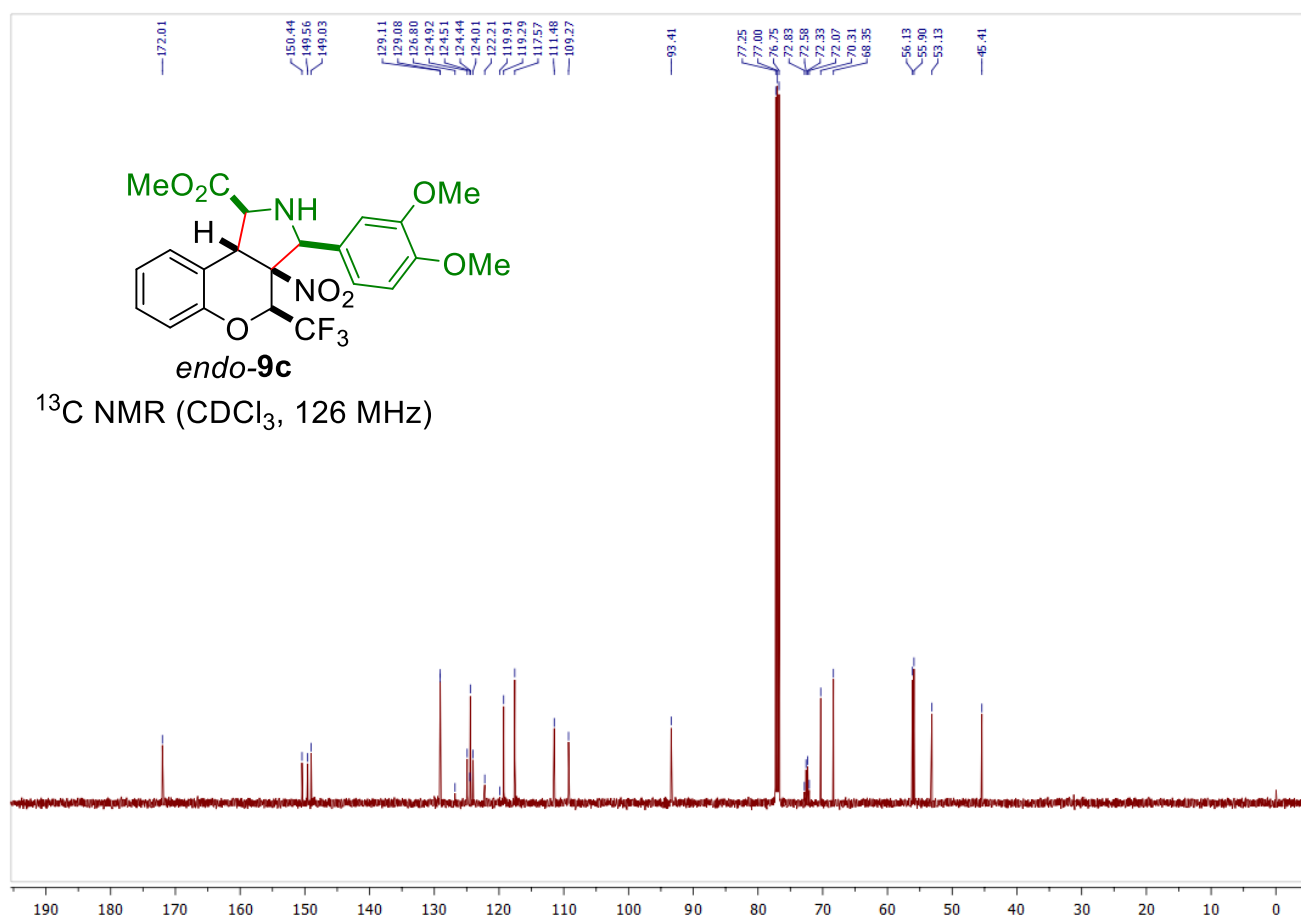
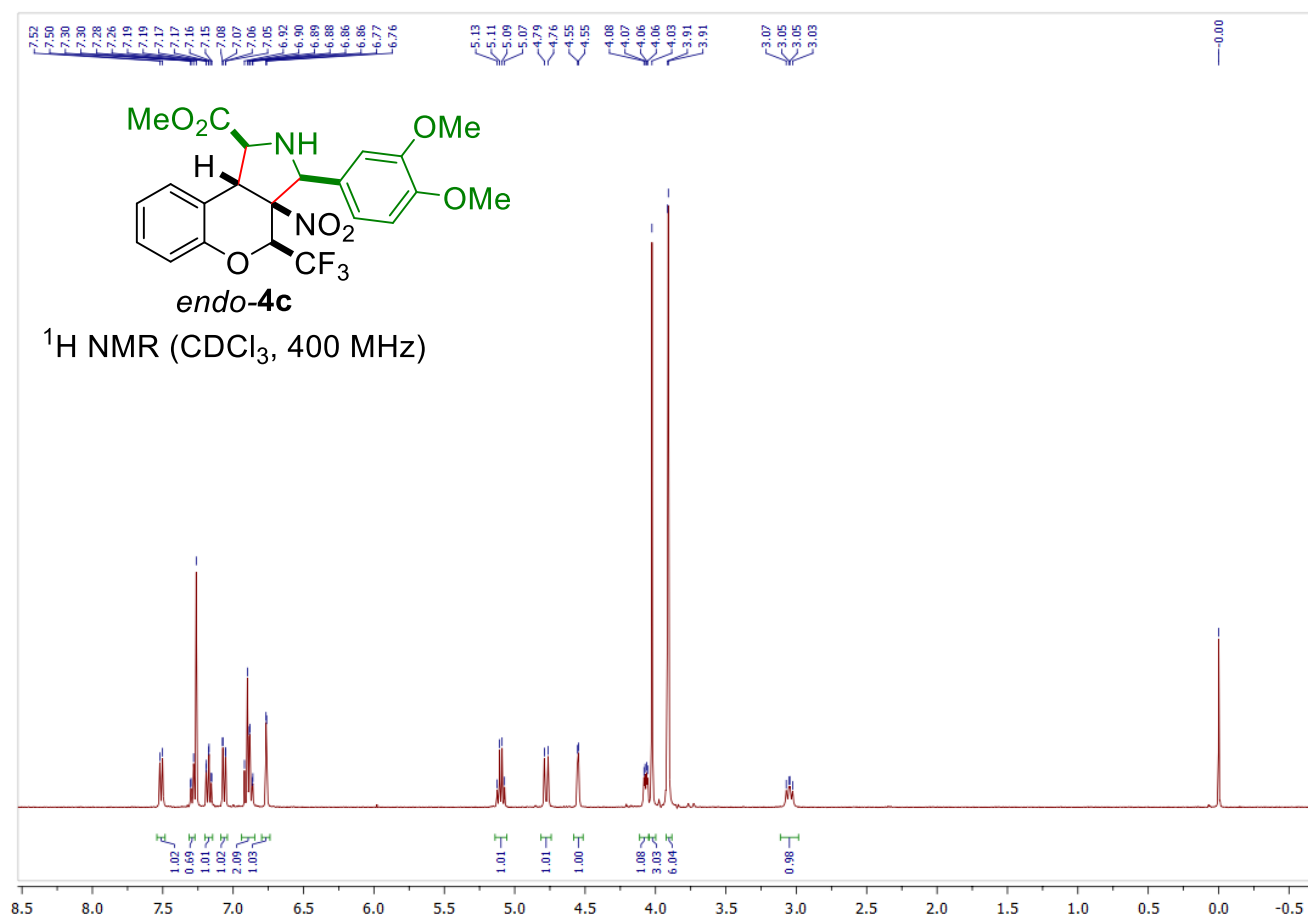


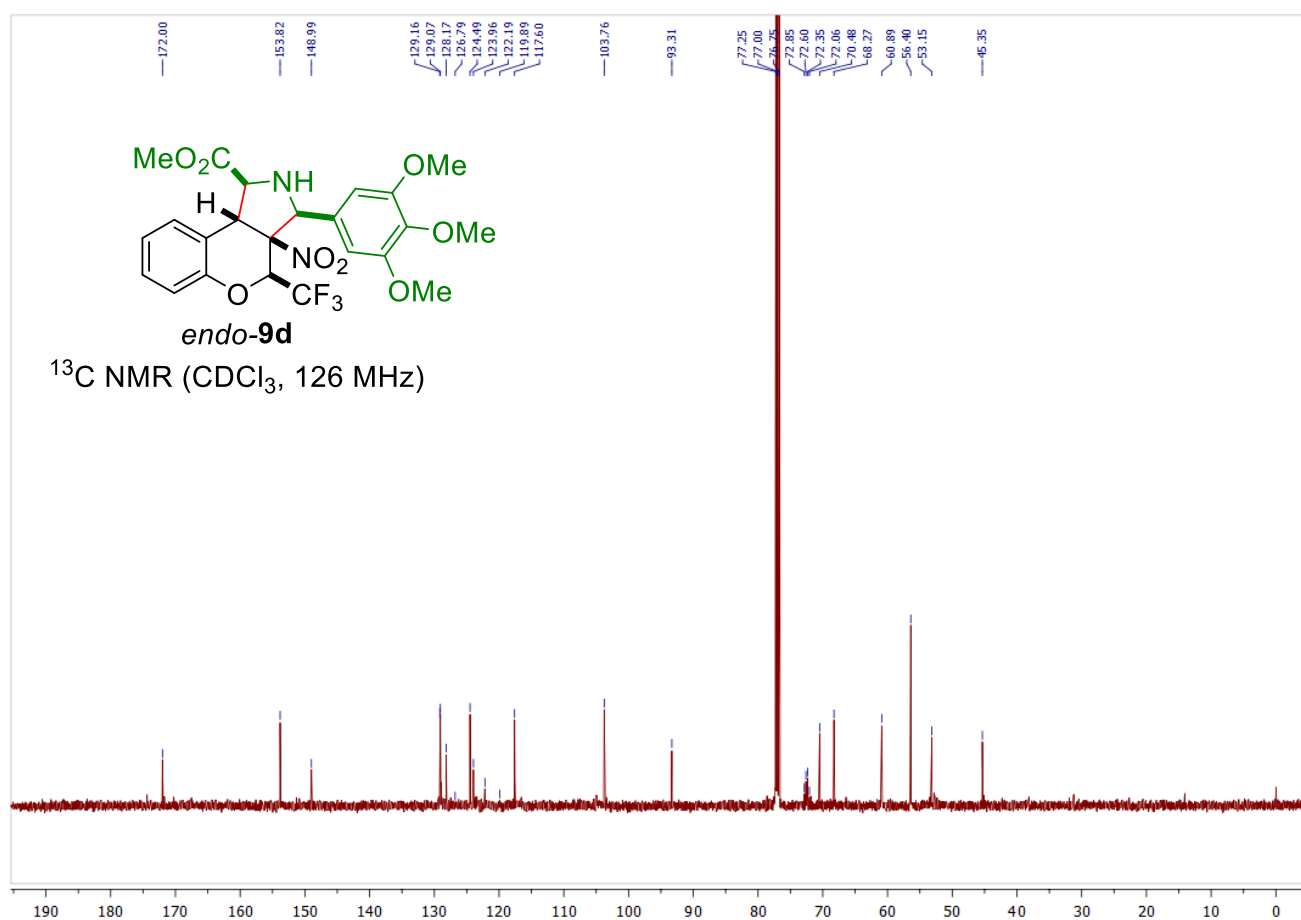
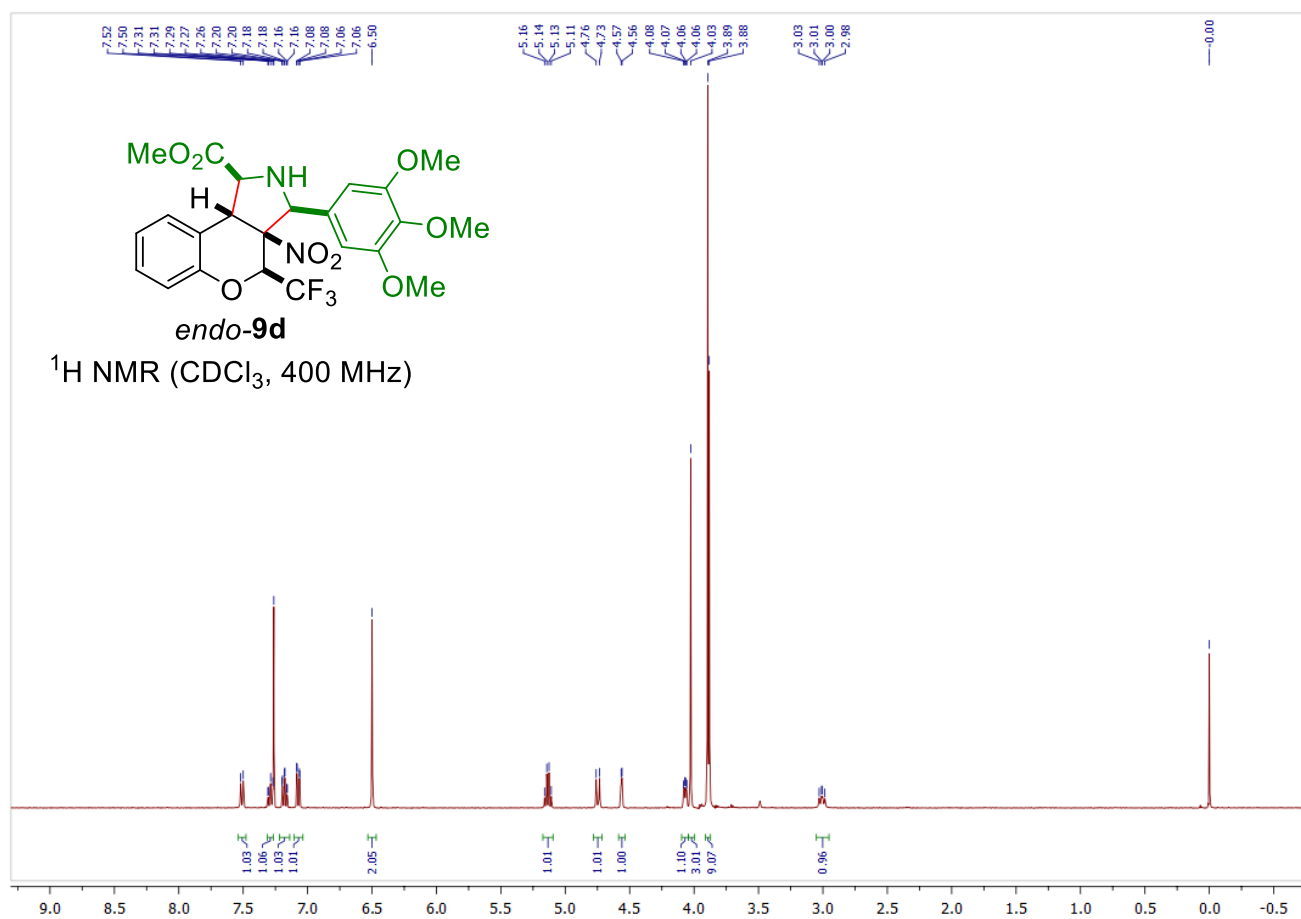


