

[4+2] Annulation Reaction of *in situ* Generated Azoalkenes with Azlactones: Access to 4,5-Dihydropyridazin-3(2H)-ones

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Supporting Information

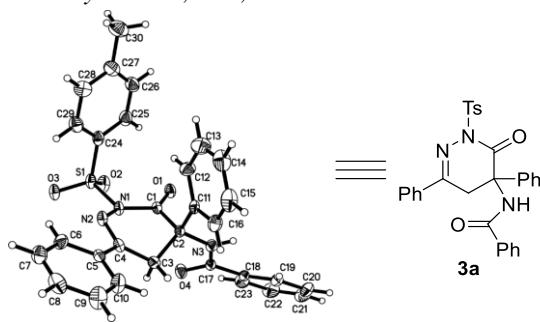
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1. X-ray crystal structure of compound

Single crystals of compound **3a** were prepared from the mixture solvent of dichloromethane and EtOH. A suitable crystal was selected for structure determination on a Xcalibur, Eos, Gemini diffractometer. The crystal was kept at 293(2) 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 minimisation.

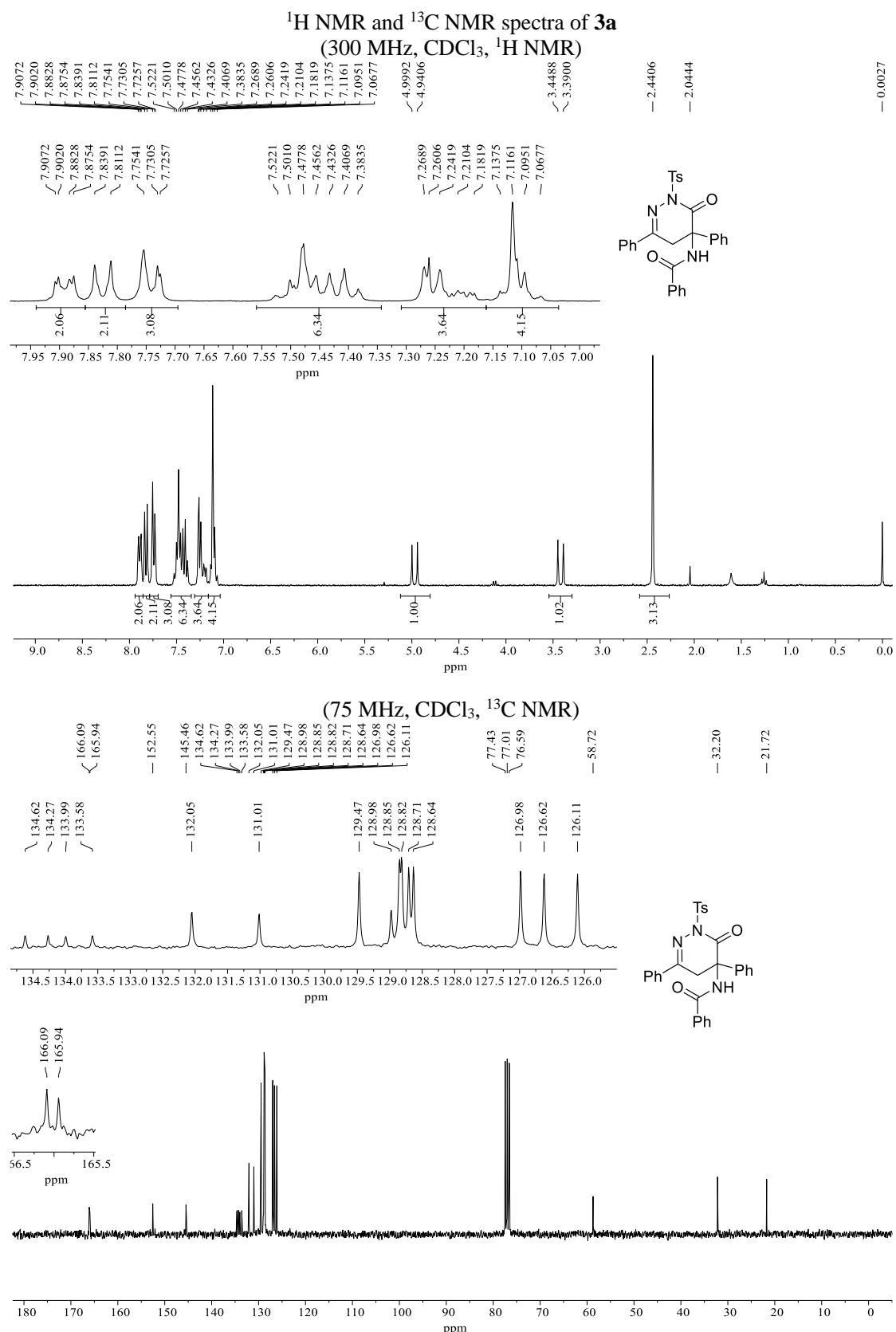
- [1] Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J; Howard, J. A. K.; Puschmann, H. *J. Appl. Cryst.* **2009**, *42*, 339-341.
- [2] Sheldrick, G. M. *Acta Cryst.* **2008**, *A64*, 112-122.
- [3] Sheldrick, G. M. *Acta Cryst.* **2015**, *C71*, 3-8.



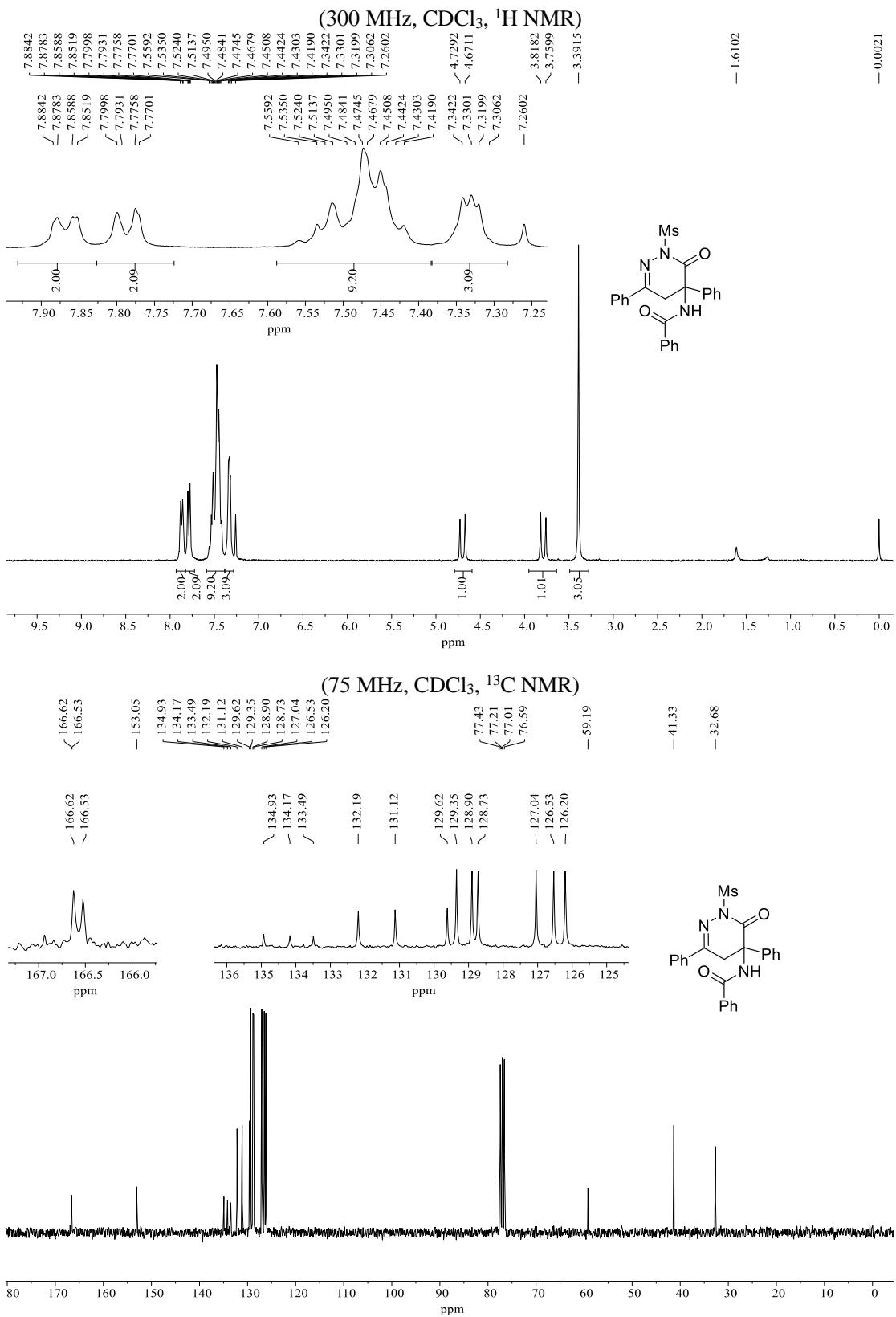
ORTEP of **3a** (at 50% level)
Crystal data and structure refinement for **3a** (CCDC 2012663).

Identification code	3a
Empirical formula	C ₃₀ H ₂₅ N ₃ O ₄ S
Formula weight	523.59
Temperature/K	293(2)
Crystal system	monoclinic
Space group	C2/c
a/Å	30.4299(4)
b/Å	8.50556(7)
c/Å	22.1713(3)
α/°	90
β/°	112.6255(16)
γ/°	90
Volume/Å ³	5296.82(13)
Z	8
ρ _{calc} g/cm ³	1.313
μ/mm ⁻¹	1.422
F(000)	2192.0
Crystal size/mm ³	0.19 × 0.15 × 0.12
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.512 to 134.154
Index ranges	-36 ≤ h ≤ 33, -10 ≤ k ≤ 6, -26 ≤ l ≤ 26
Reflections collected	11203
Independent reflections	4716 [R _{int} = 0.0235, R _{sigma} = 0.0257]
Data/restraints/parameters	4716/5/340
Goodness-of-fit on F ²	1.044
Final R indexes [I>=2σ (I)]	R ₁ = 0.0517, wR ₂ = 0.1409
Final R indexes [all data]	R ₁ = 0.0599, wR ₂ = 0.1518
Largest diff. peak/hole / e Å ⁻³	0.23/-0.47