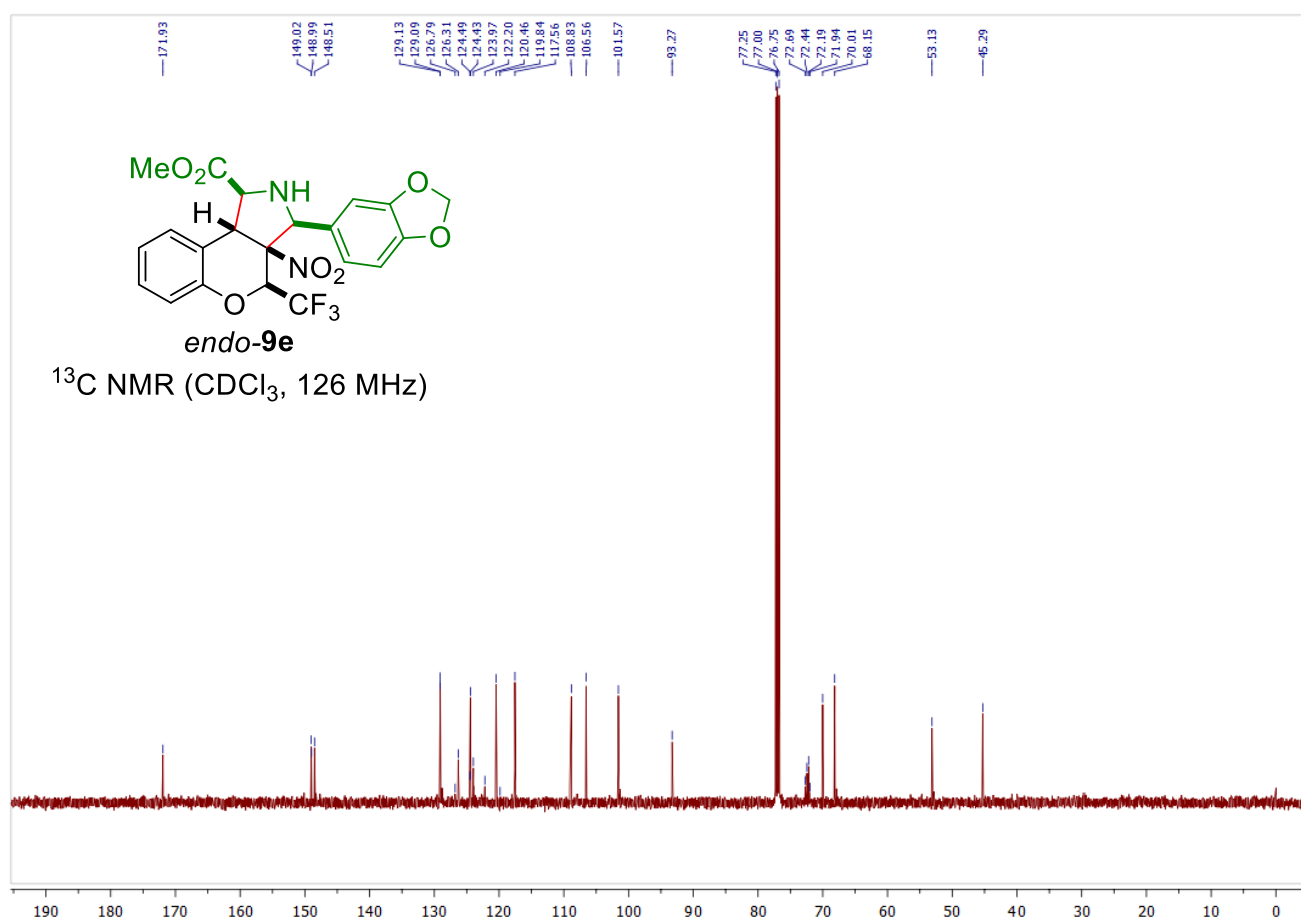
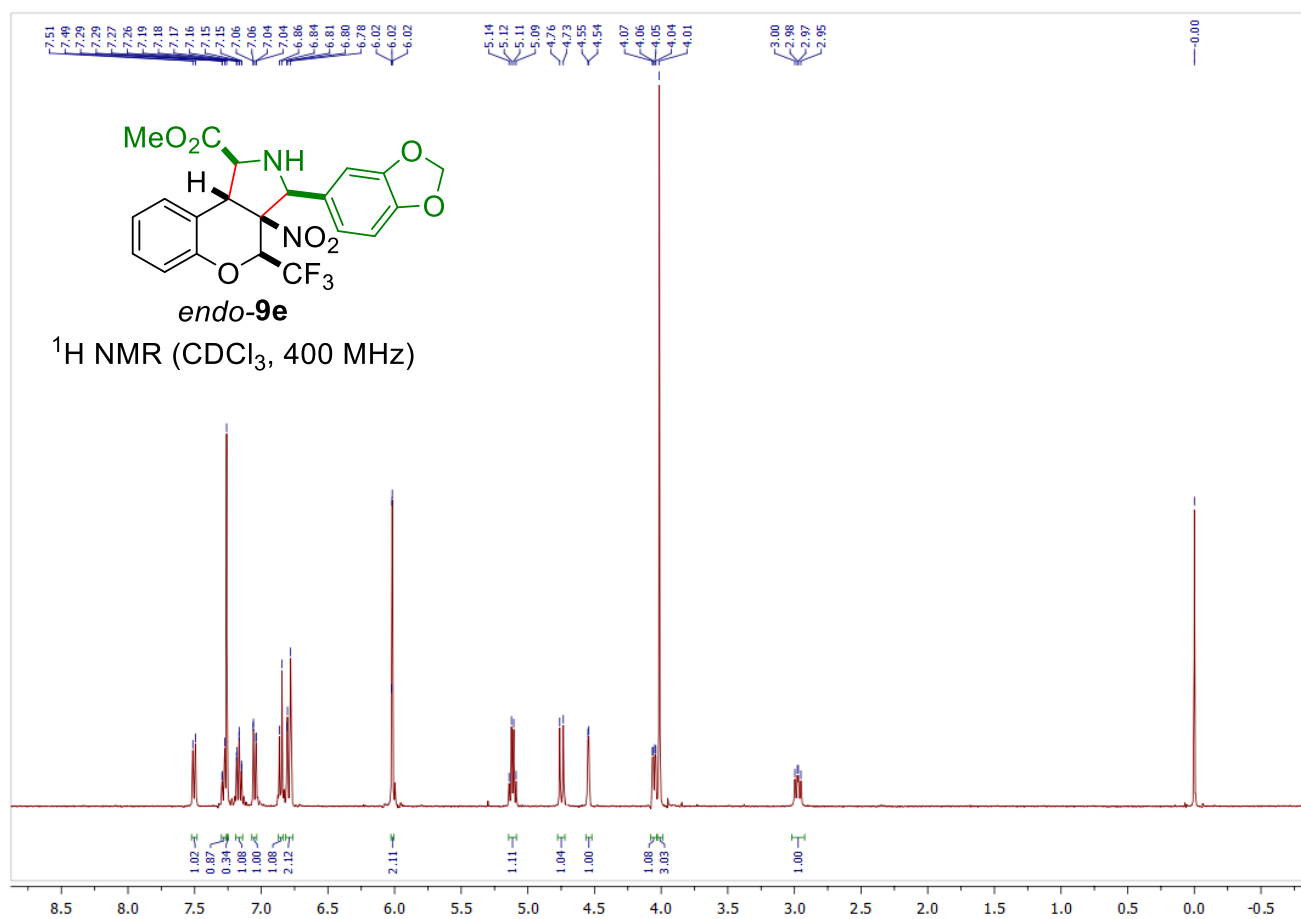


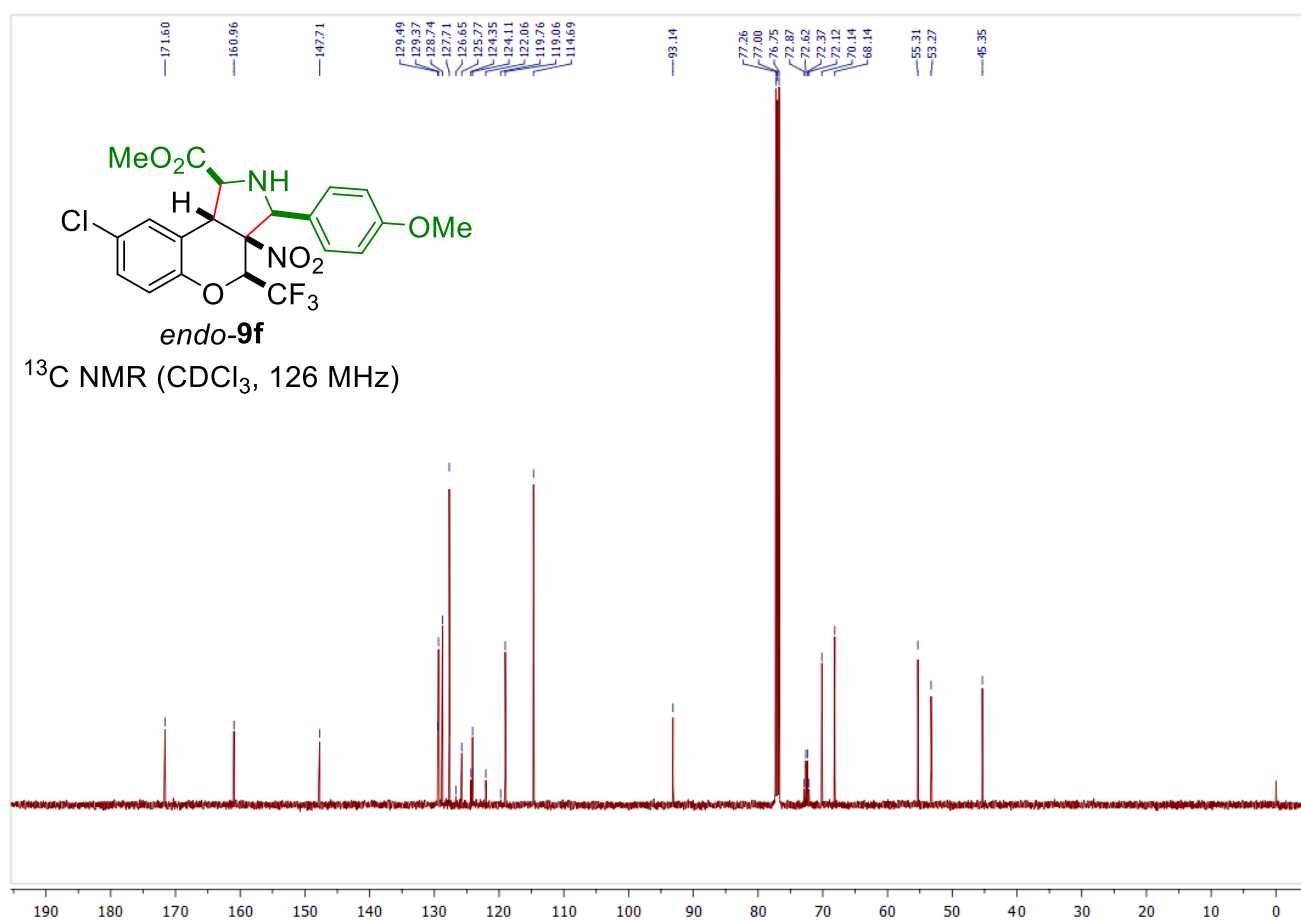
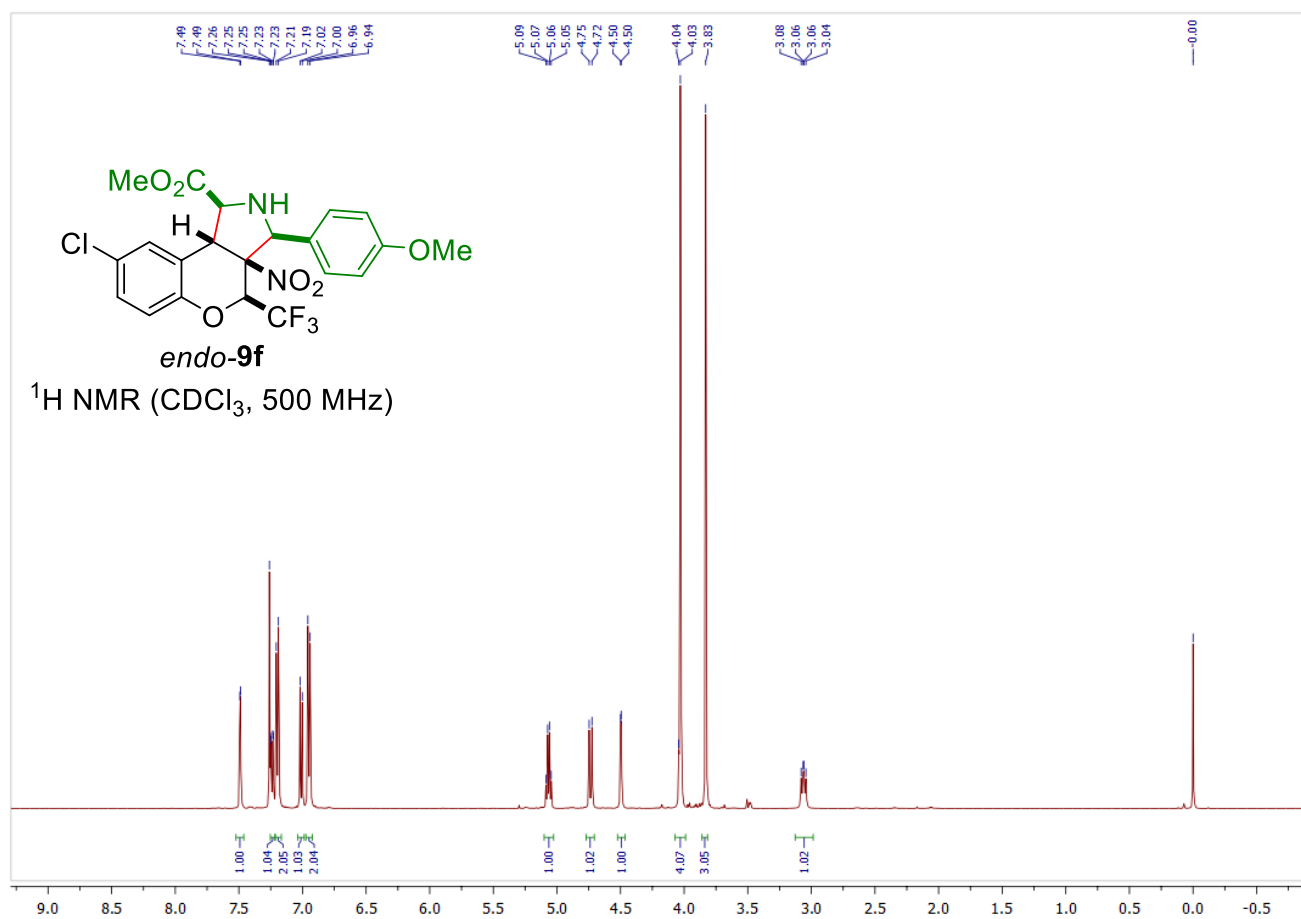


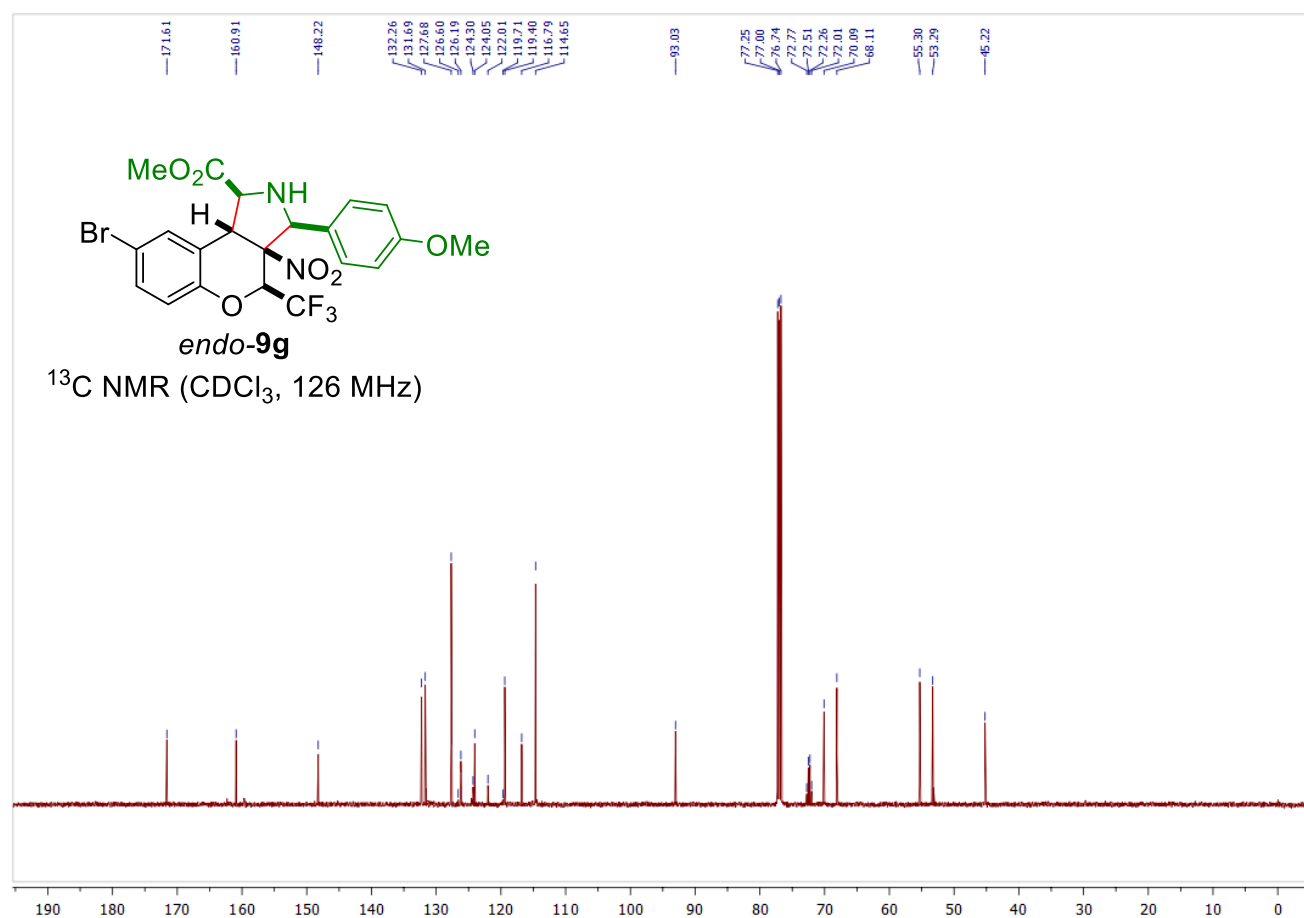
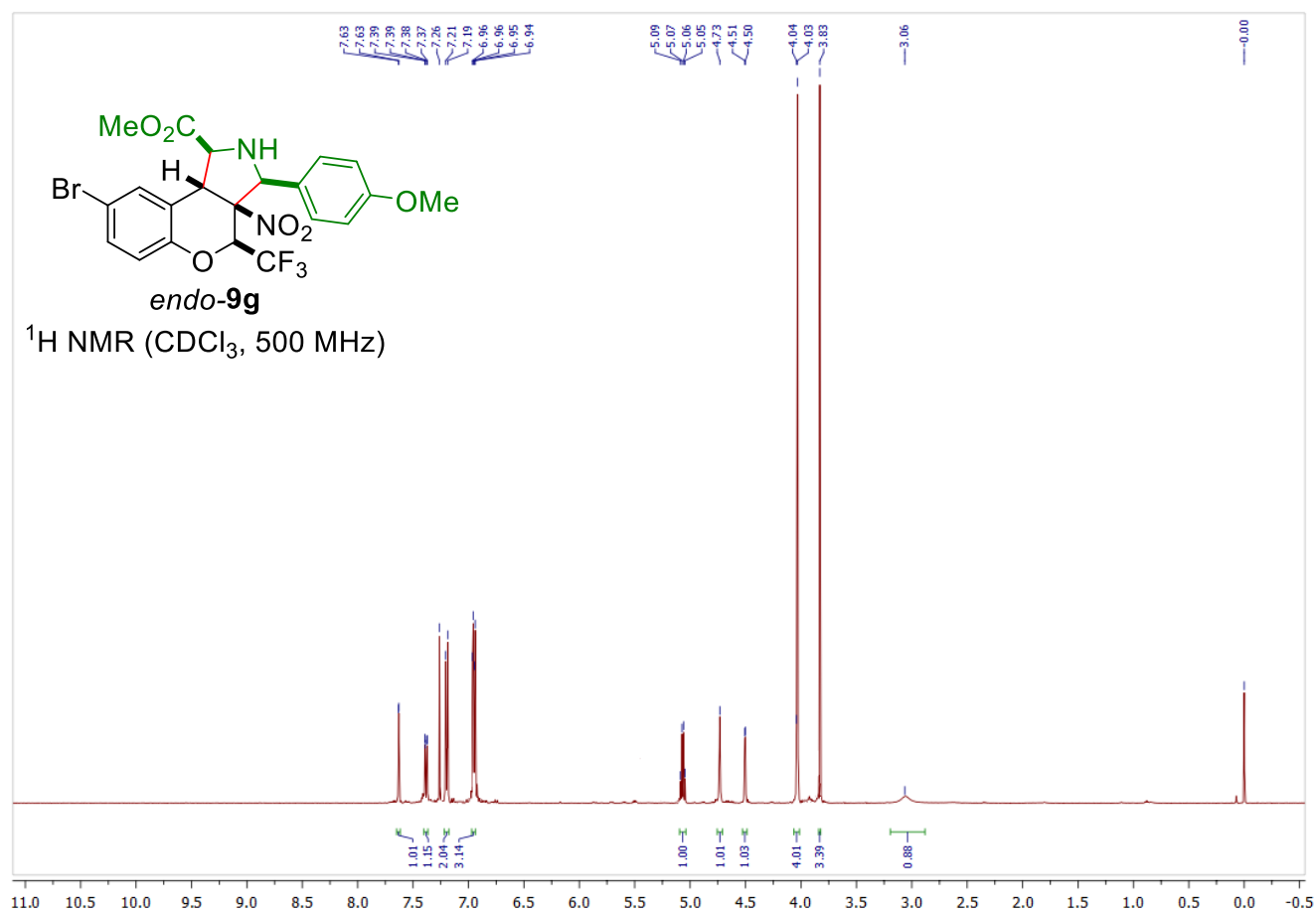