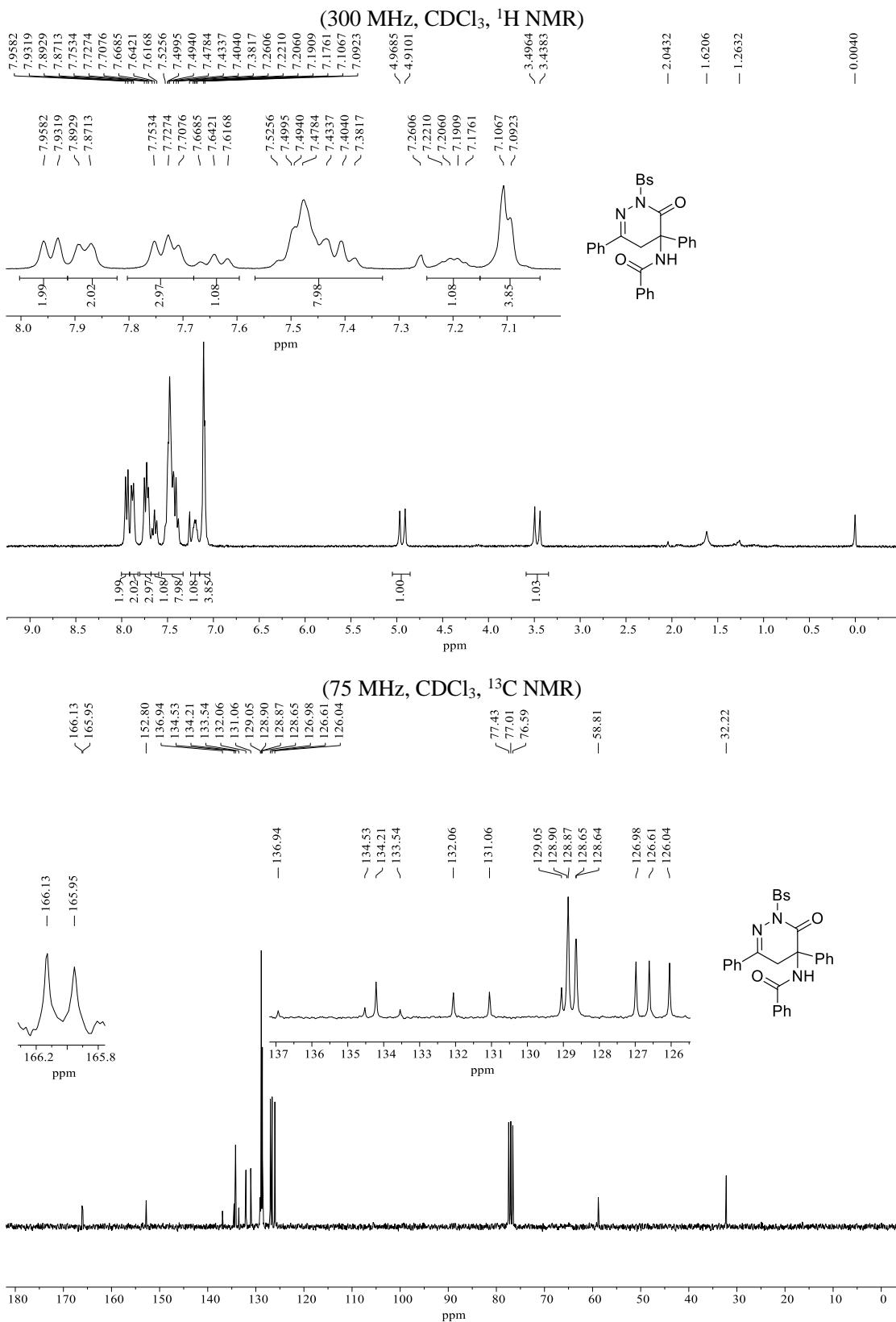
2 ^1H NMR and ^{13}C NMR spectra for compounds 3 and 5-7.