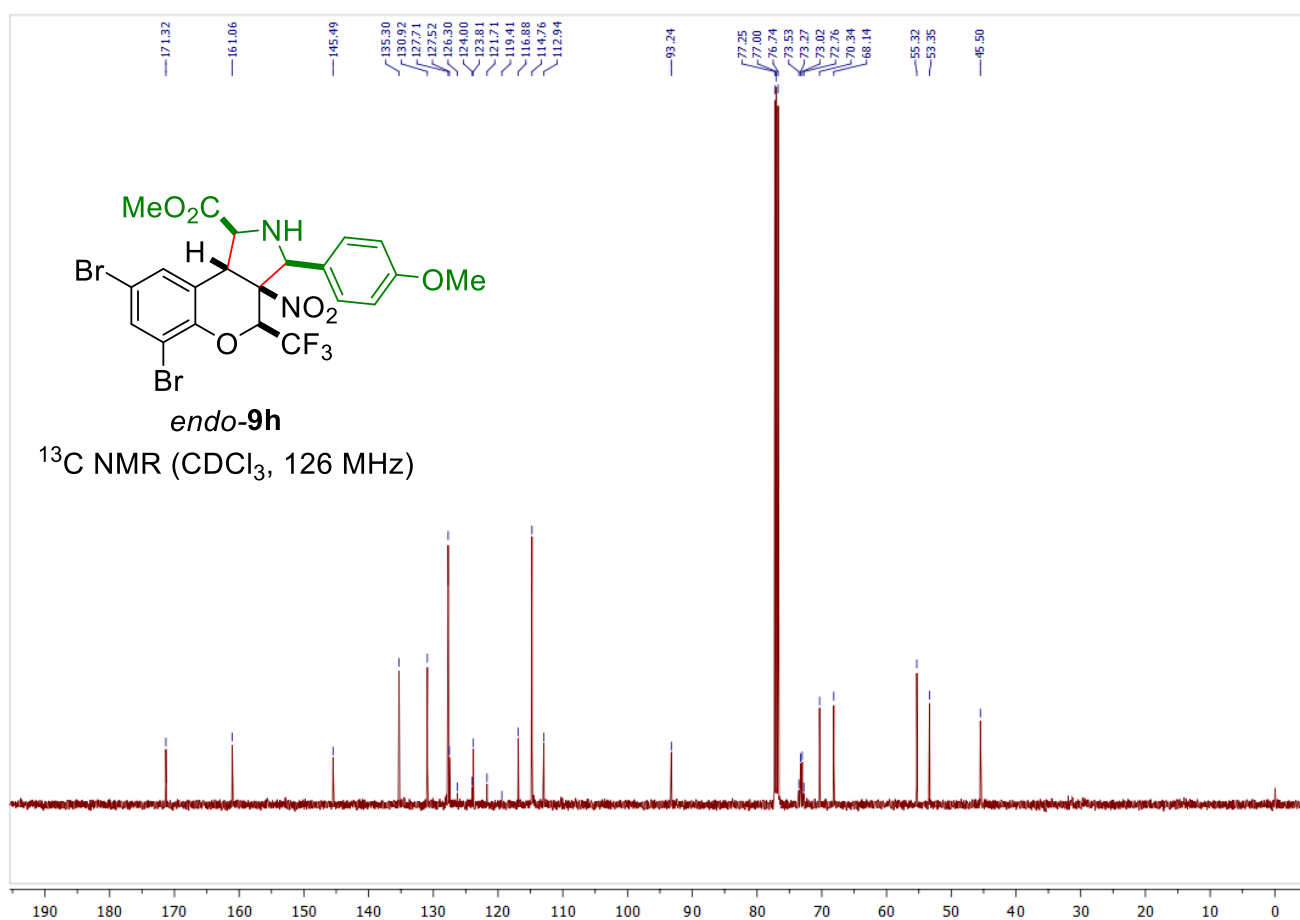
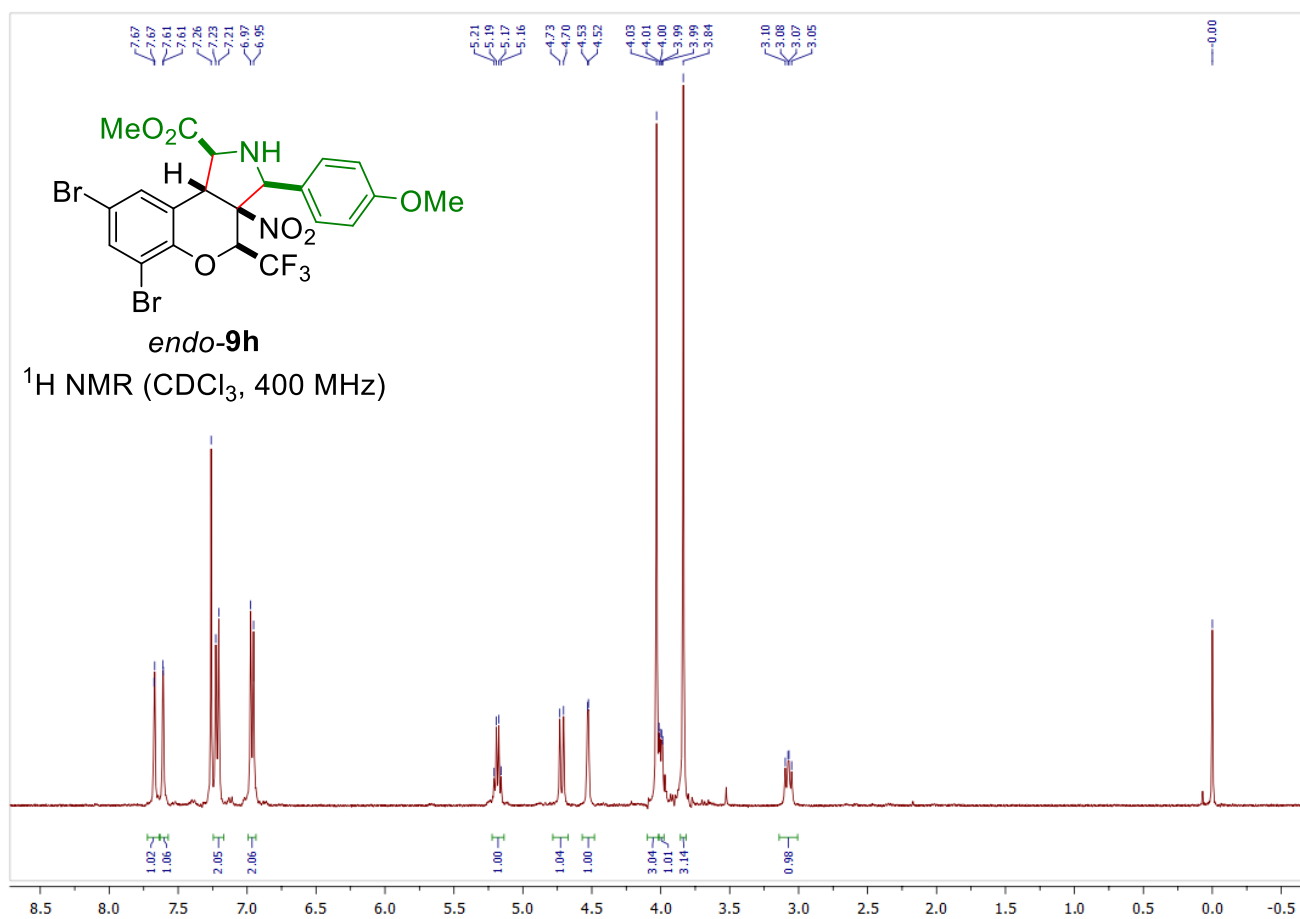


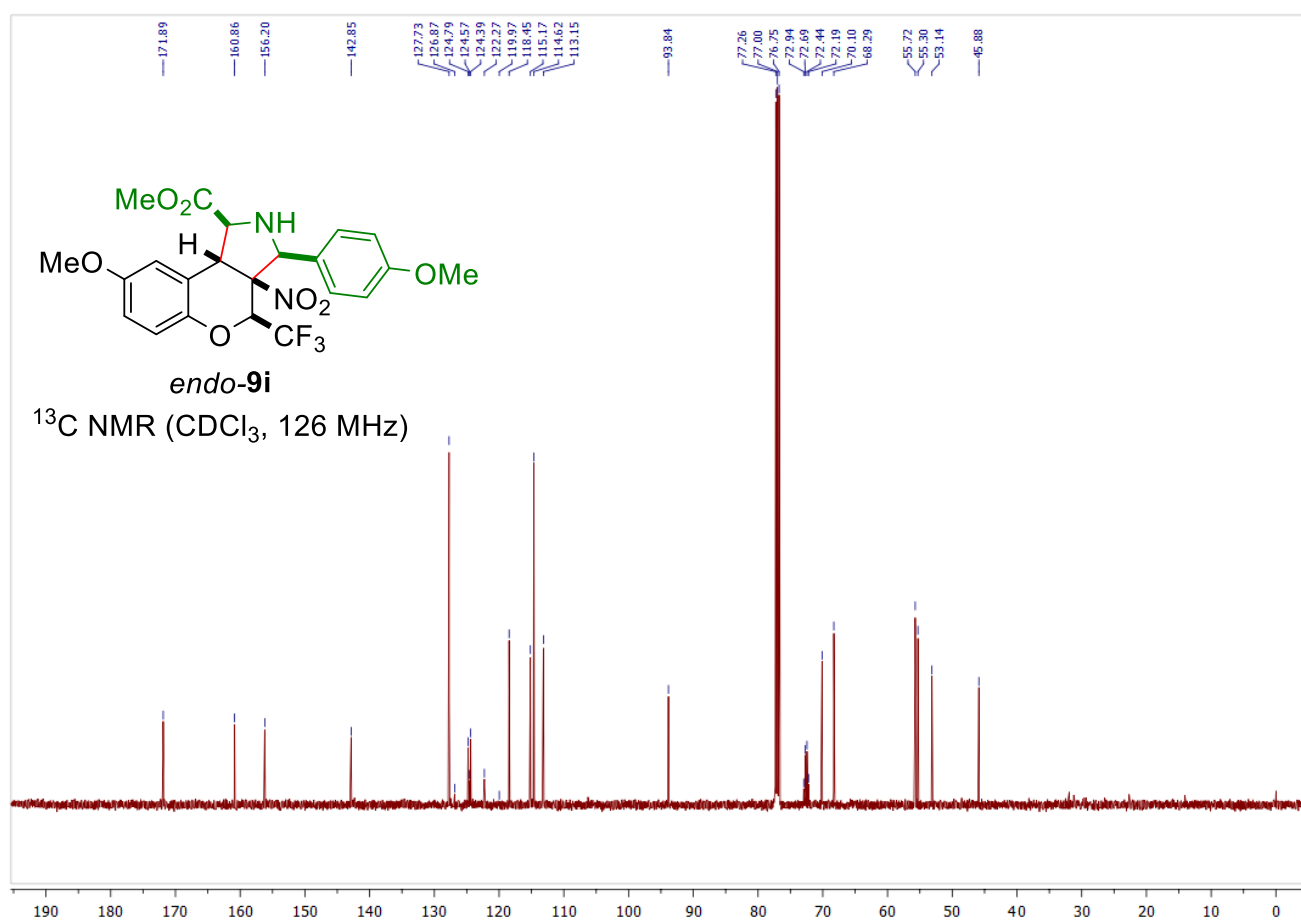
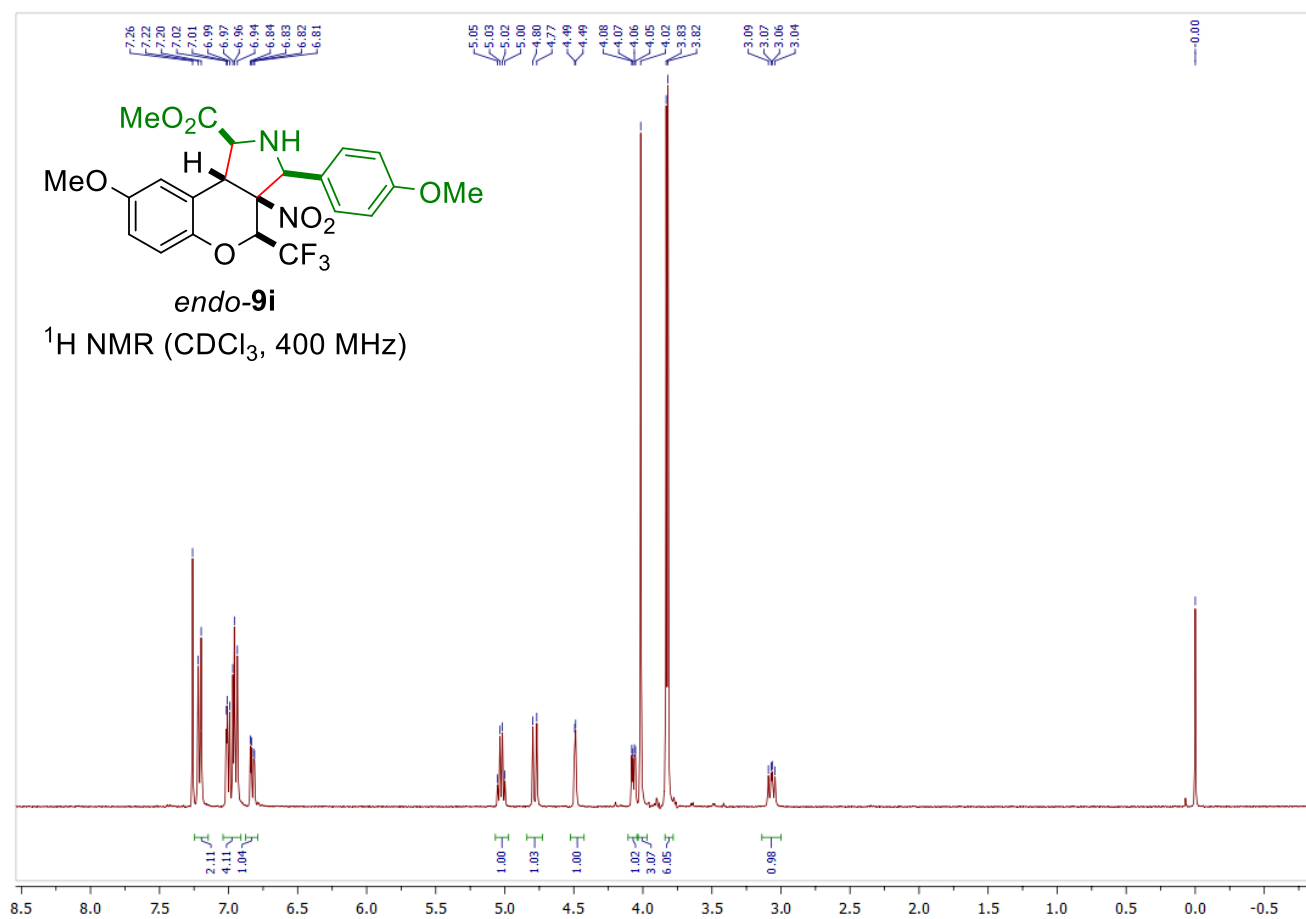