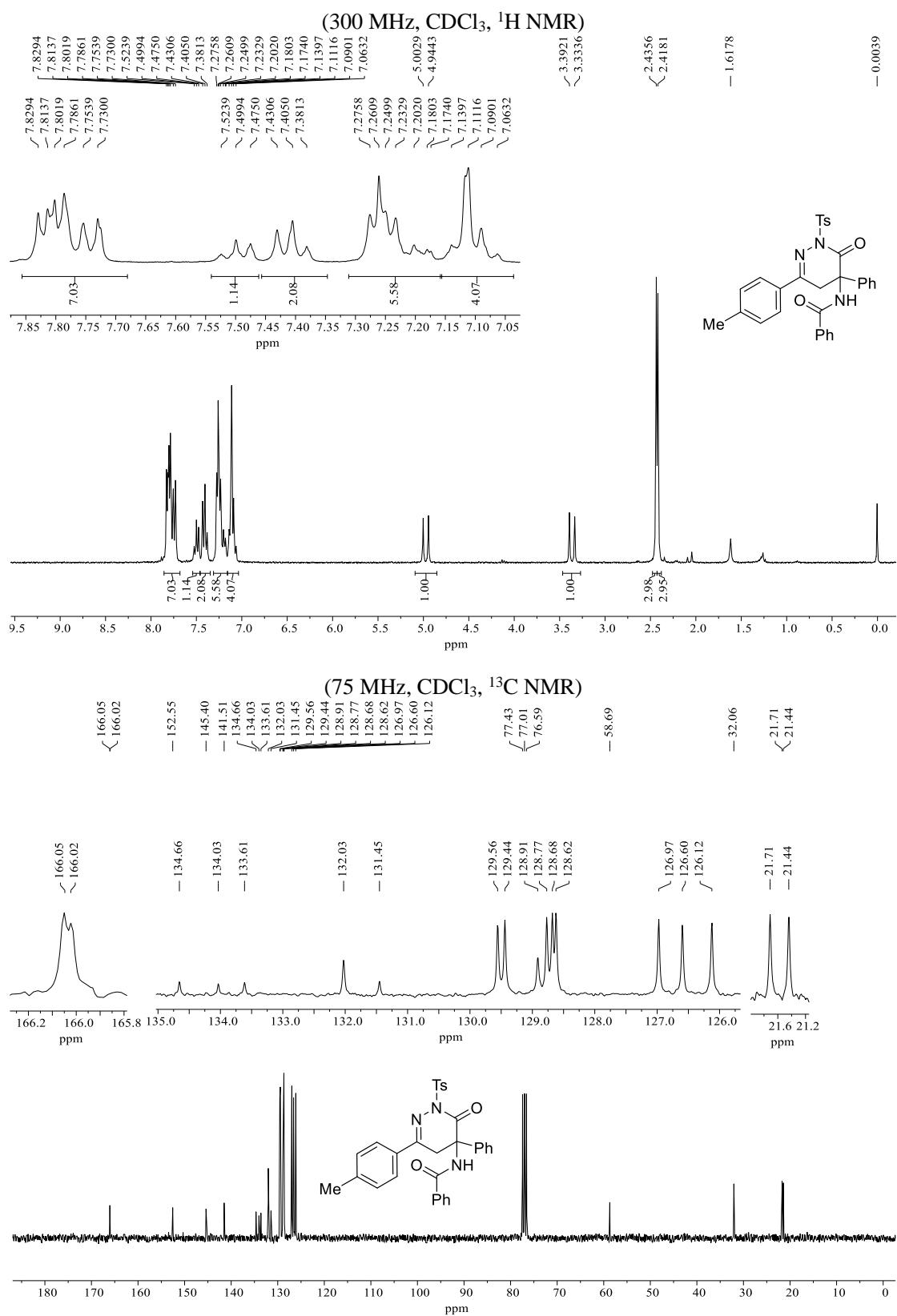
¹H NMR and ¹³C NMR spectra of 3b



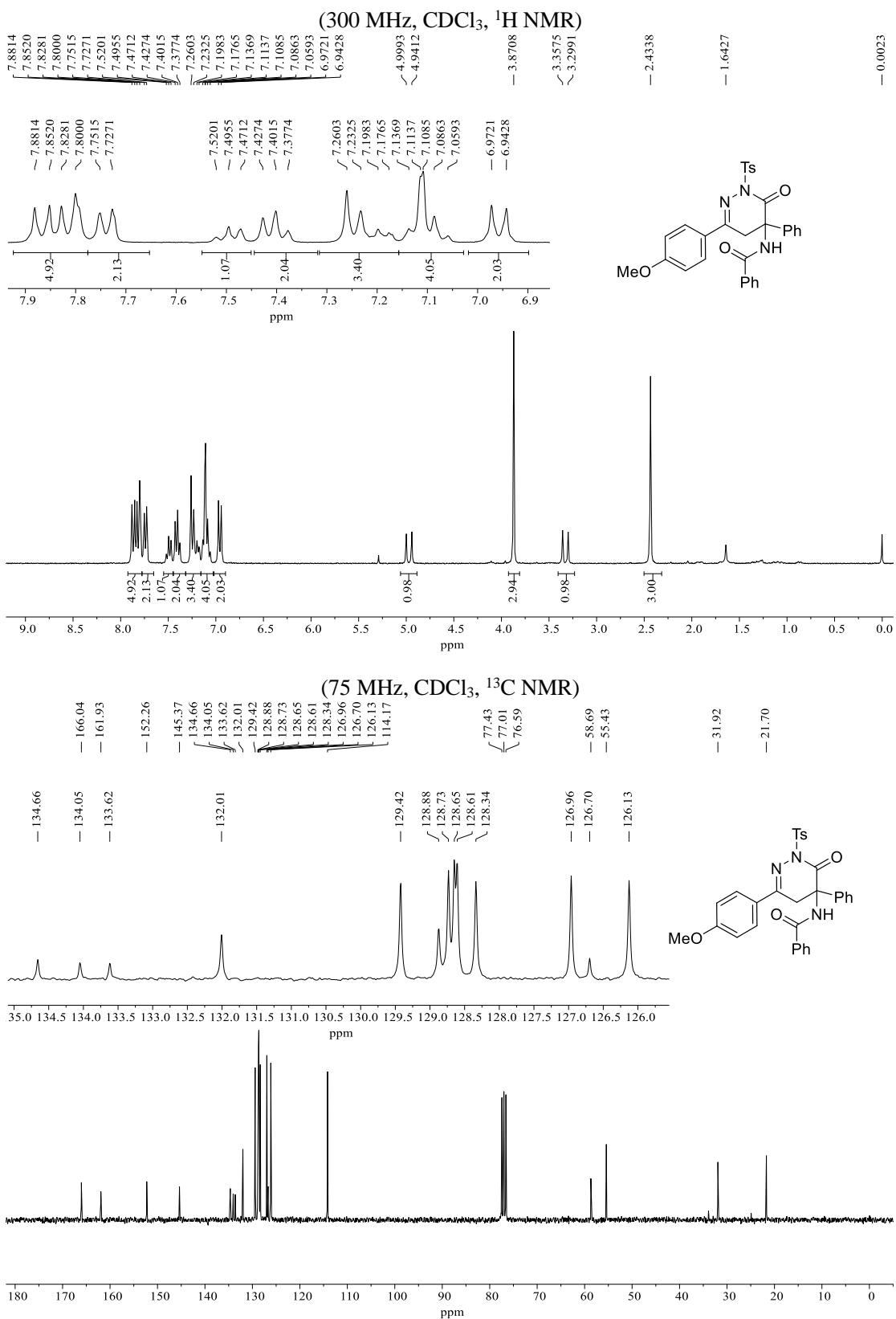
¹H NMR and ¹³C NMR spectra of **3c**



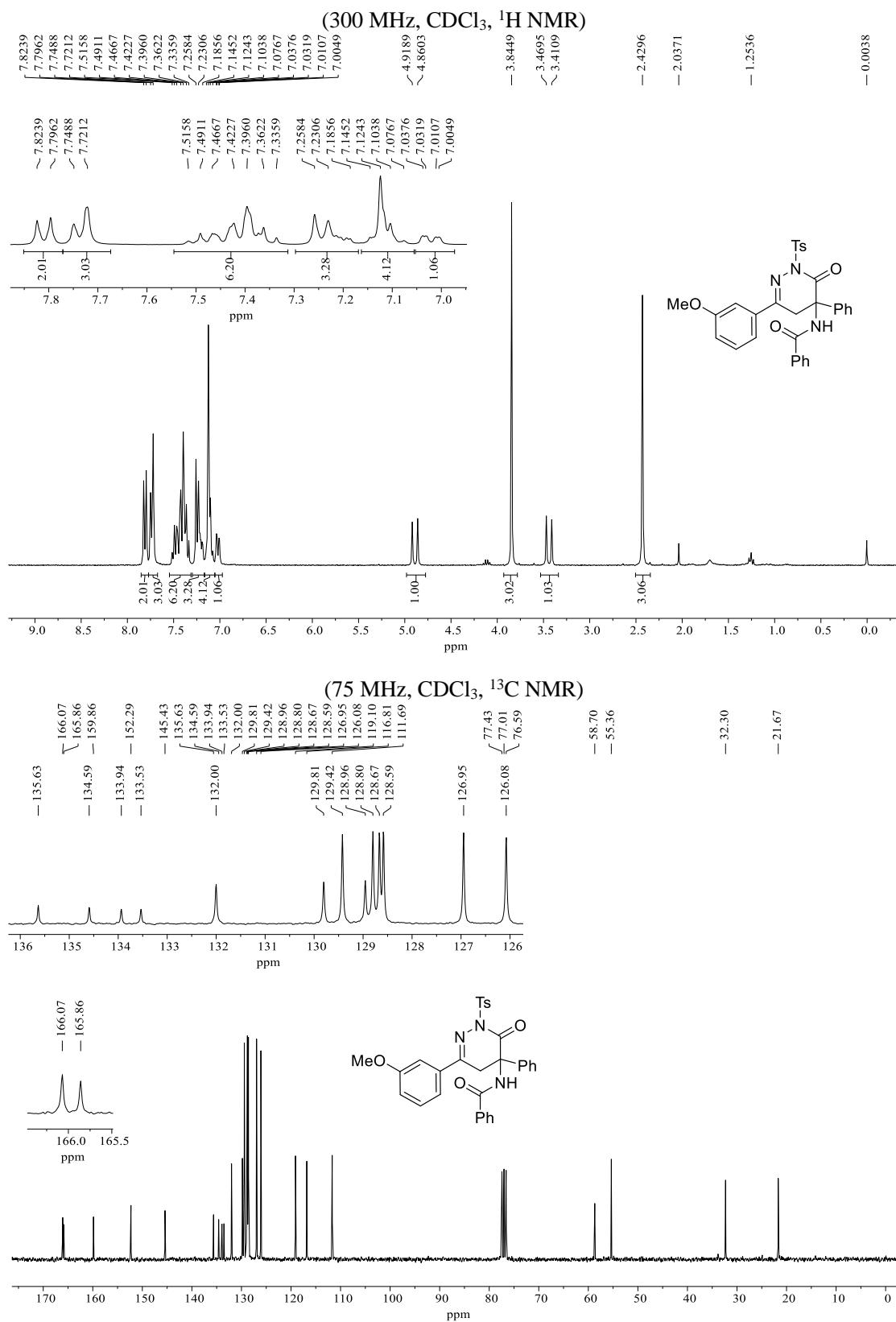
¹H NMR and ¹³C NMR spectra of **3d**



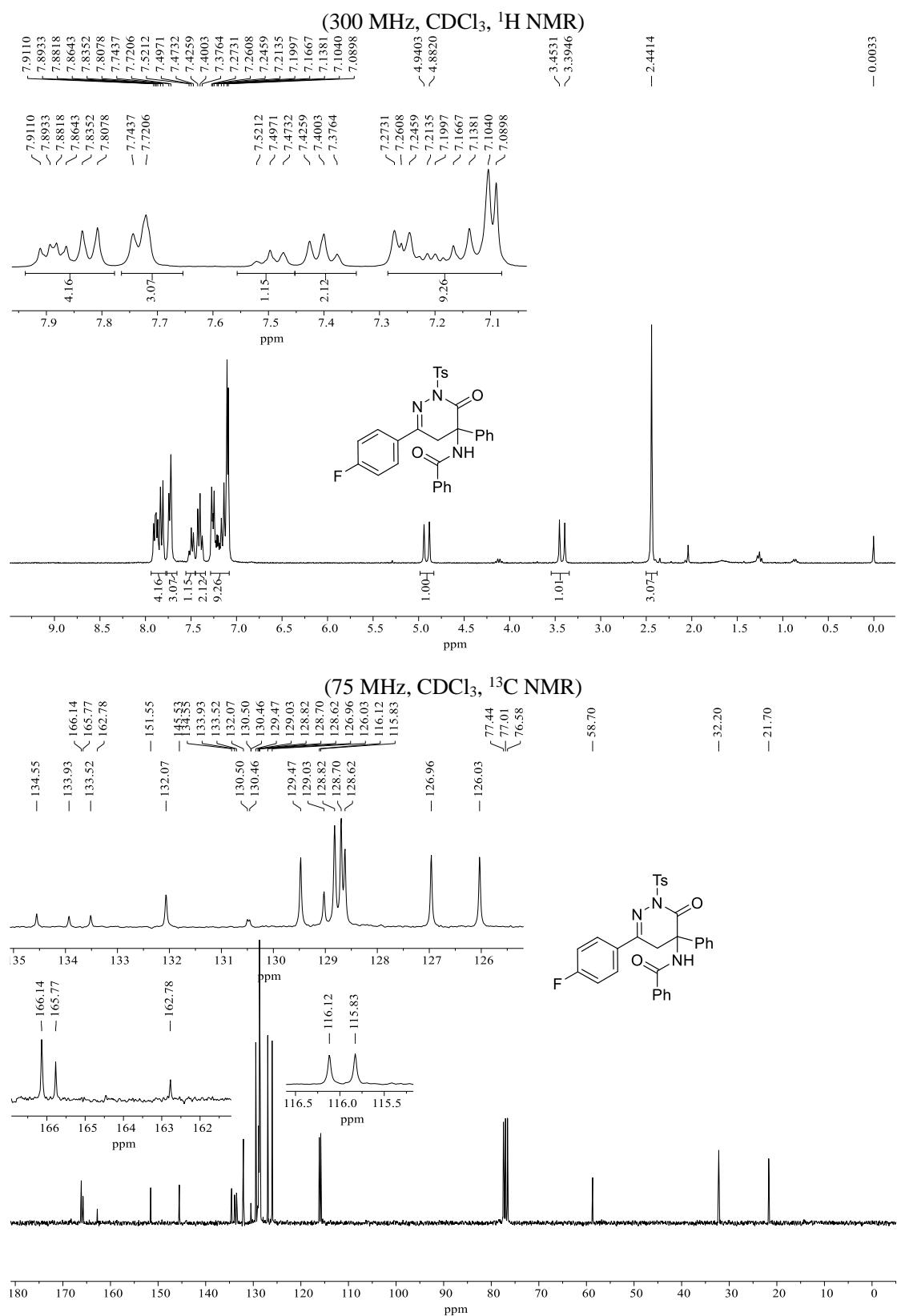
¹H NMR and ¹³C NMR spectra of **3e**



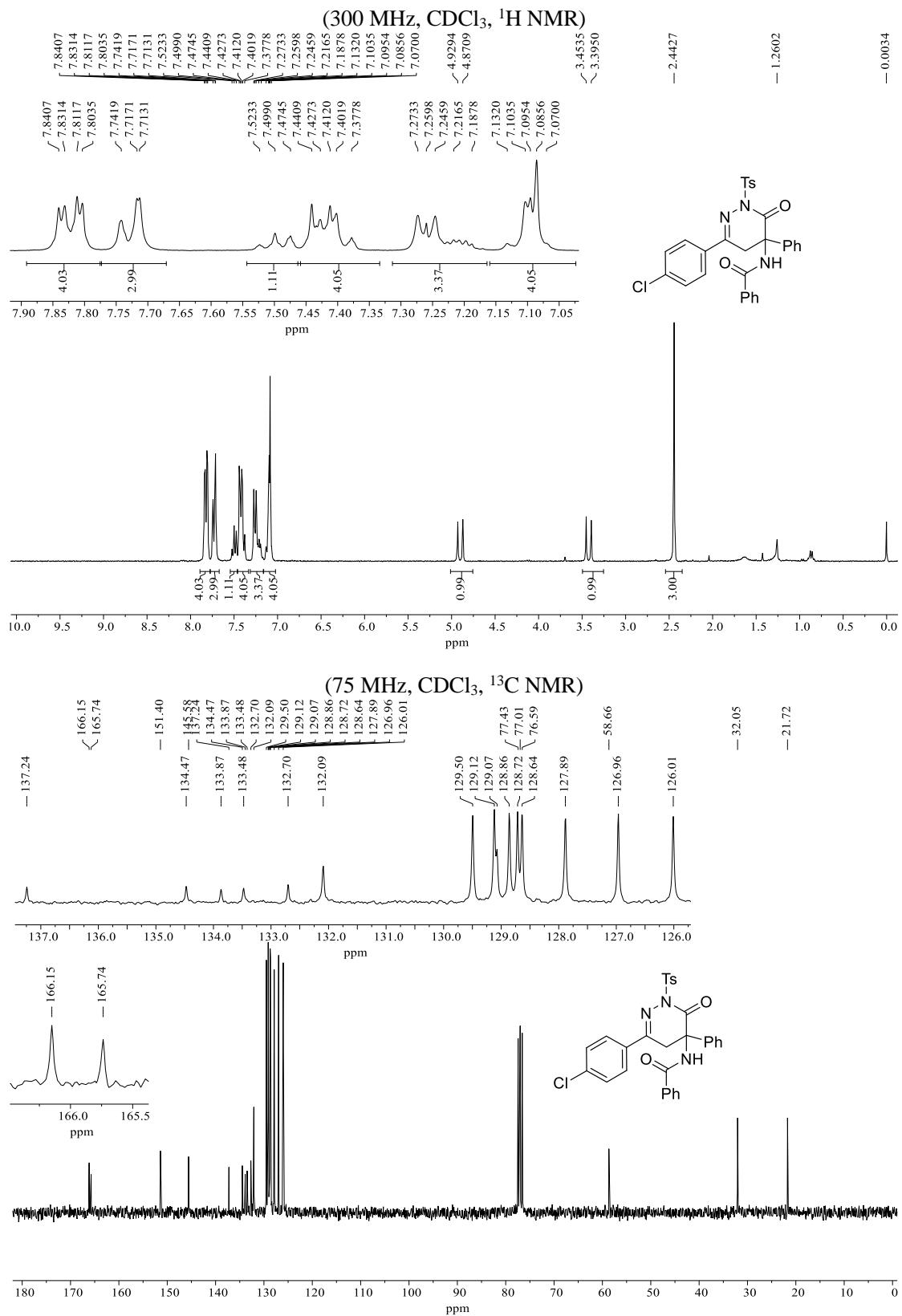
¹H NMR and ¹³C NMR spectra of **3f**



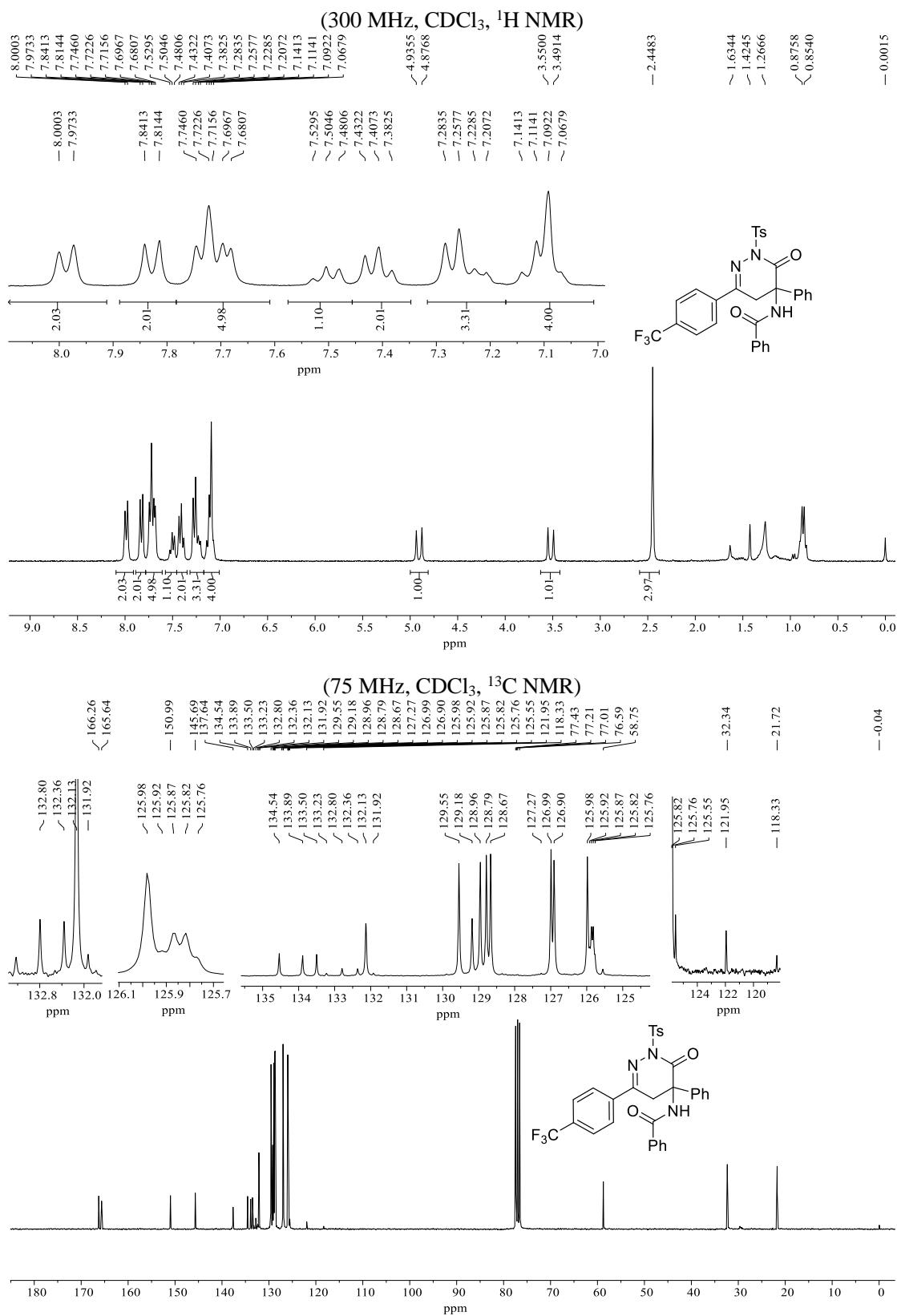
¹H NMR and ¹³C NMR spectra of **3g**



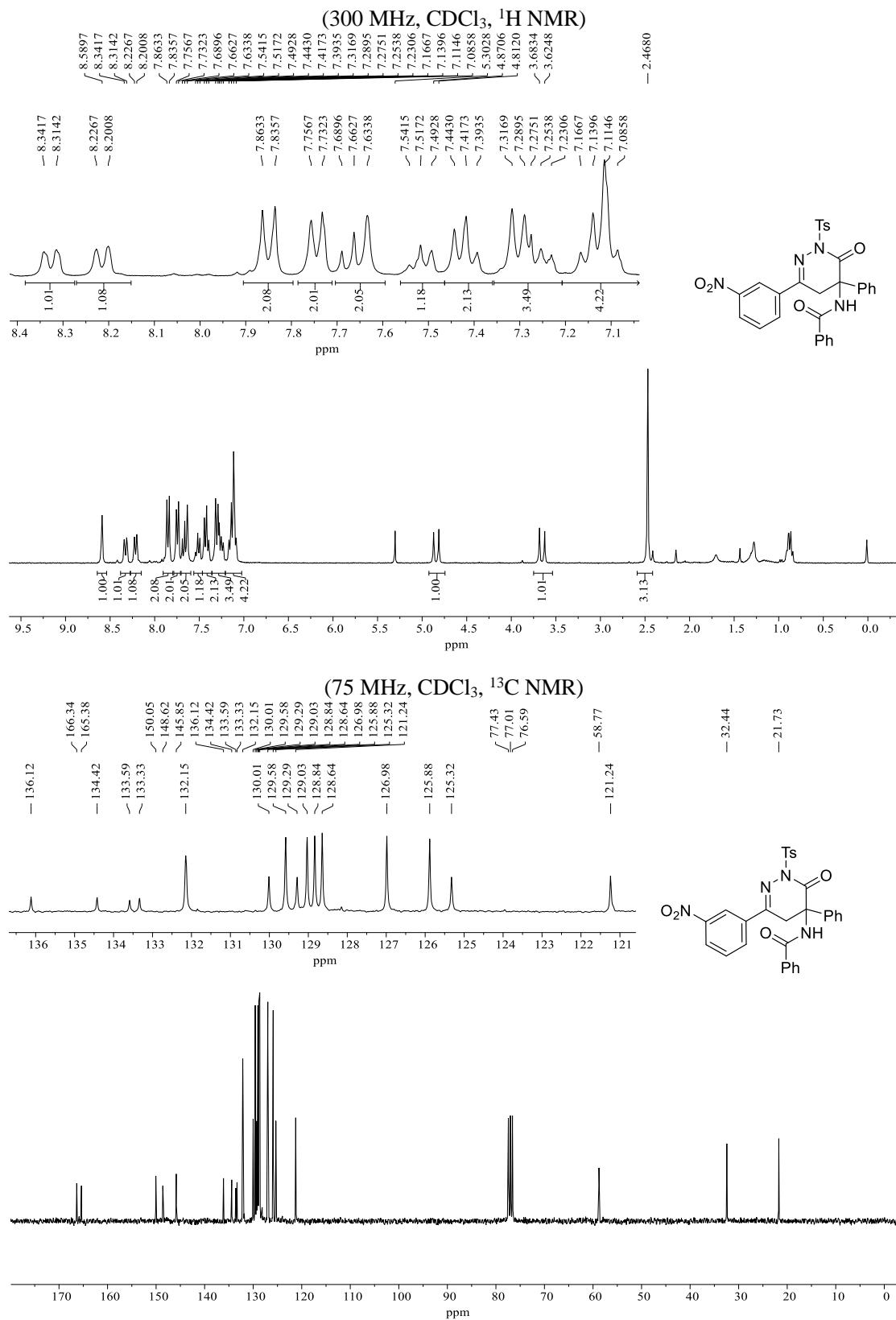