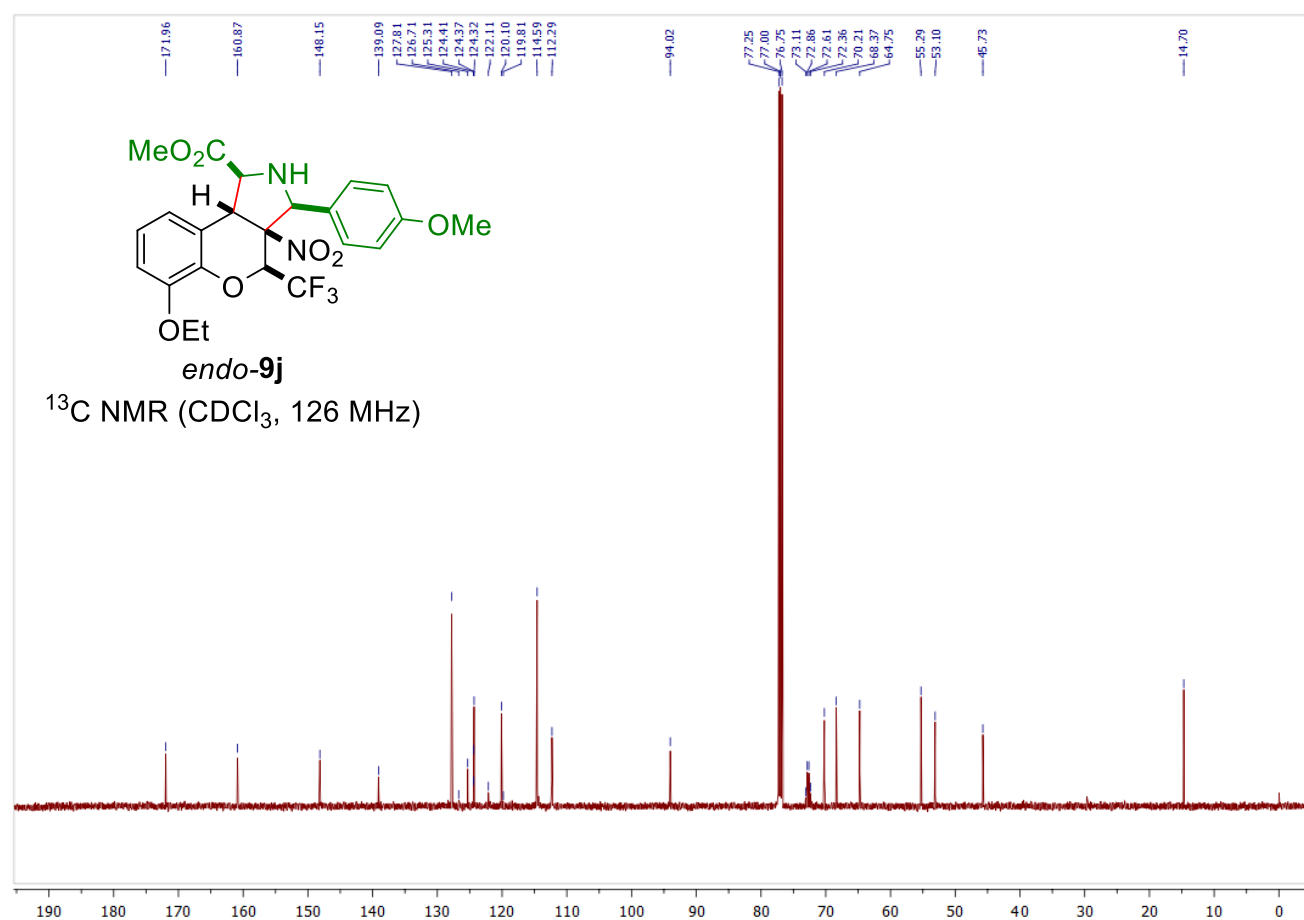
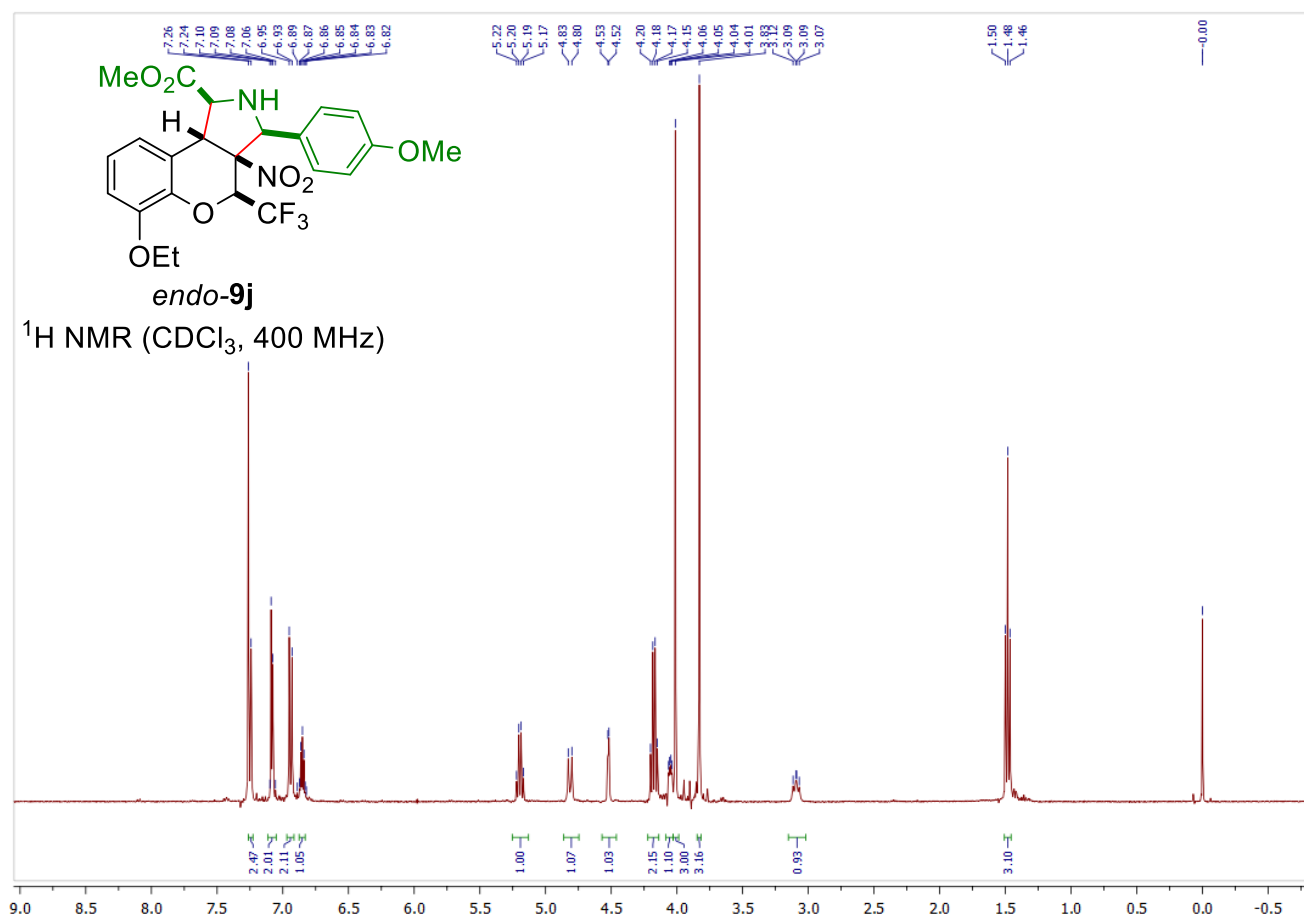


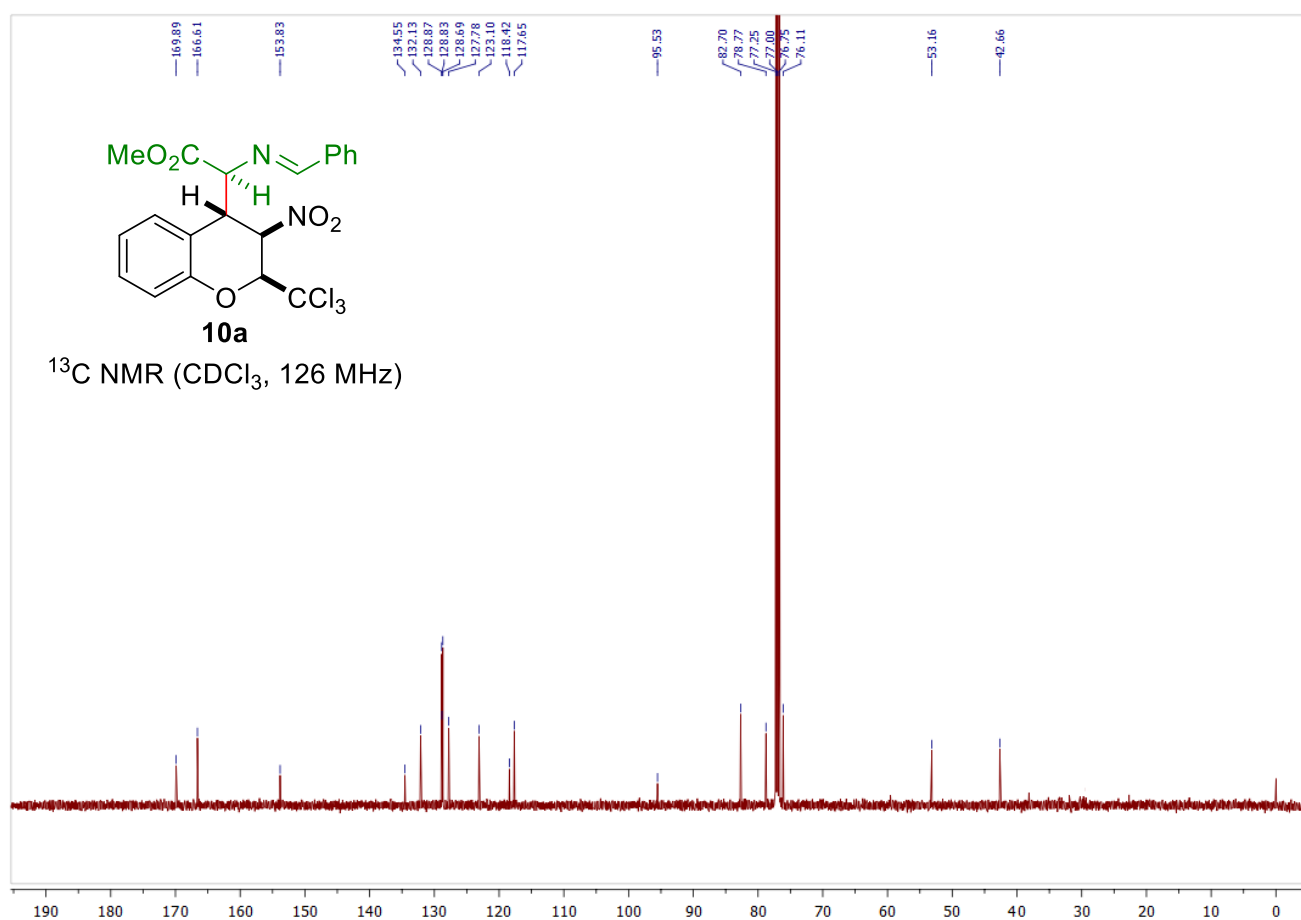
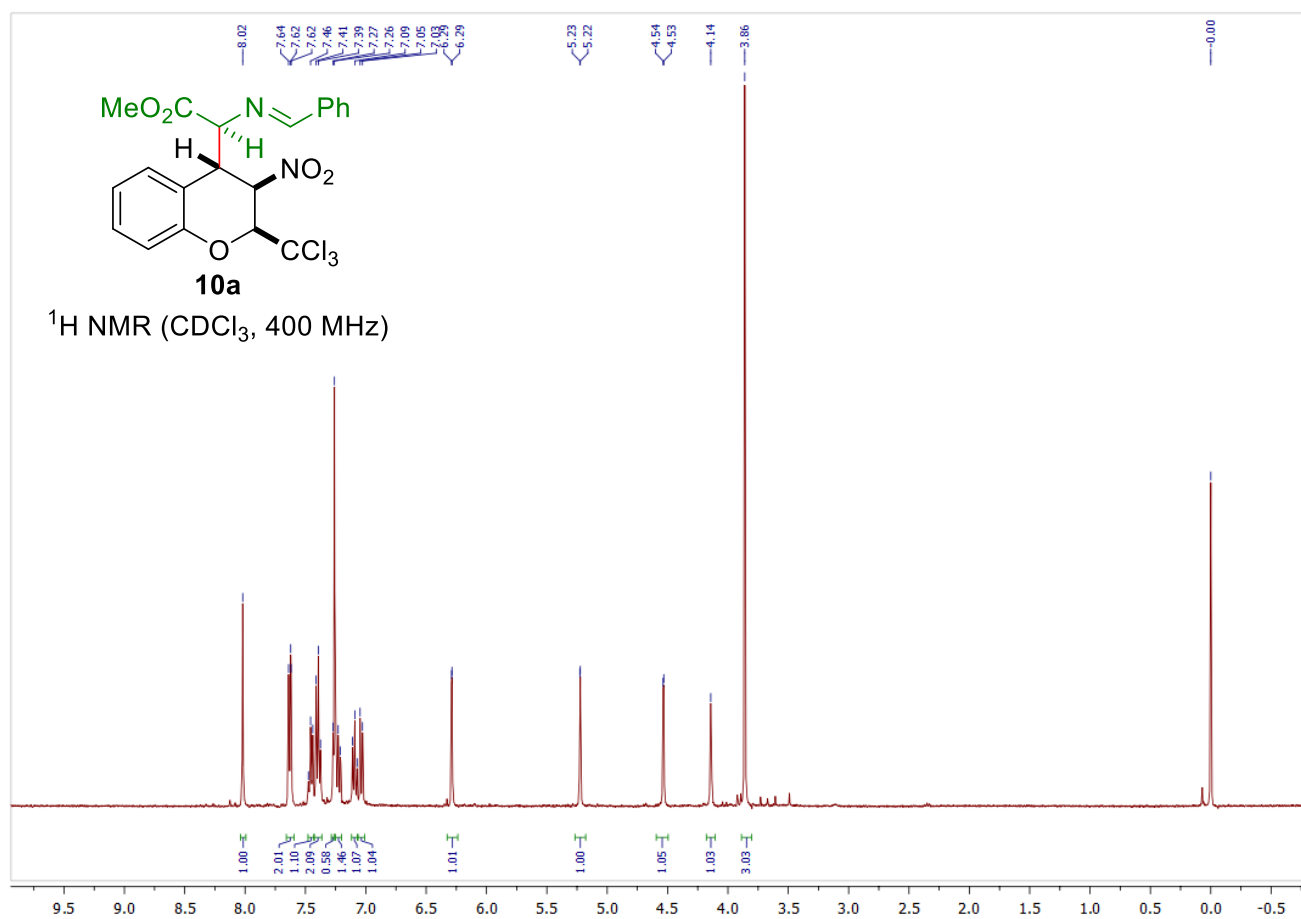