¹H NMR and ¹³C NMR spectra of 3h



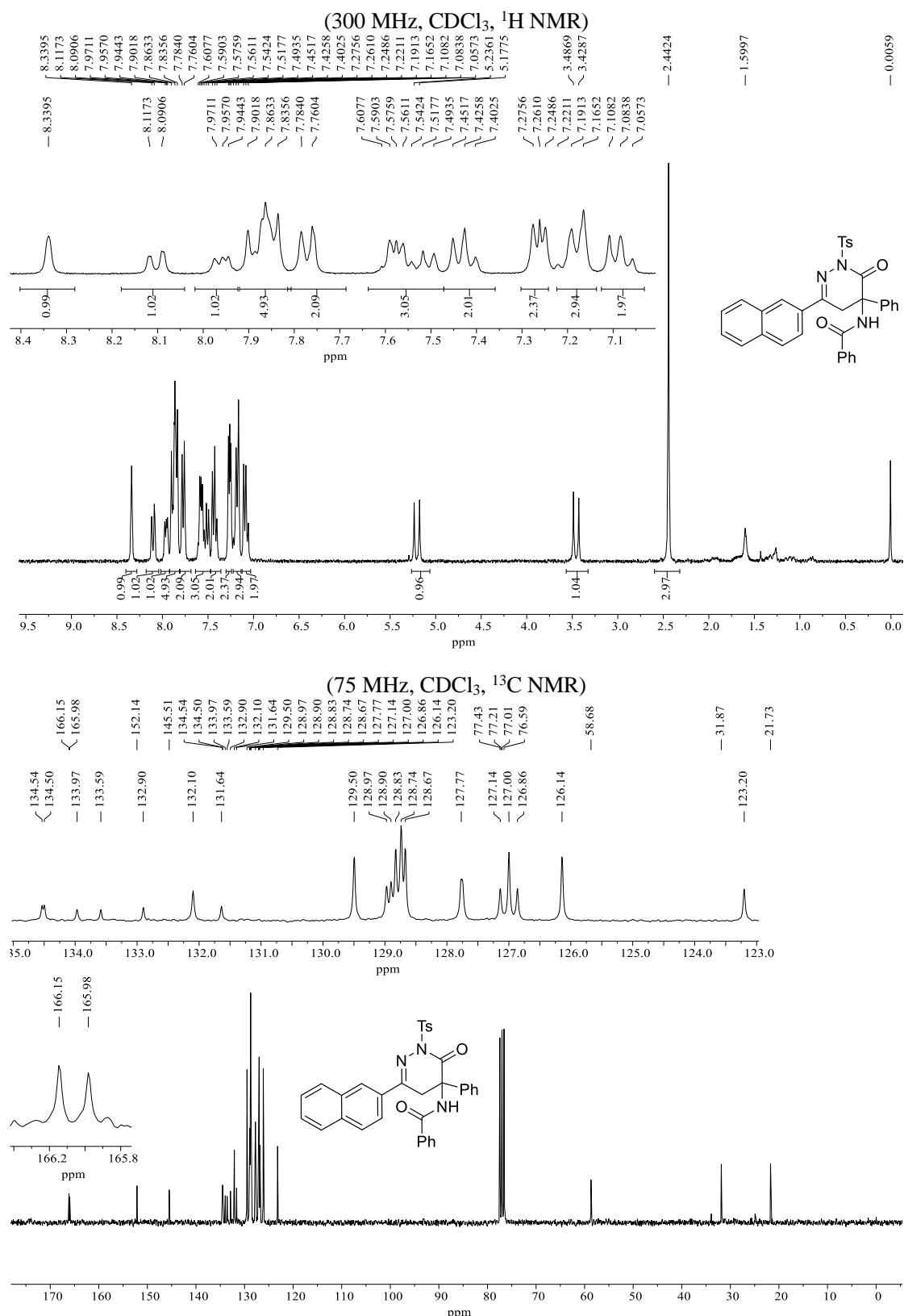
¹H NMR and ¹³C NMR spectra of **3i**



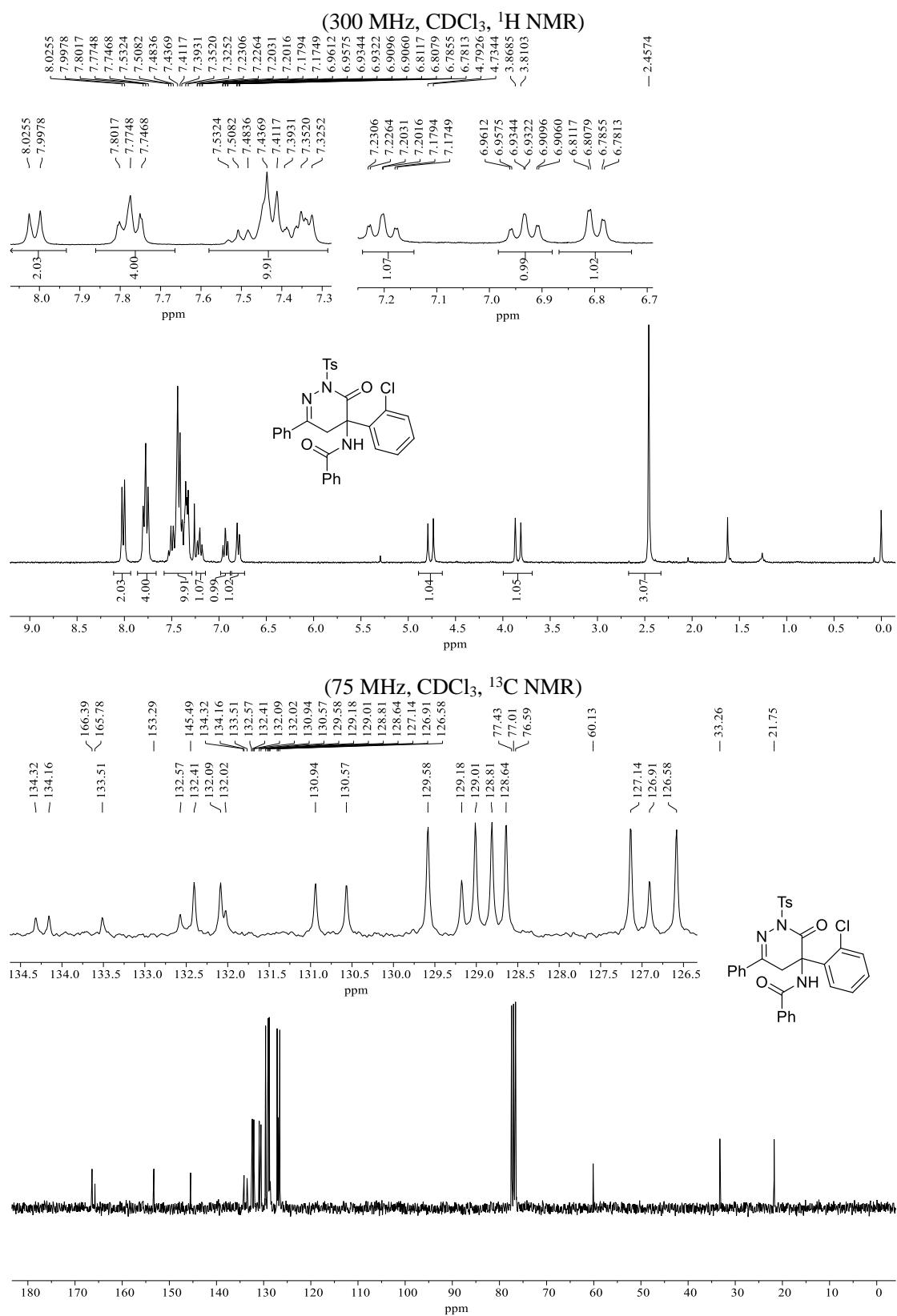
¹H NMR and ¹³C NMR spectra of **3j**



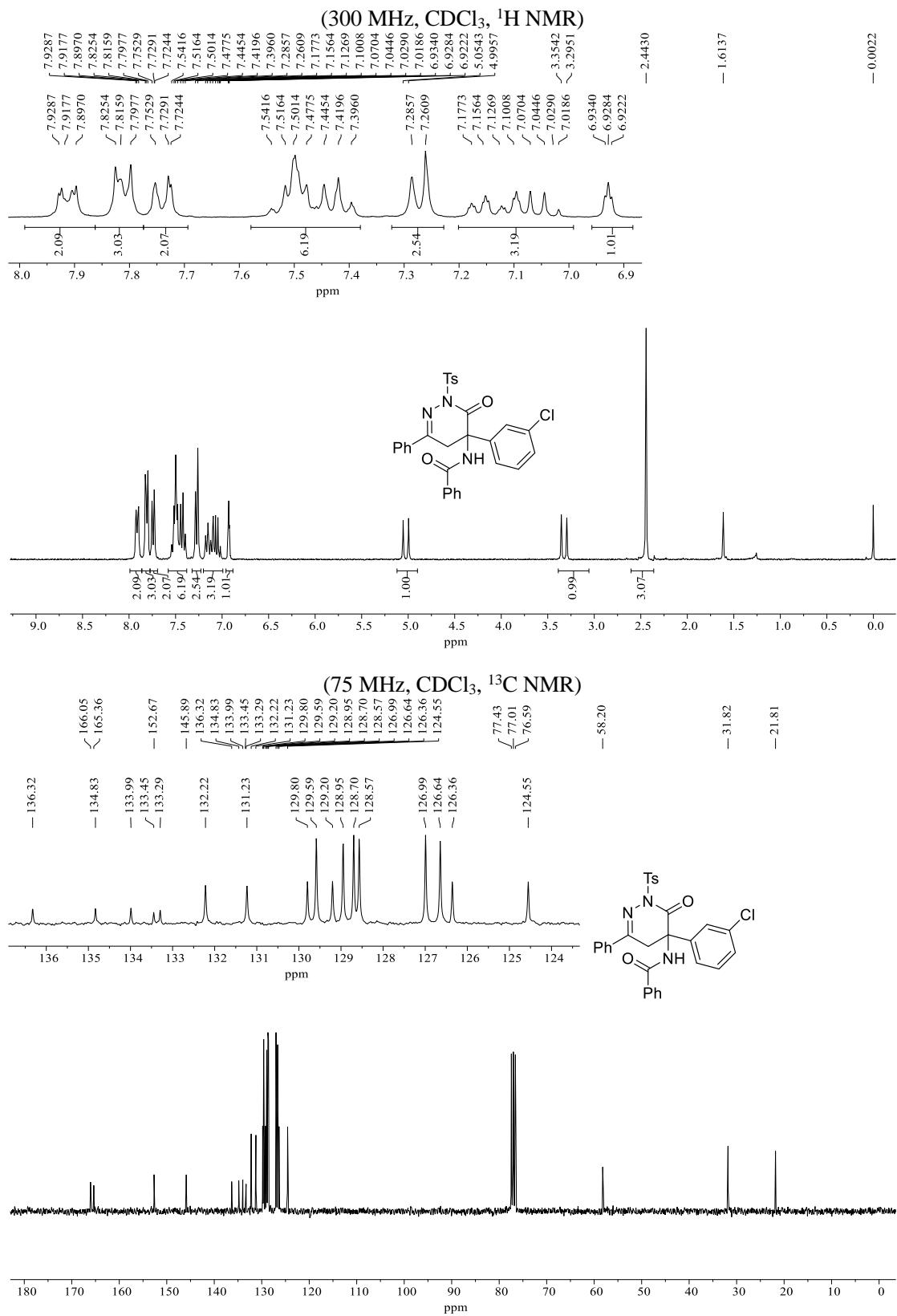
¹H NMR and ¹³C NMR spectra of **3k**



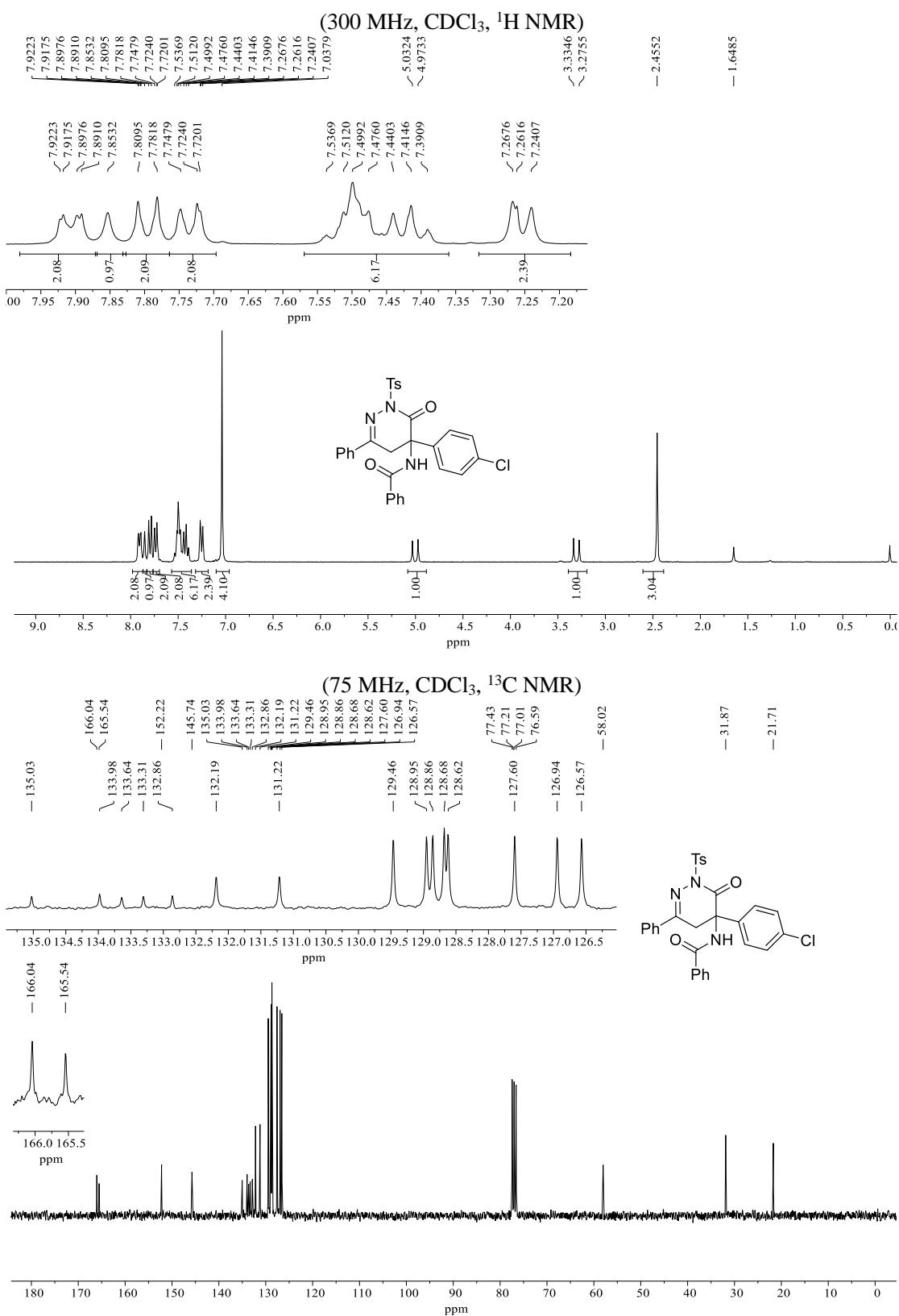
¹H NMR and ¹³C NMR spectra of **3I**



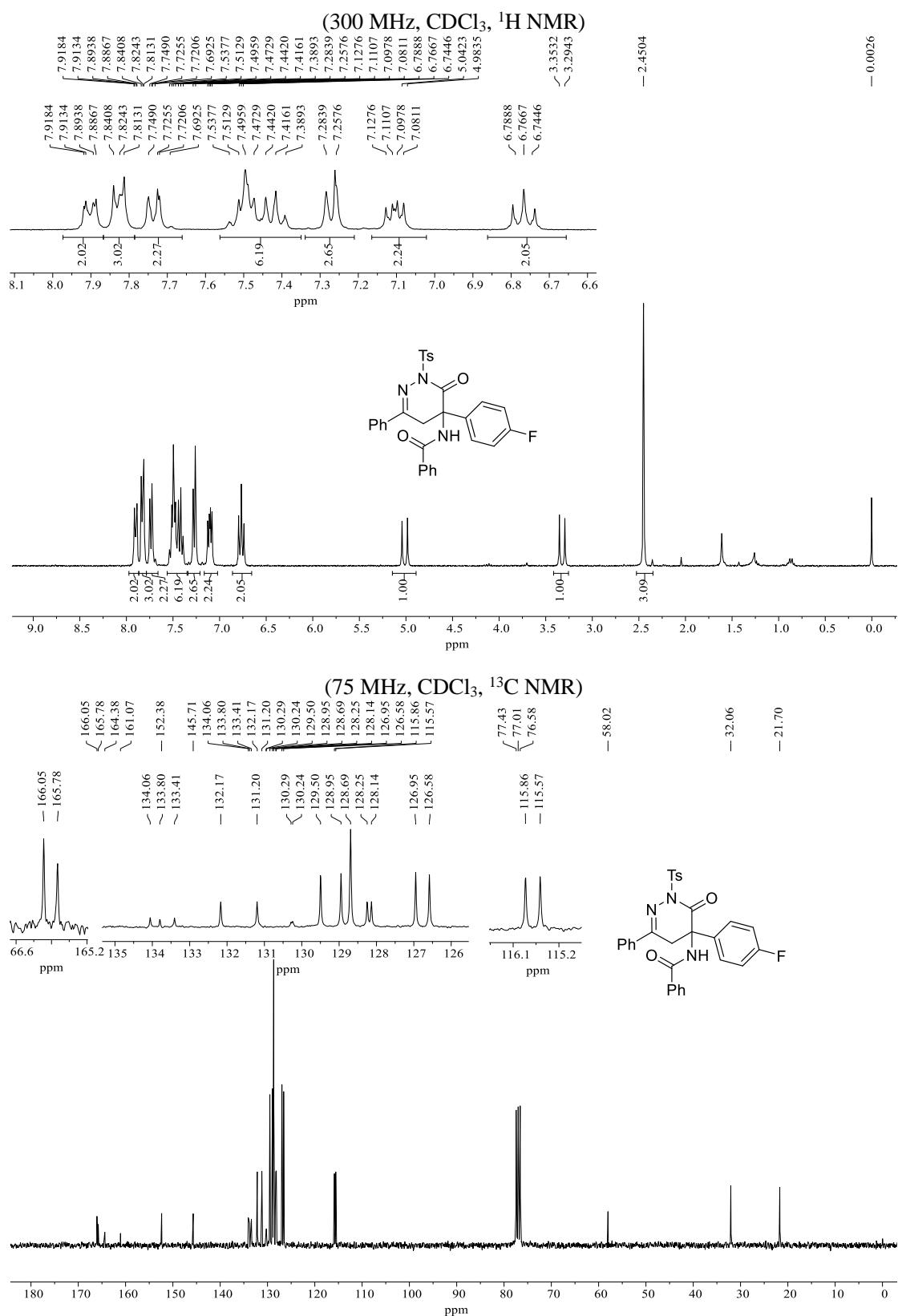
¹H NMR and ¹³C NMR spectra of **3m**



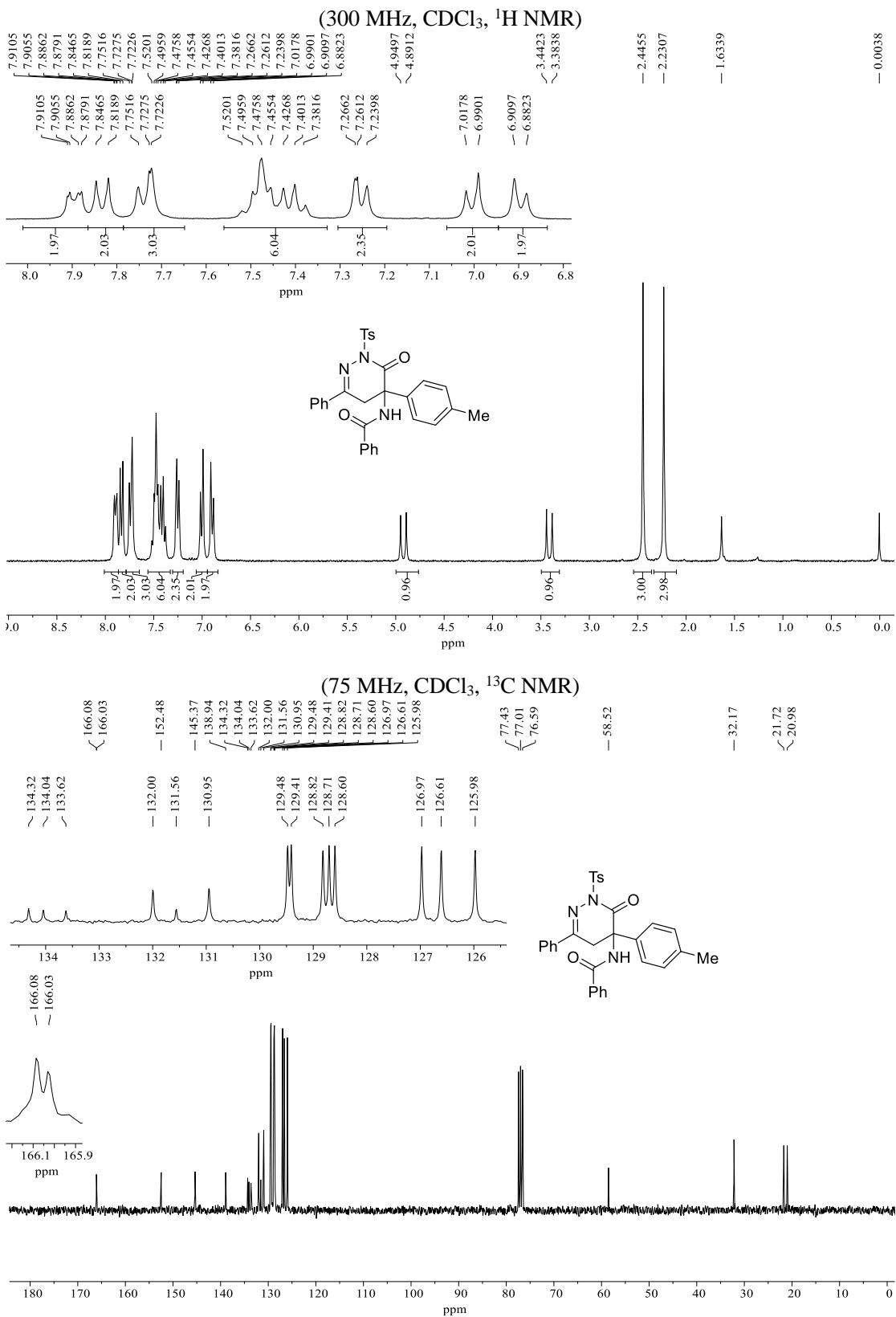