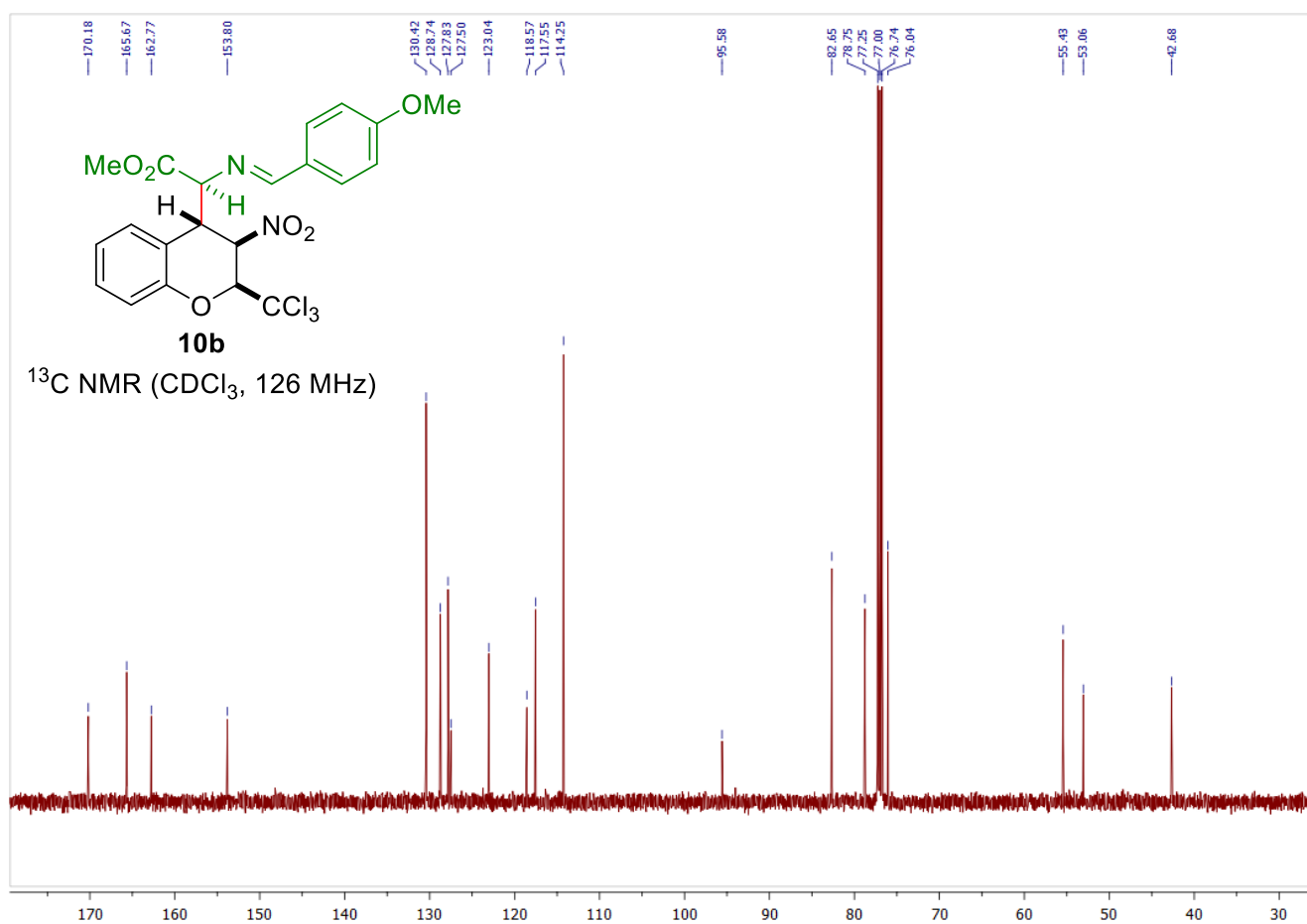
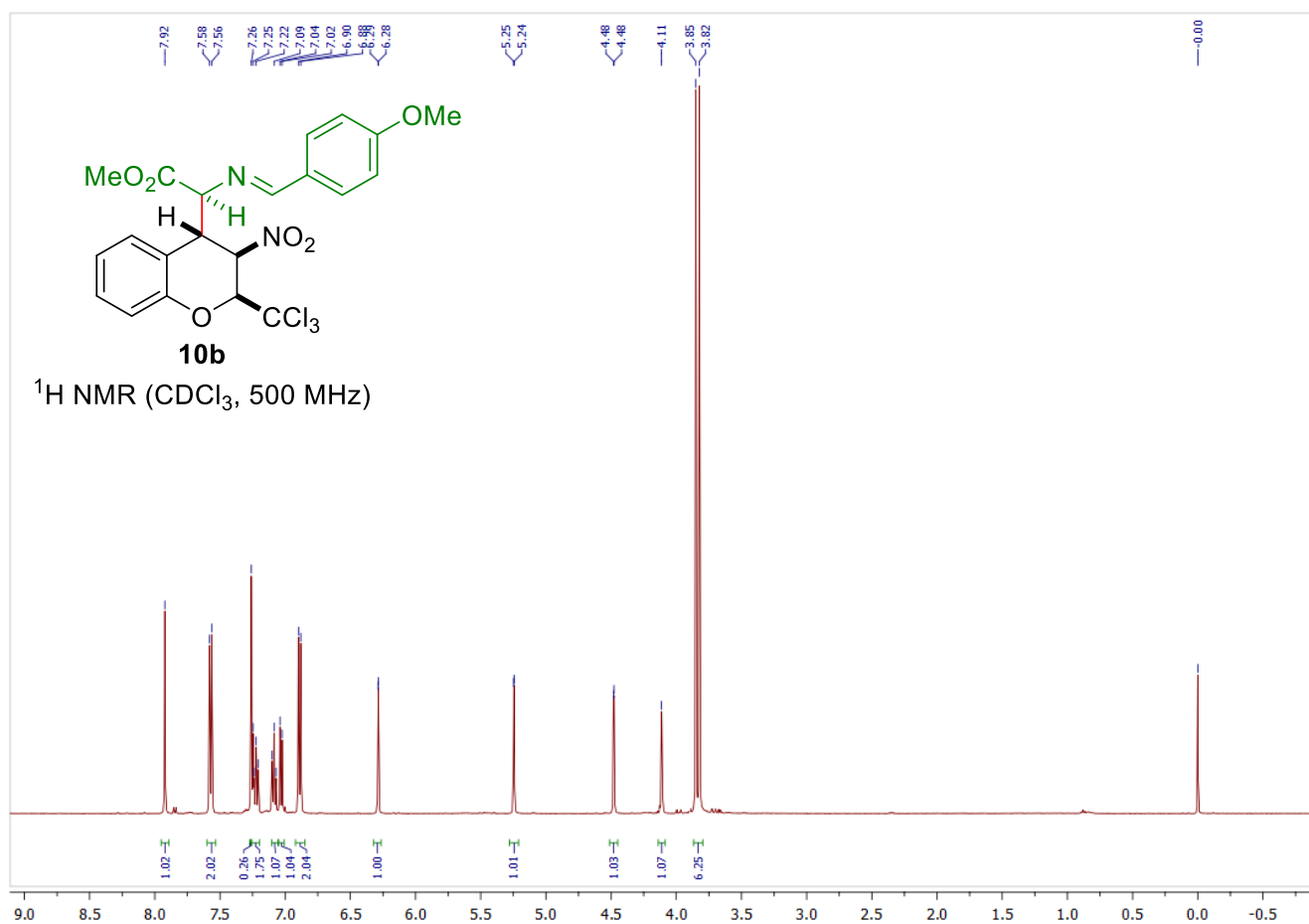


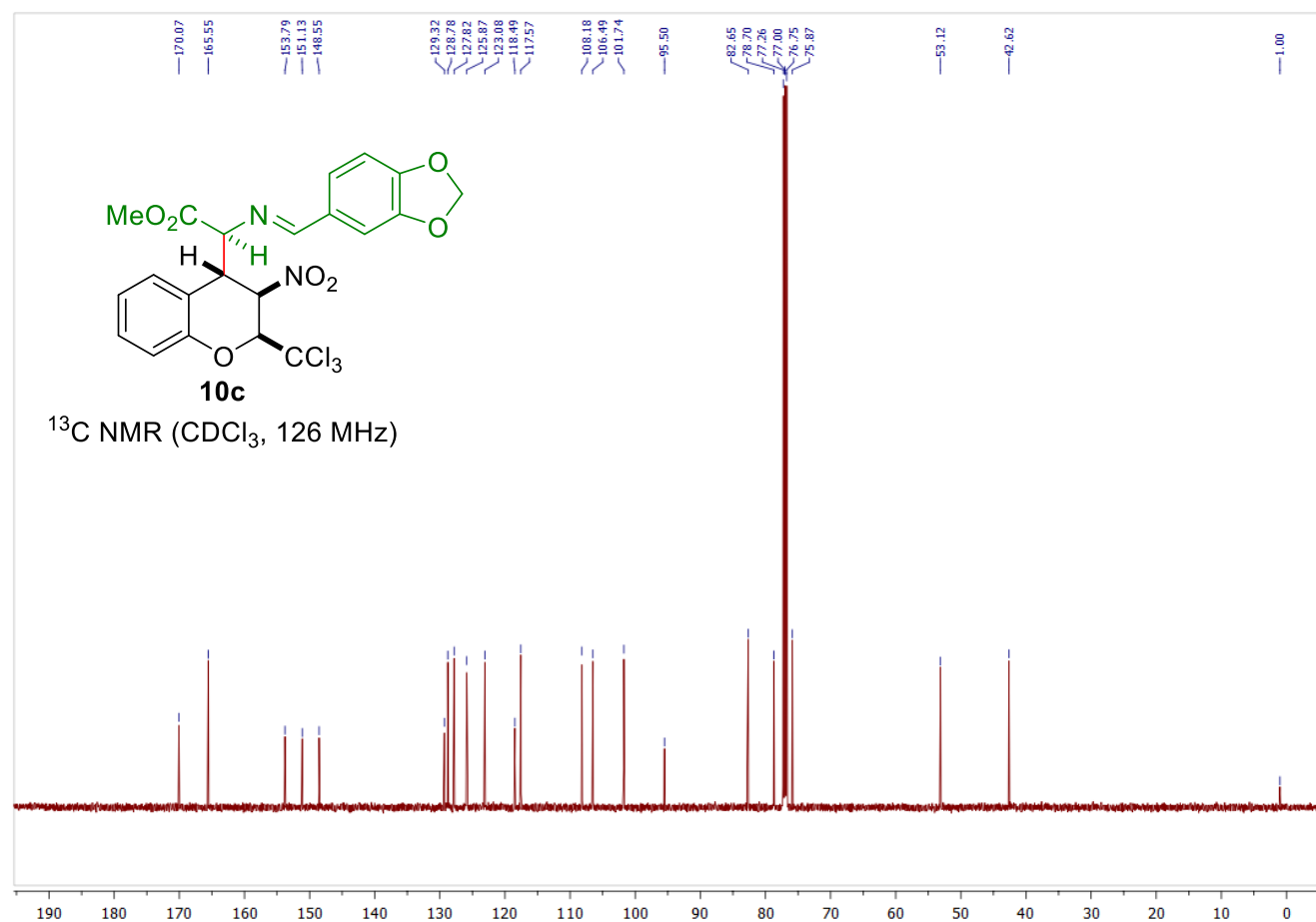
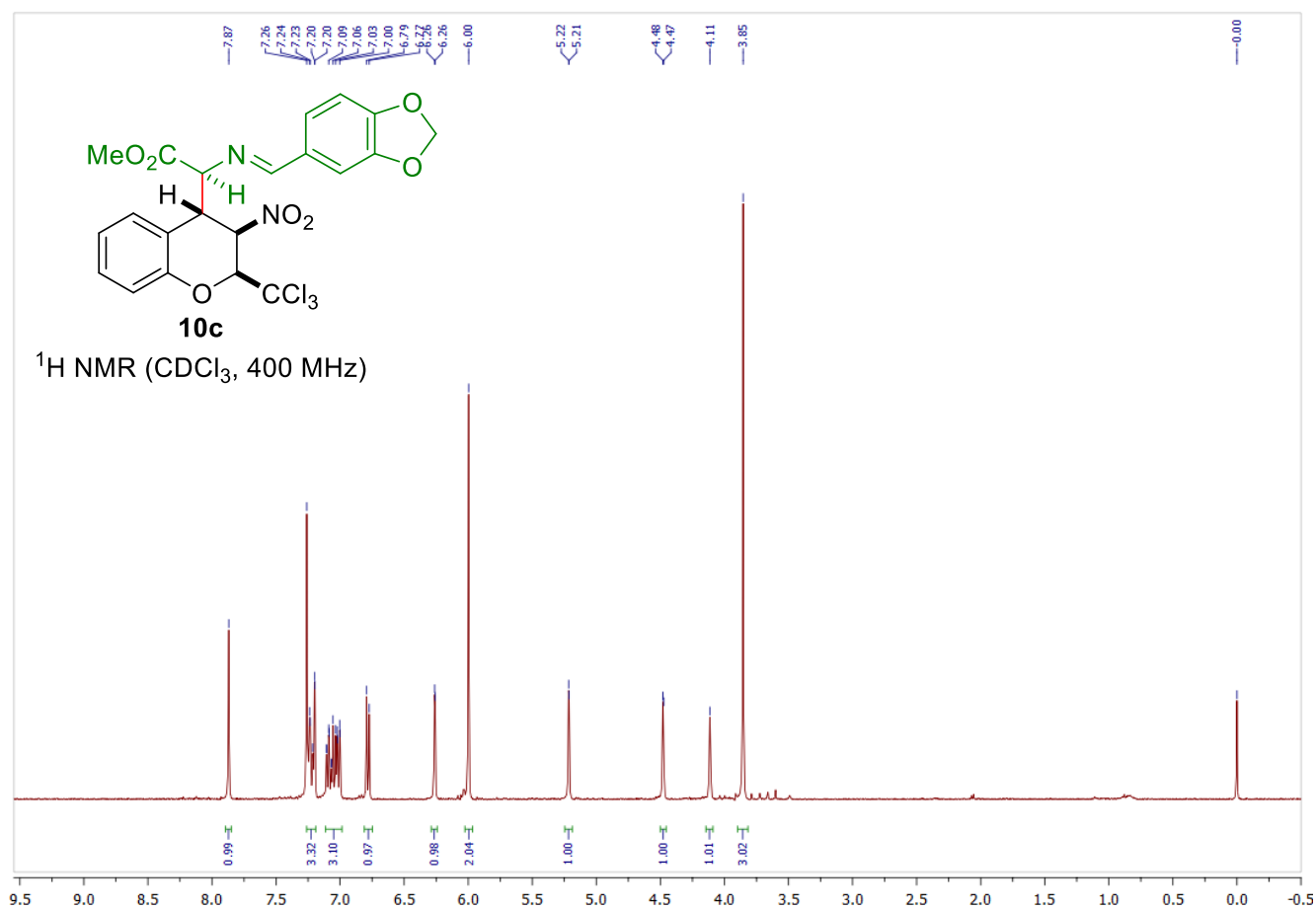


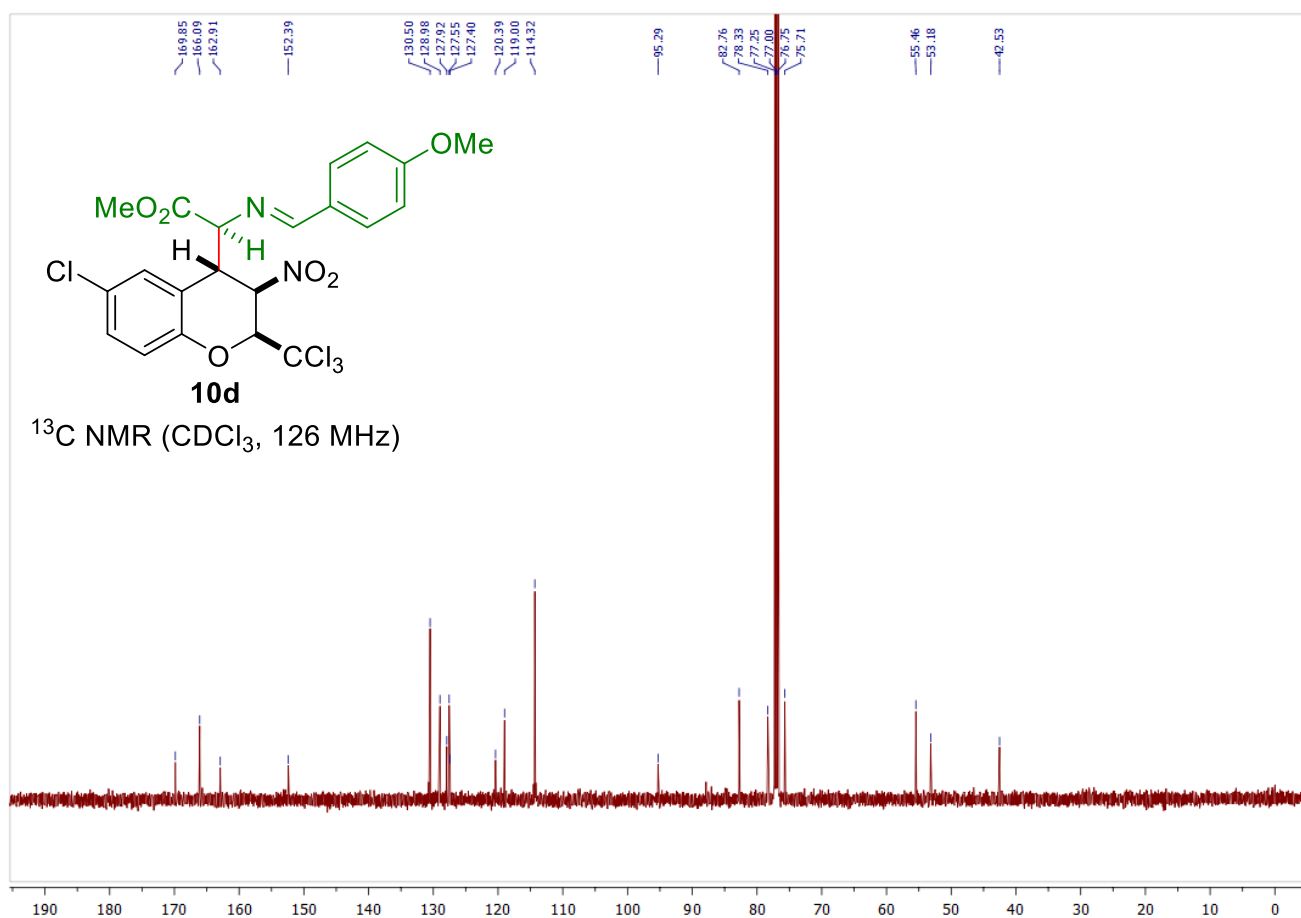
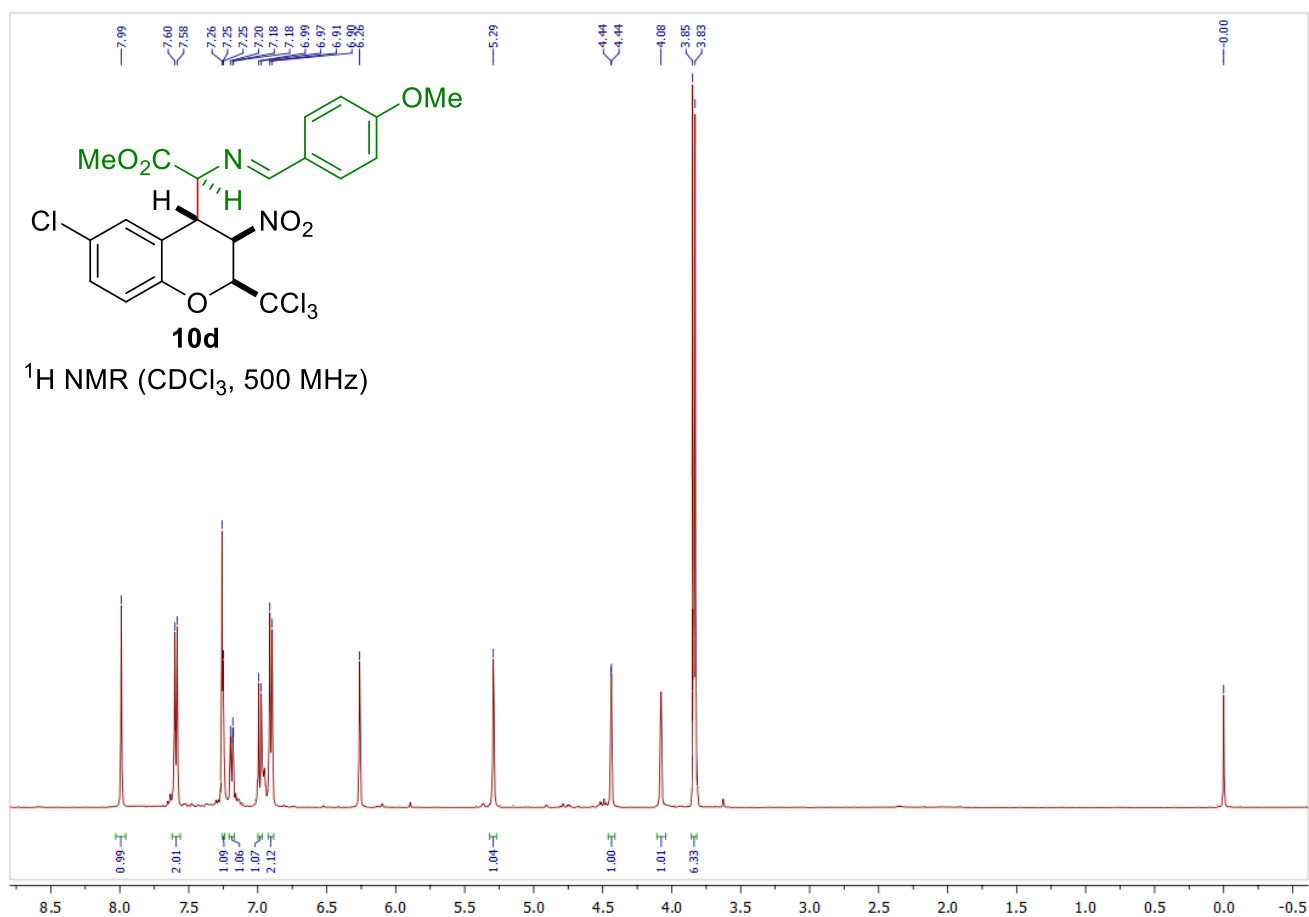