¹H NMR and ¹³C NMR spectra of 3n



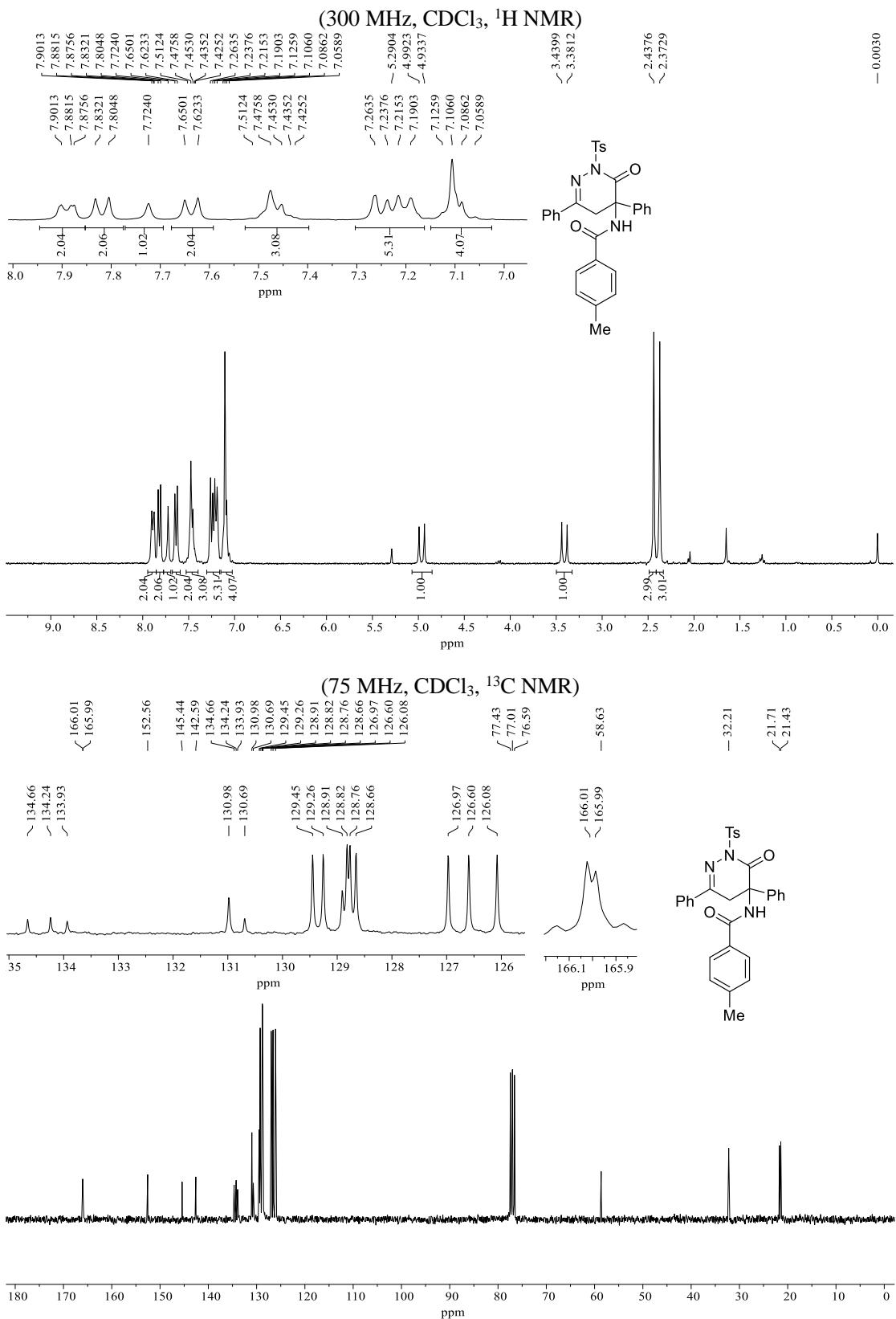
¹H NMR and ¹³C NMR spectra of **3o**



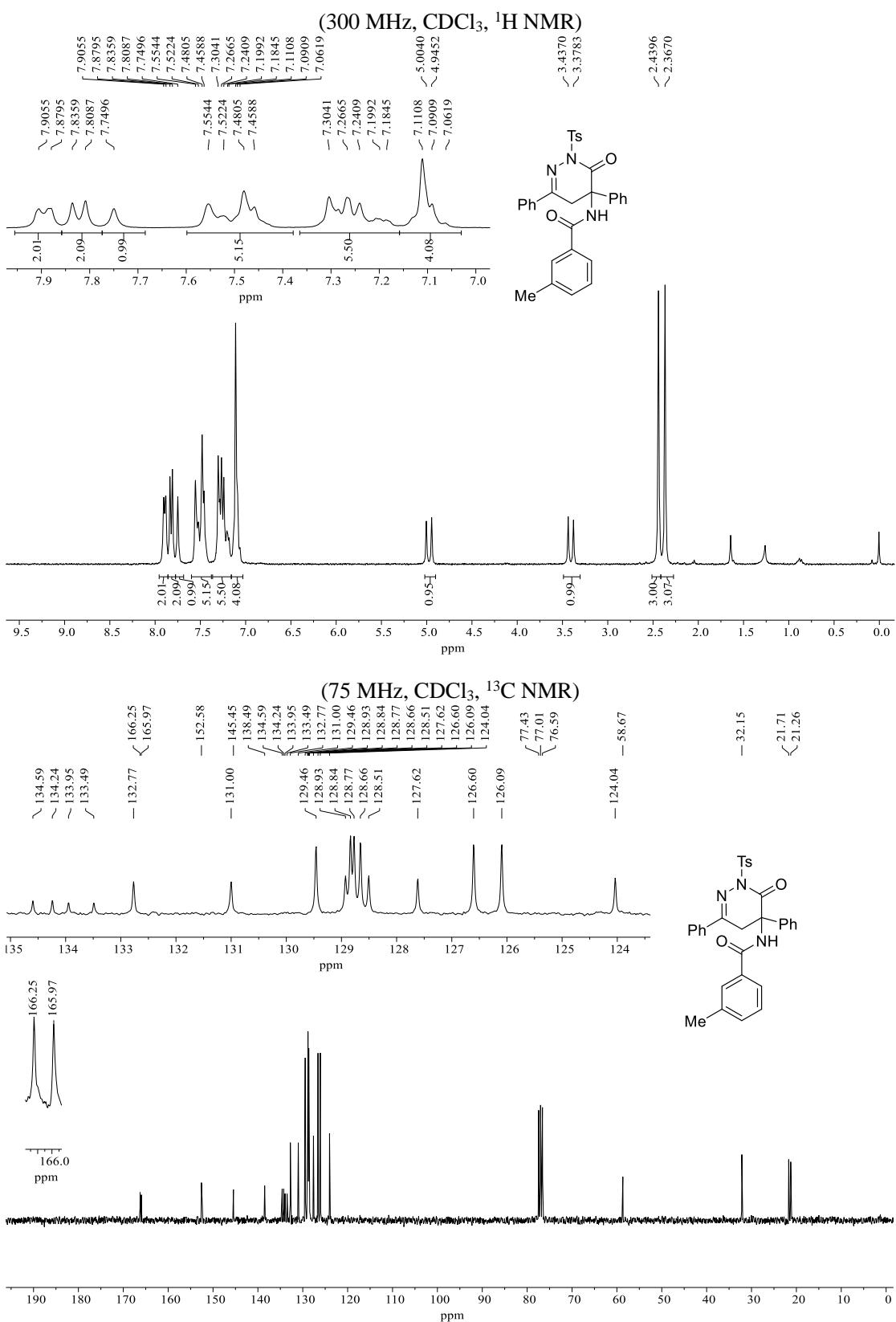
¹H NMR and ¹³C NMR spectra of 3p



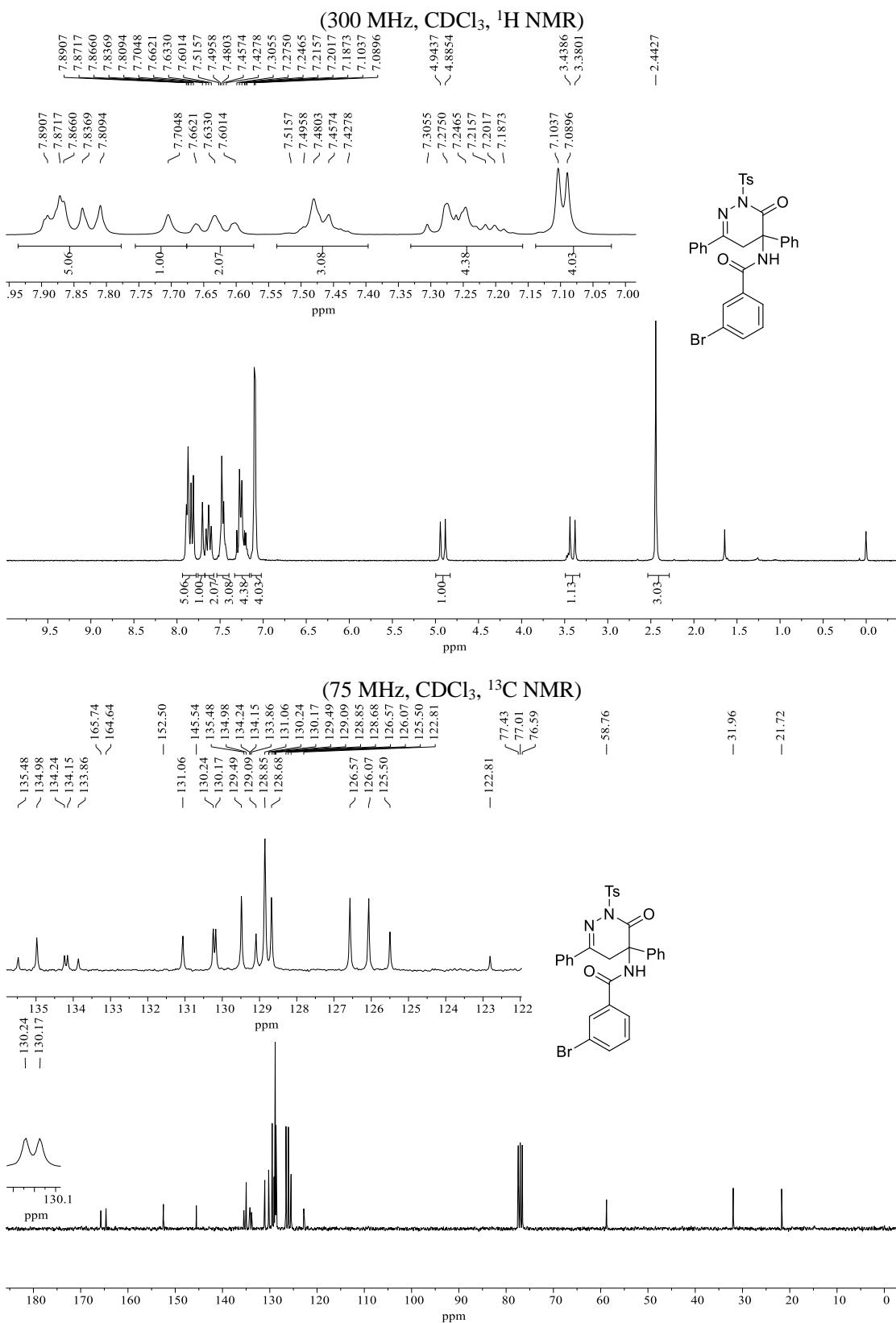
¹H NMR and ¹³C NMR spectra of **3q**