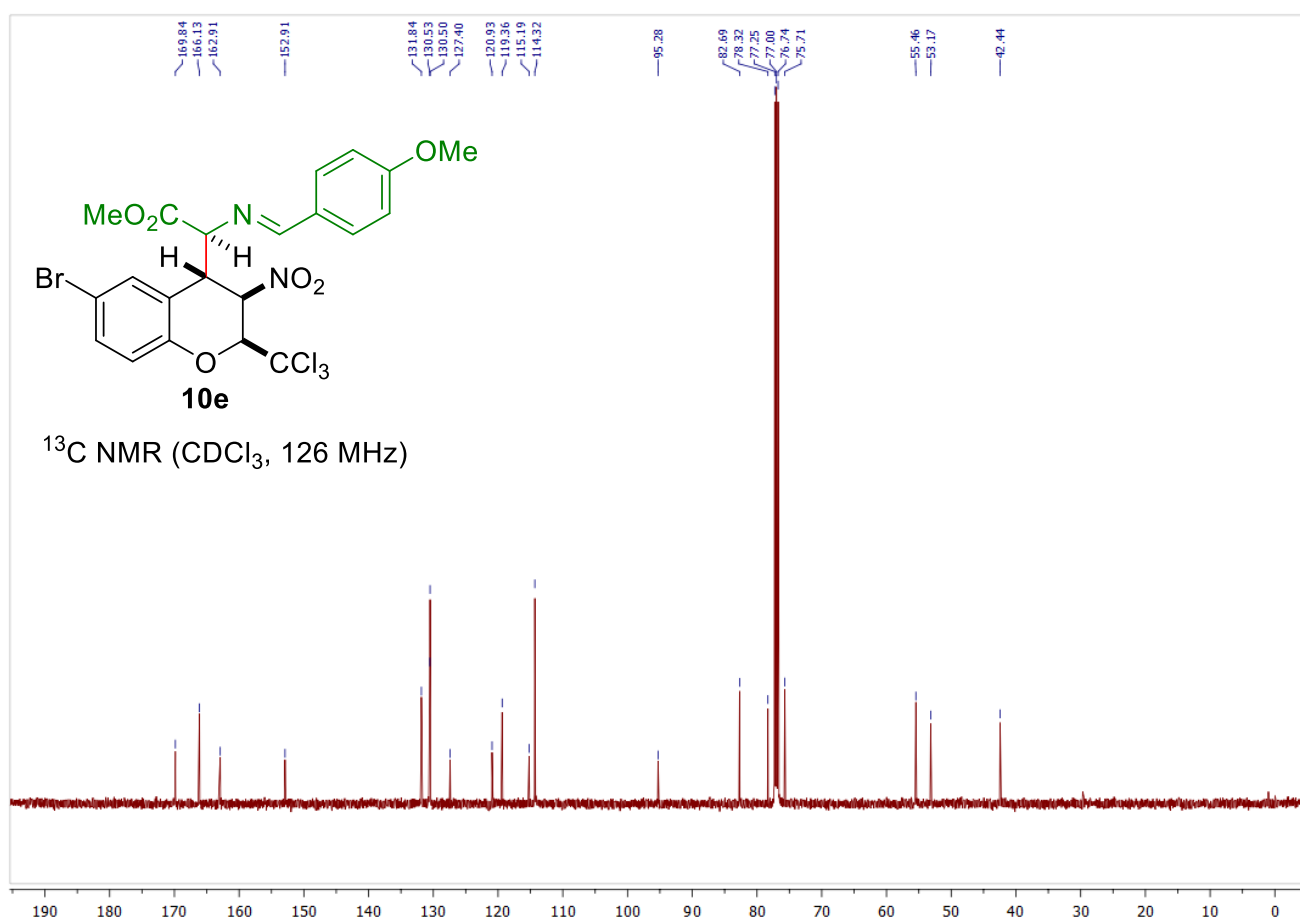
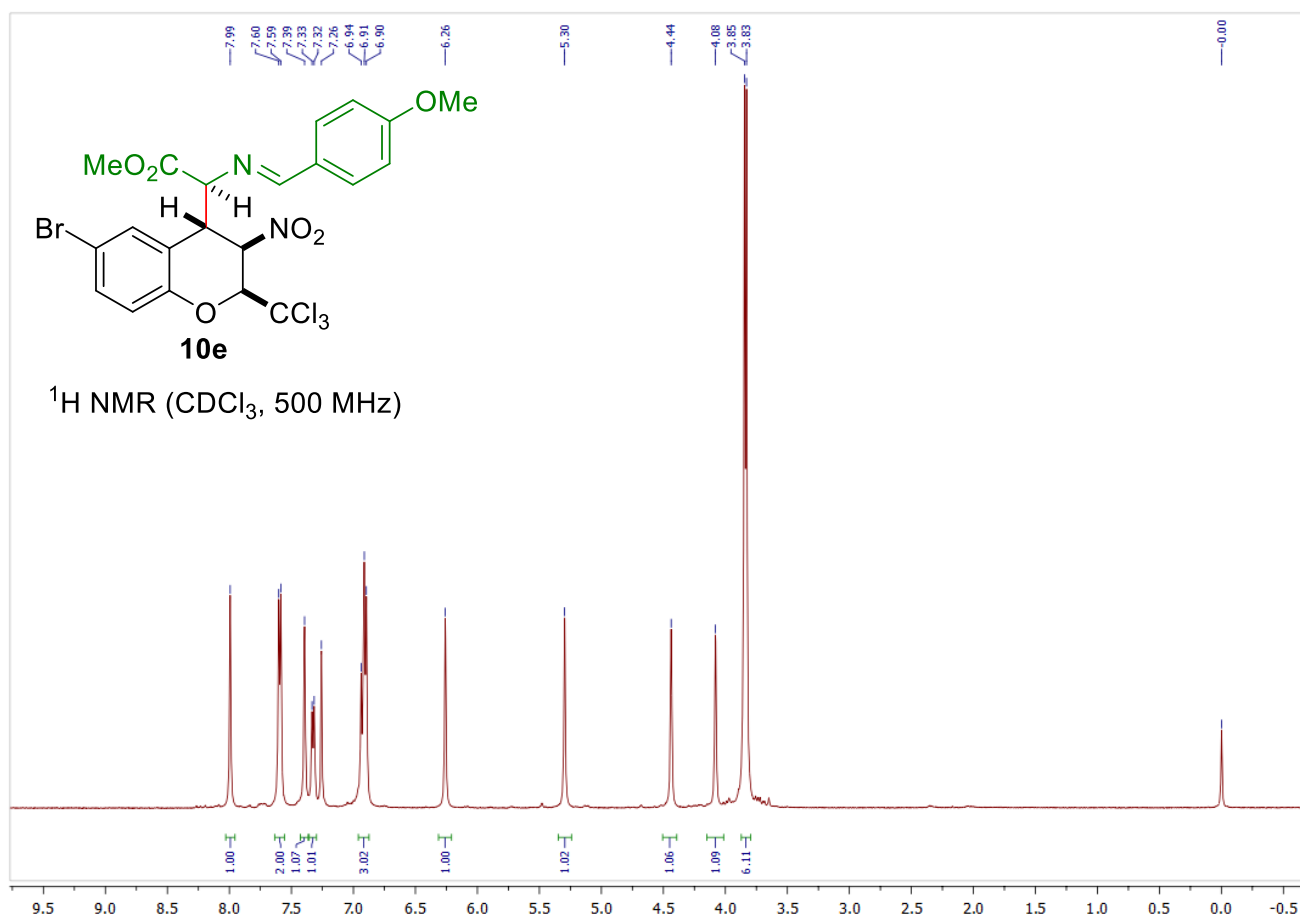


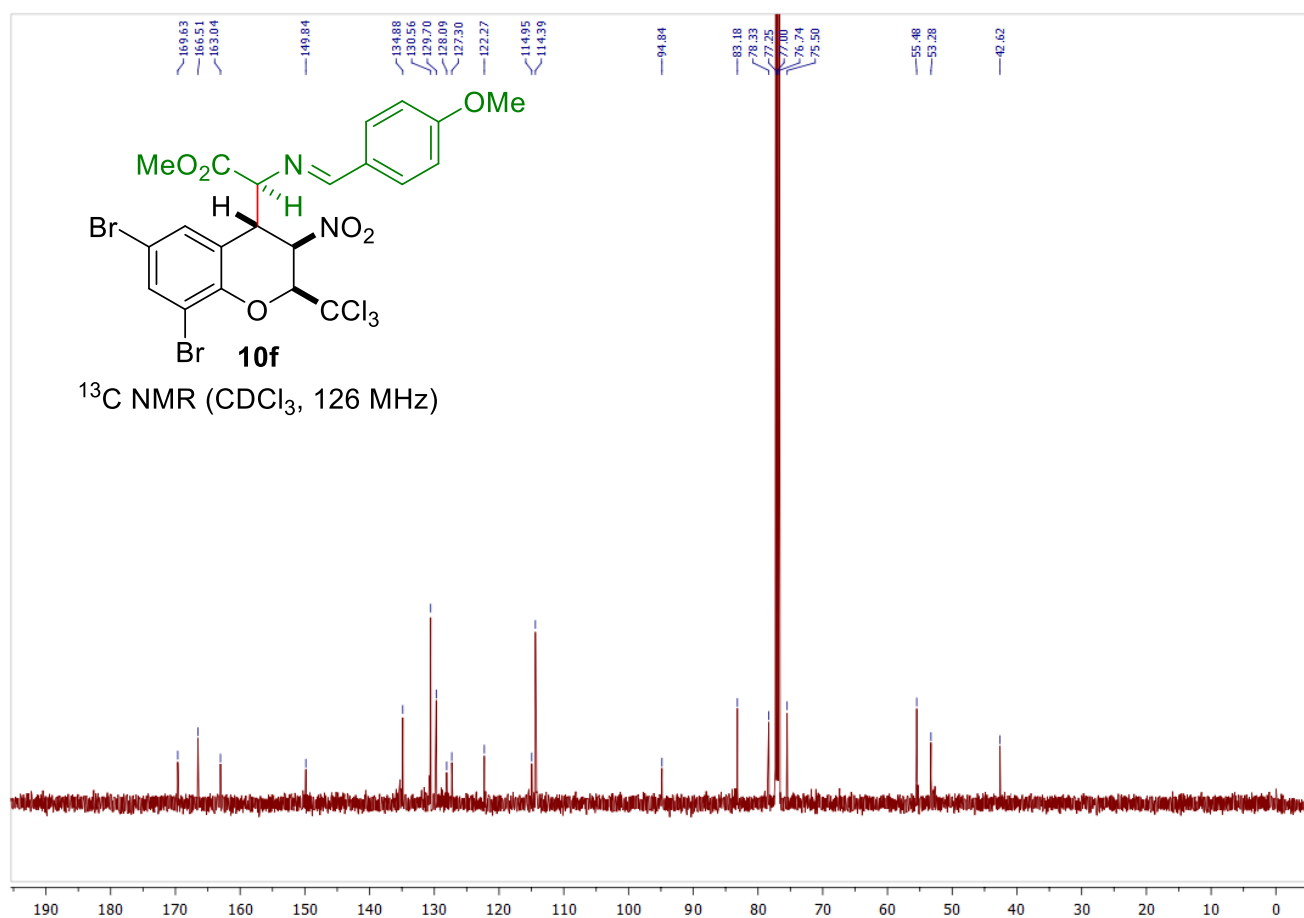
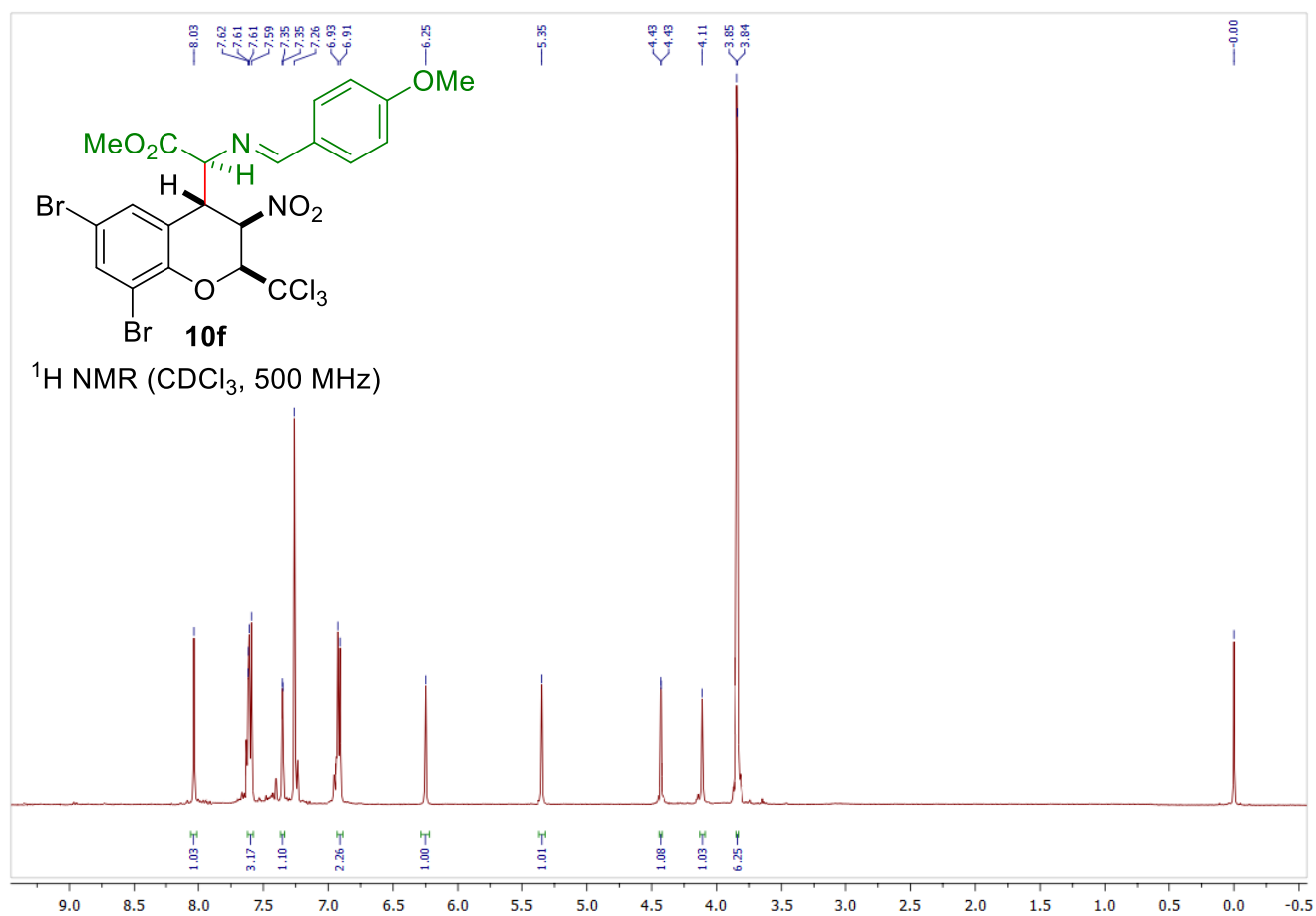


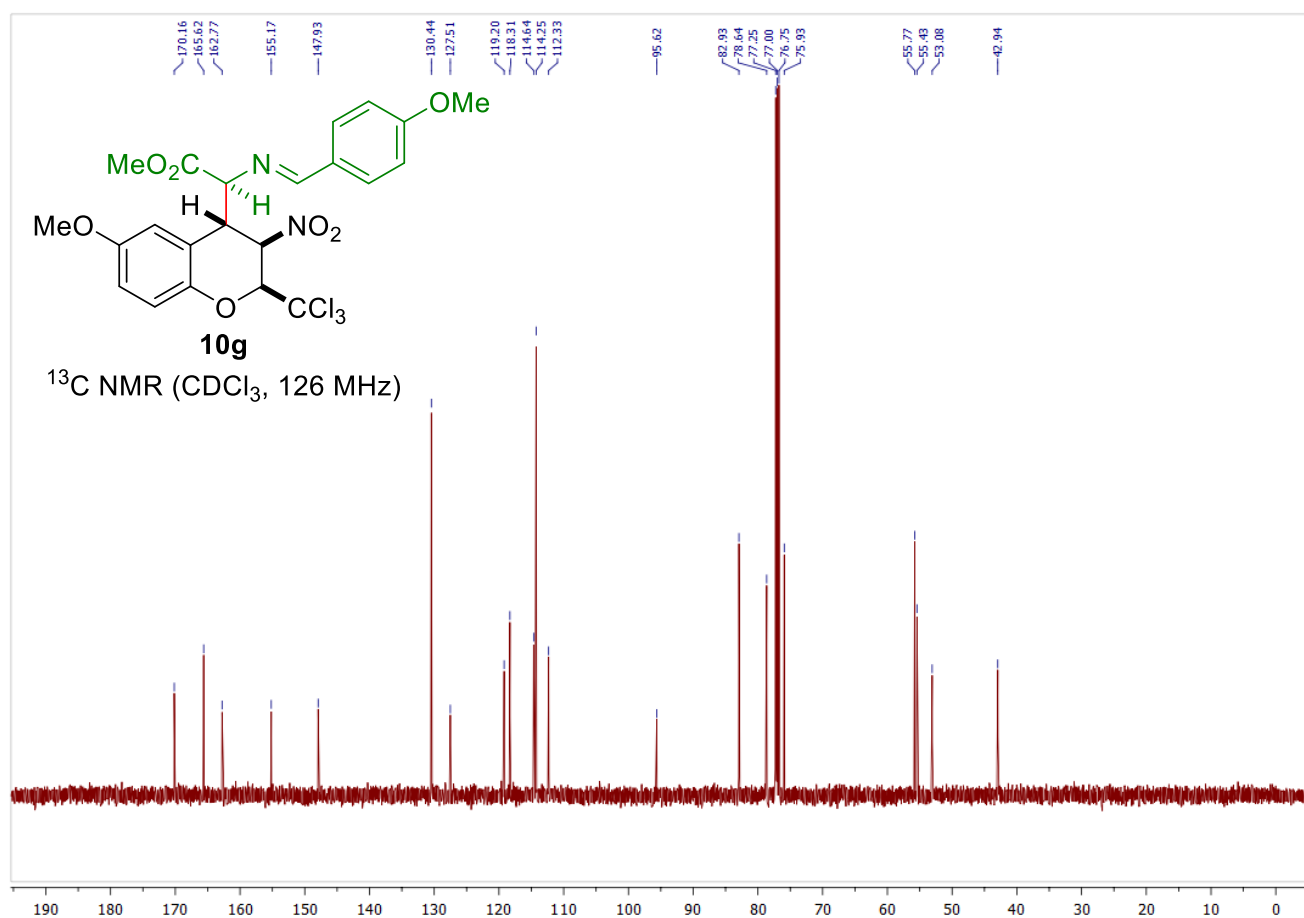
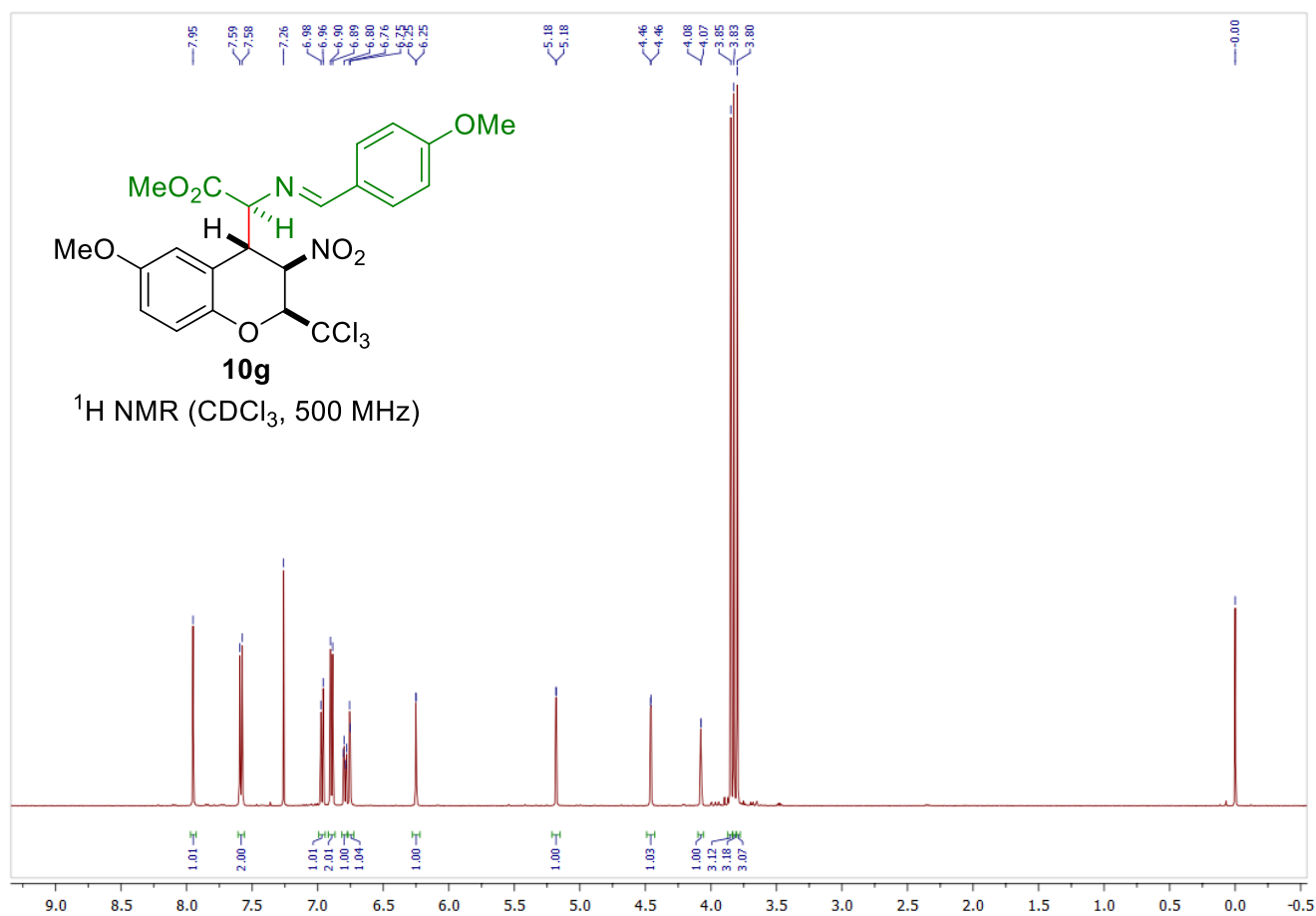


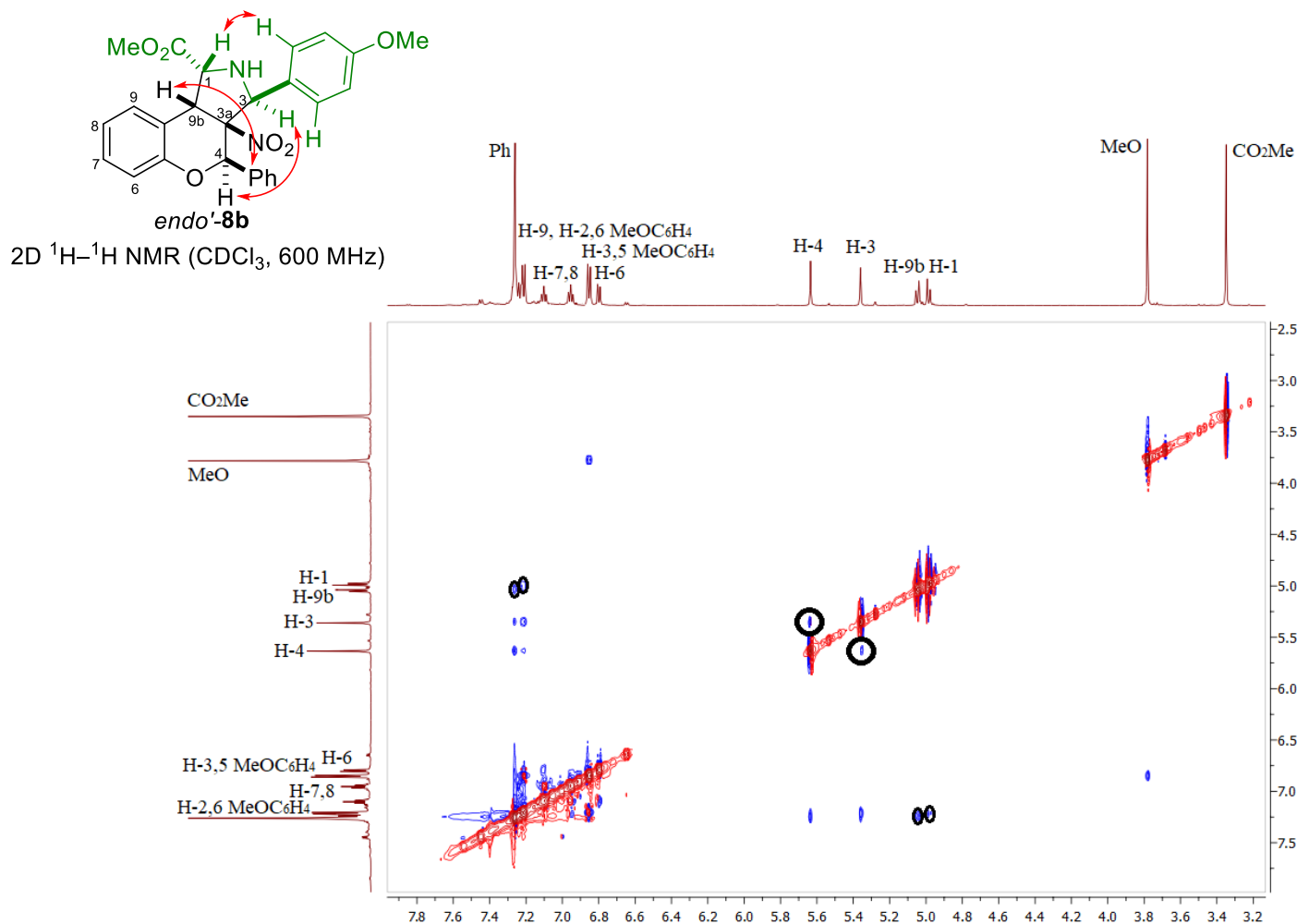
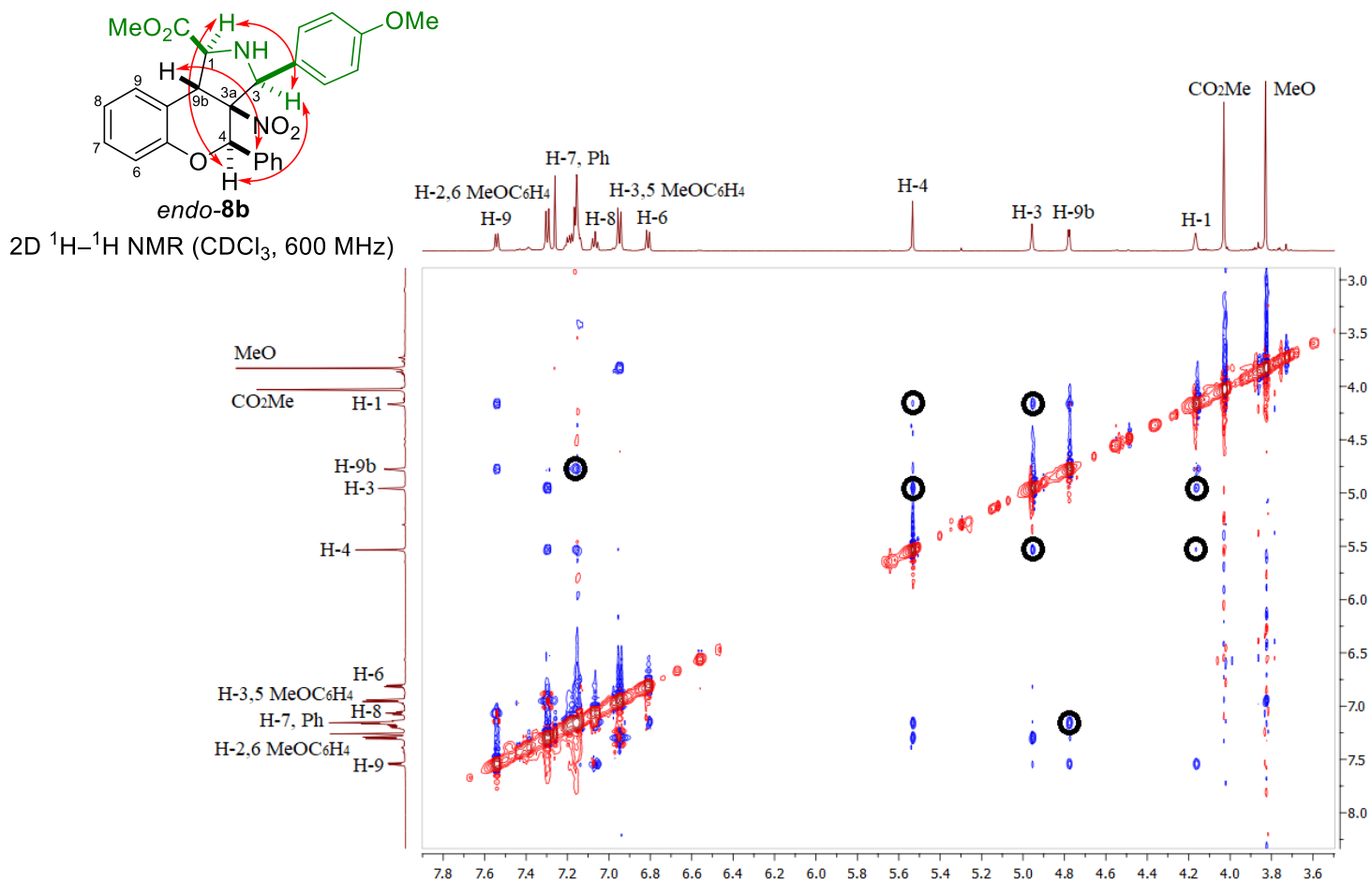


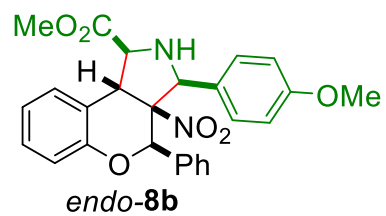




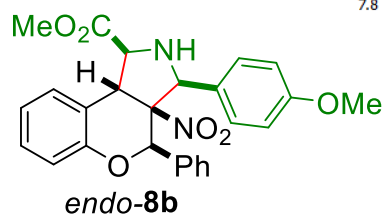
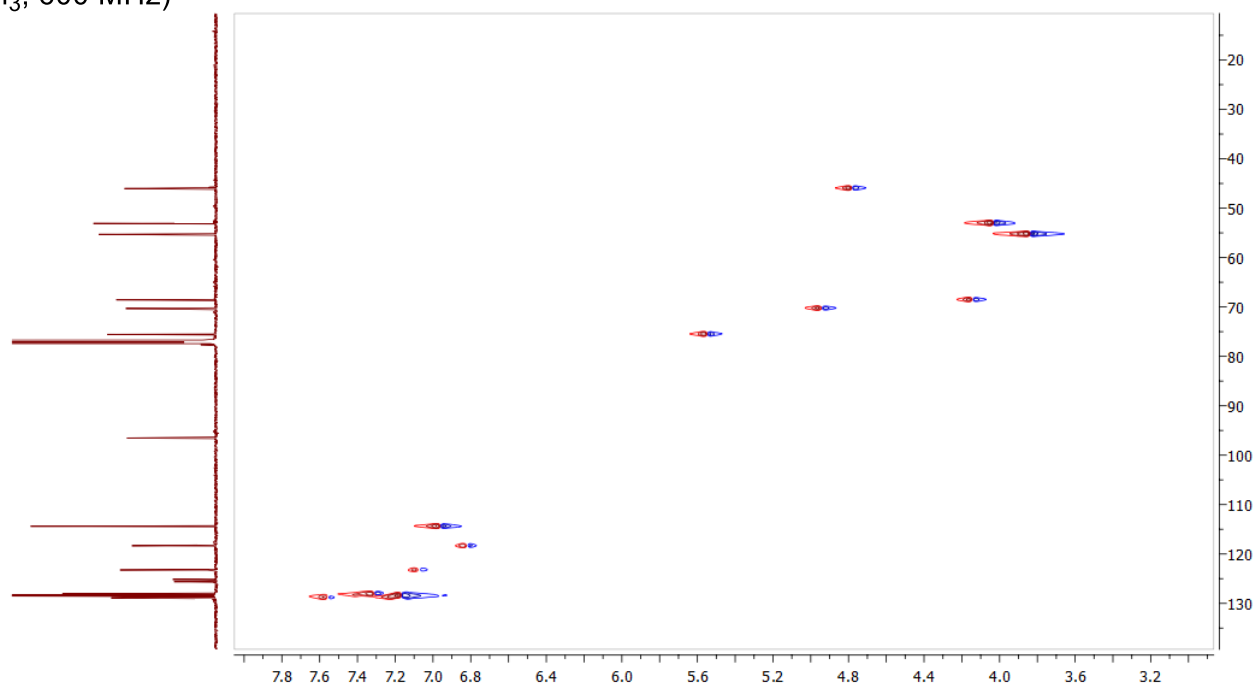




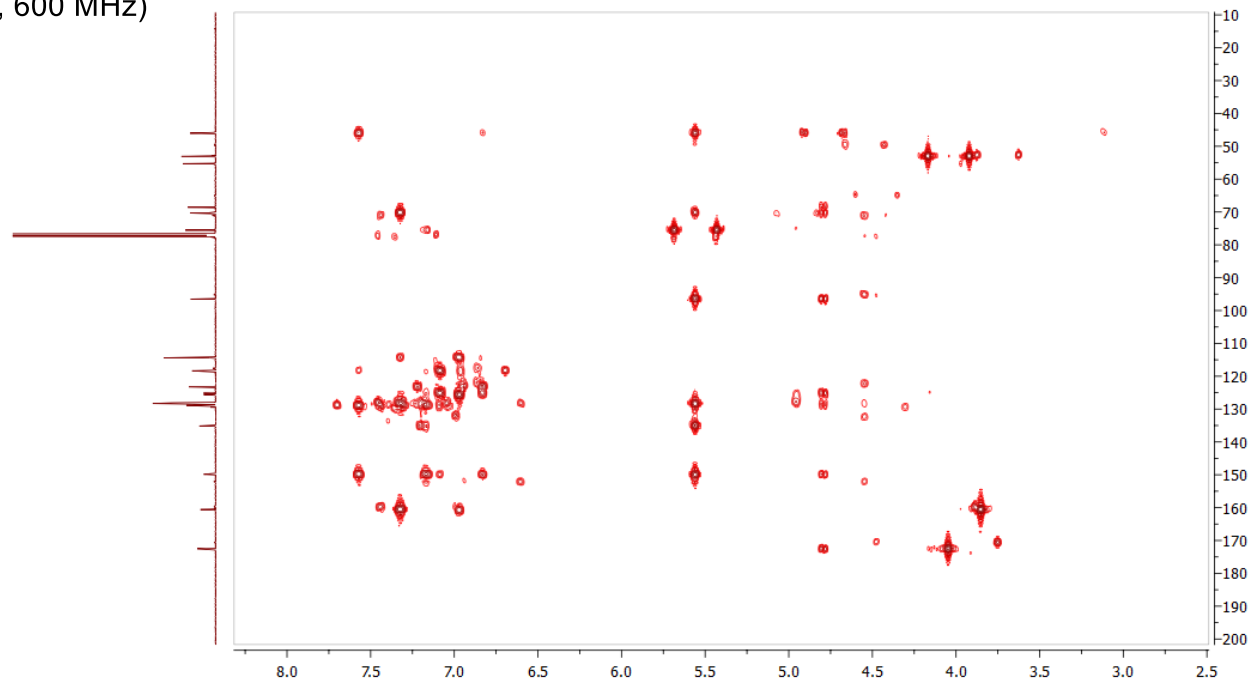


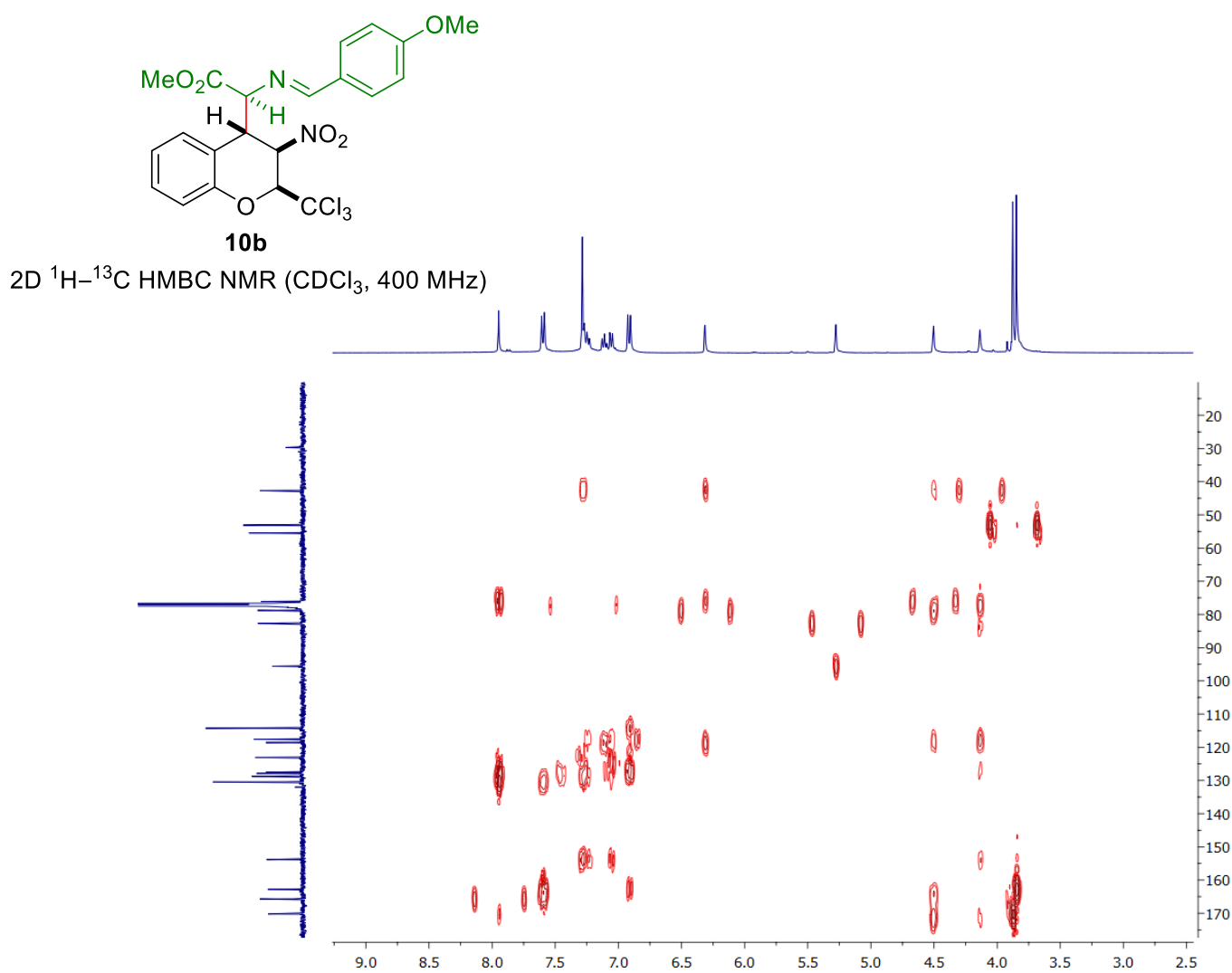
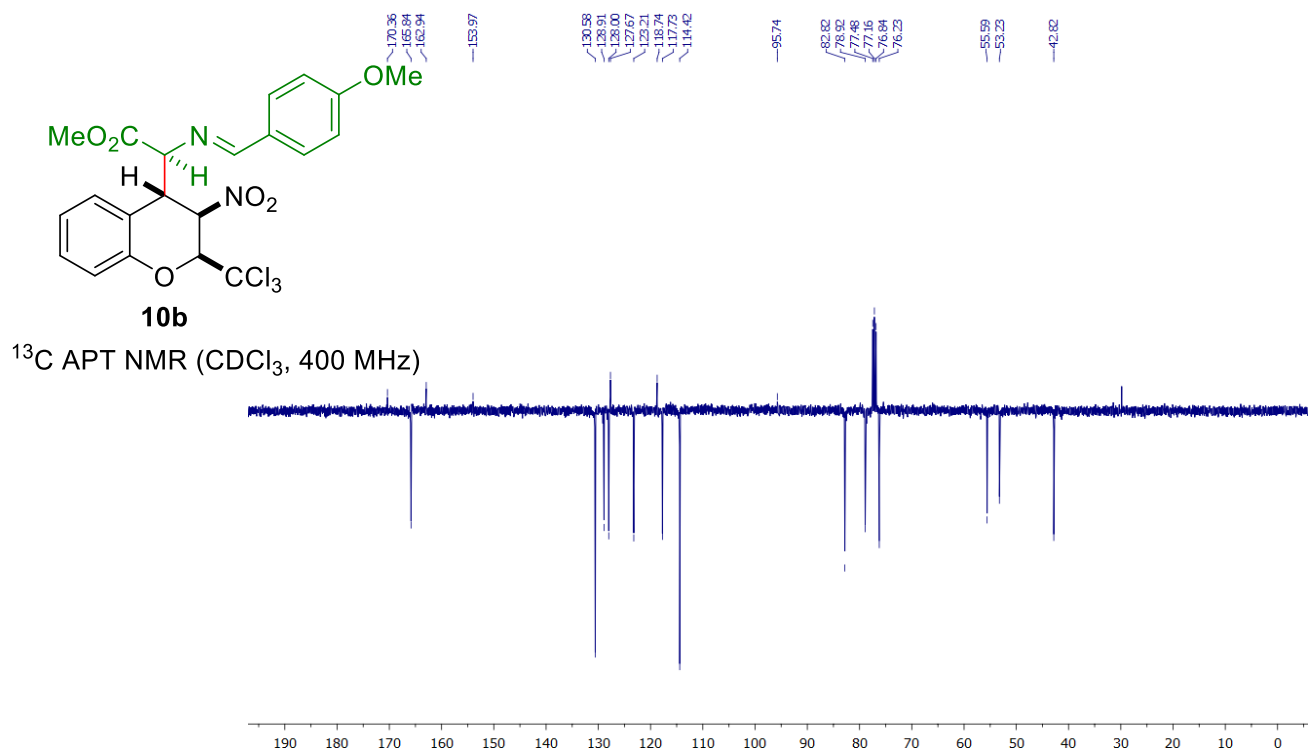


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(CDCl_3 , 600 MHz)



2D ^1H - ^{13}C HMBC NMR
(CDCl_3 , 600 MHz)

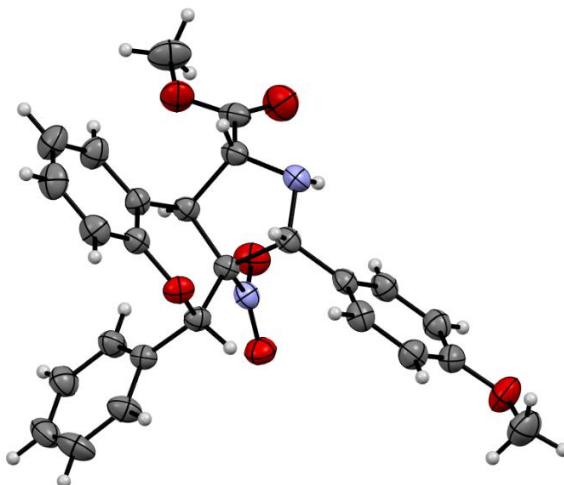




X-ray crystallography

Crystals of compounds *endo-8b*, *endo-9a* and **10c** were grown by slow evaporation of an ethyl acetate solution. Diffraction data for *endo-8b* and *endo-9a*, **10c** were collected on an Xcalibur Eos and Xcalibur 3 automatic diffractometers, respectively, using the standard procedure (Mo-K α radiation ($\lambda = 0.71073$ Å), graphite monochromator, ω -scanning, 295(2) K). An empirical adjustment for absorption was introduced. The structures were solved by direct methods and refined by the full-matrix least-squares method using the SHELX-97 program package [1]. All non-hydrogen atoms were refined with anisotropic atomic displacement and hydrogen atoms were included at the calculated positions using a riding model. The geometrical parameters were analysed using the program OLEX2 [2].

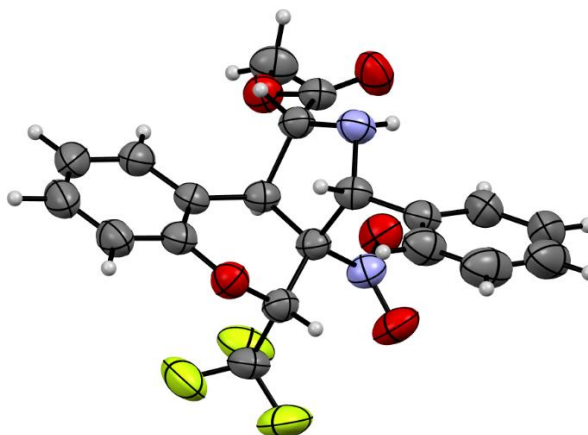
Crystal data for *endo-8b* (C₂₆H₂₄N₂O₆, 460.49). Triclinic crystals, space group $P\bar{1}$, $a = 9.9193(7)$, $b = 11.0749(6)$ and $c = 11.3940(6)$ Å, $\alpha = 88.132(4)$, $\beta = 68.824(6)$ and $\gamma = 75.357(5)^\circ$, $V = 1126.86(11)$ Å³, $D_c = 1.357$, absorption coefficient $\mu = 0.097$ mm⁻¹, $Z = 2$. The intensities of 5100 independent reflections ($R_{\text{int}} = 0.0185$) were measured. The final discrepancy factors $R_1 = 0.0492$, $wR_2 = 0.1034$, GooF = 1.000 for 3339 reflections with $I > 2\sigma(I)$; $R_1 = 0.0852$, $wR_2 = 0.1218$ (all data). Largest different peaks and holes: 0.21 and -0.22 e Å⁻³. Completeness to $\Theta = 26.32$ (98.67%). Deposition number CCDC 2213942.



Molecular structure of compound *endo-8b* (ORTEP drawing, 50% probability level)

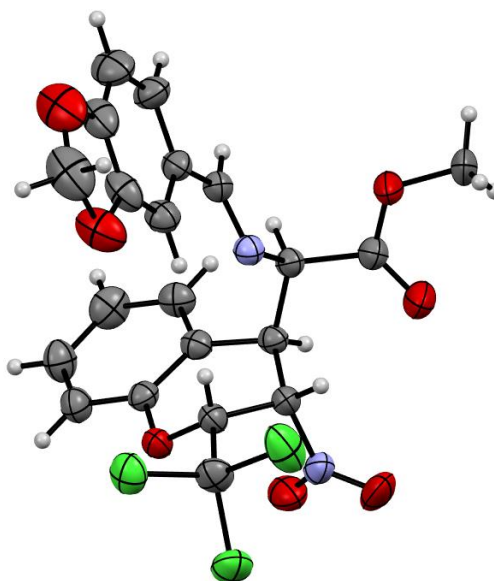
Crystal data for *endo-9a* (C₂₀H₁₇F₃N₂O₅, 422.36). Monoclinic crystals, space group $P2_1/n$, $a = 16.1467(13)$, $b = 12.4054(7)$ and $c = 19.6639(17)$ Å, $\beta = 106.101(8)^\circ$, $V = 3784.3(5)$ Å³, $D_c = 1.483$, absorption coefficient $\mu = 0.126$ mm⁻¹, $Z = 8$. The intensities of 10373 independent reflections ($R_{\text{int}} = 0.0544$) were measured. The final discrepancy factors $R_1 = 0.0524$, $wR_2 = 0.0978$, GooF = 0.982 for

4180 reflections with $I > 2\sigma(I)$; $R_1 = 0.1600$, $wR_2 = 0.1492$ (all data). Largest different peaks and holes: 0.17 and $-0.20 \text{ e } \text{\AA}^{-3}$. Completeness to $\theta = 25.24$ (99.6%). Deposition number CCDC 2213944.



Molecular structure of compound *endo*-**9a** (ORTEP drawing, 50% probability level)

Crystal data for 10c ($\text{C}_{21}\text{H}_{17}\text{Cl}_3\text{N}_2\text{O}_7$, 515.72). Monoclinic crystals, space group $P2_1/n$, $a = 10.7321(16)$, $b = 15.9306(14)$ and $c = 13.532(2) \text{ \AA}$, $\beta = 96.095(12)^\circ$, $V = 2300.5(5) \text{ \AA}^3$, $D_c = 1.489$, absorption coefficient $\mu = 0.444 \text{ mm}^{-1}$, $Z = 4$. The intensities of 5629 independent reflections ($R_{\text{int}} = 0.0779$) were measured. The final discrepancy factors $R_1 = 0.0664$, $wR_2 = 0.1626$, $\text{GooF} = 0.959$ for 2034 reflections with $I > 2\sigma(I)$; $R_1 = 0.1863$, $wR_2 = 0.2342$ (all data). Largest different peaks and holes: 0.33 and $-0.41 \text{ e } \text{\AA}^{-3}$. Completeness to $\theta = 25.24$ (98.8%). Deposition number CCDC 2213943.



Molecular structure of compound **10c** (ORTEP drawing, 30% probability level)

References

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45. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX2: A complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **2009**, *42*, 339–341.