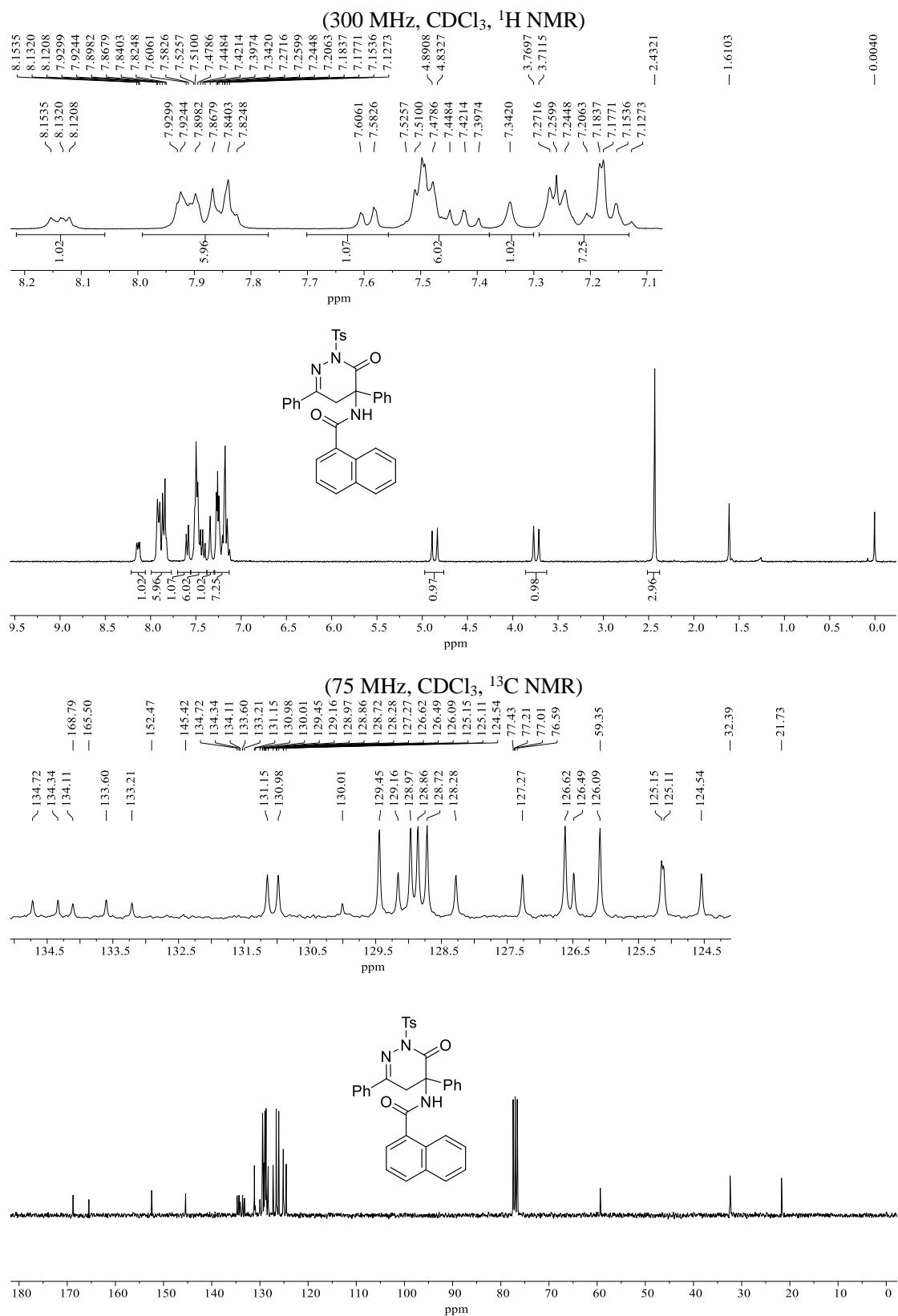
¹H NMR and ¹³C NMR spectra of **3r**



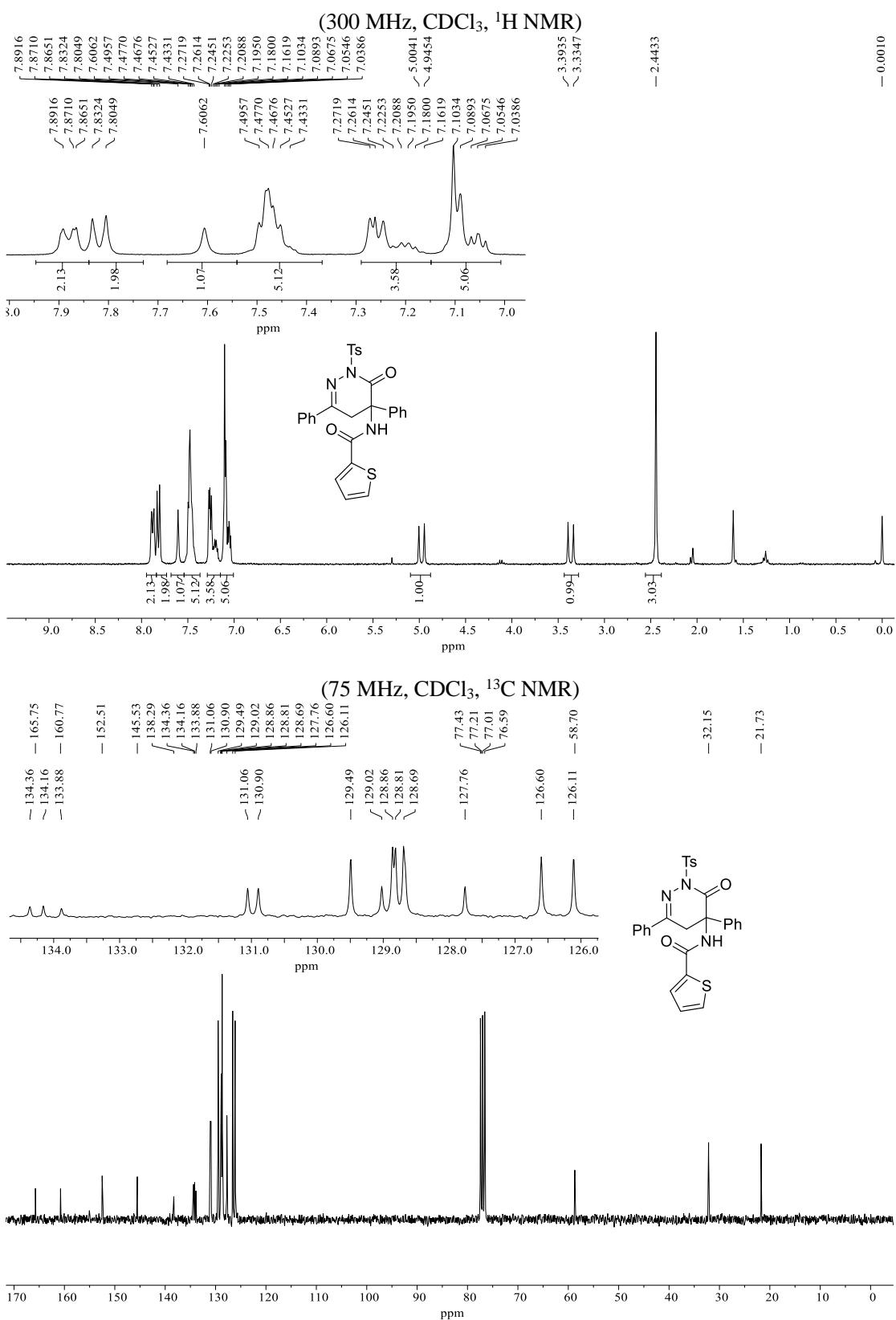
¹H NMR and ¹³C NMR spectra of **3s**



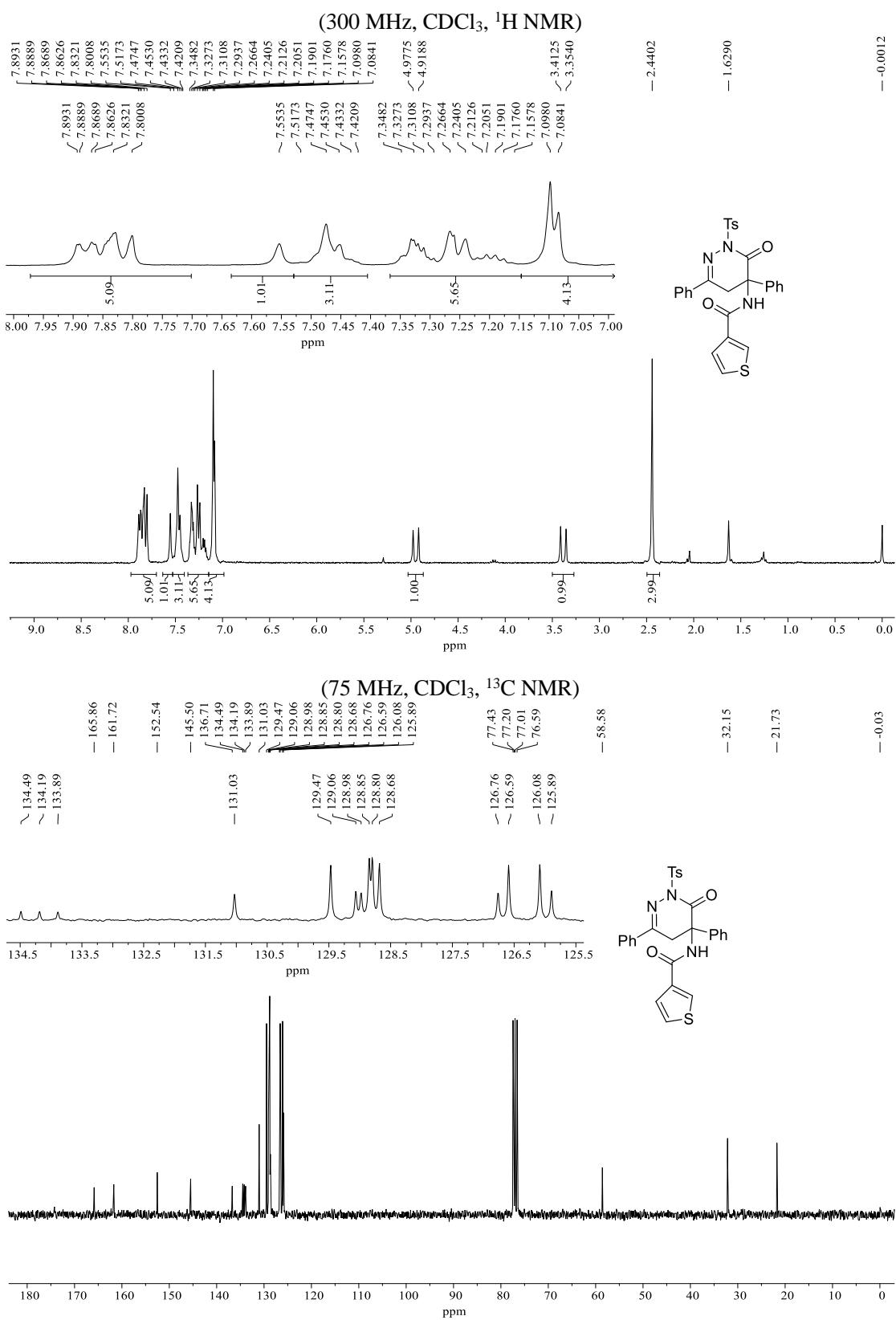
¹H NMR and ¹³C NMR spectra of **3t**



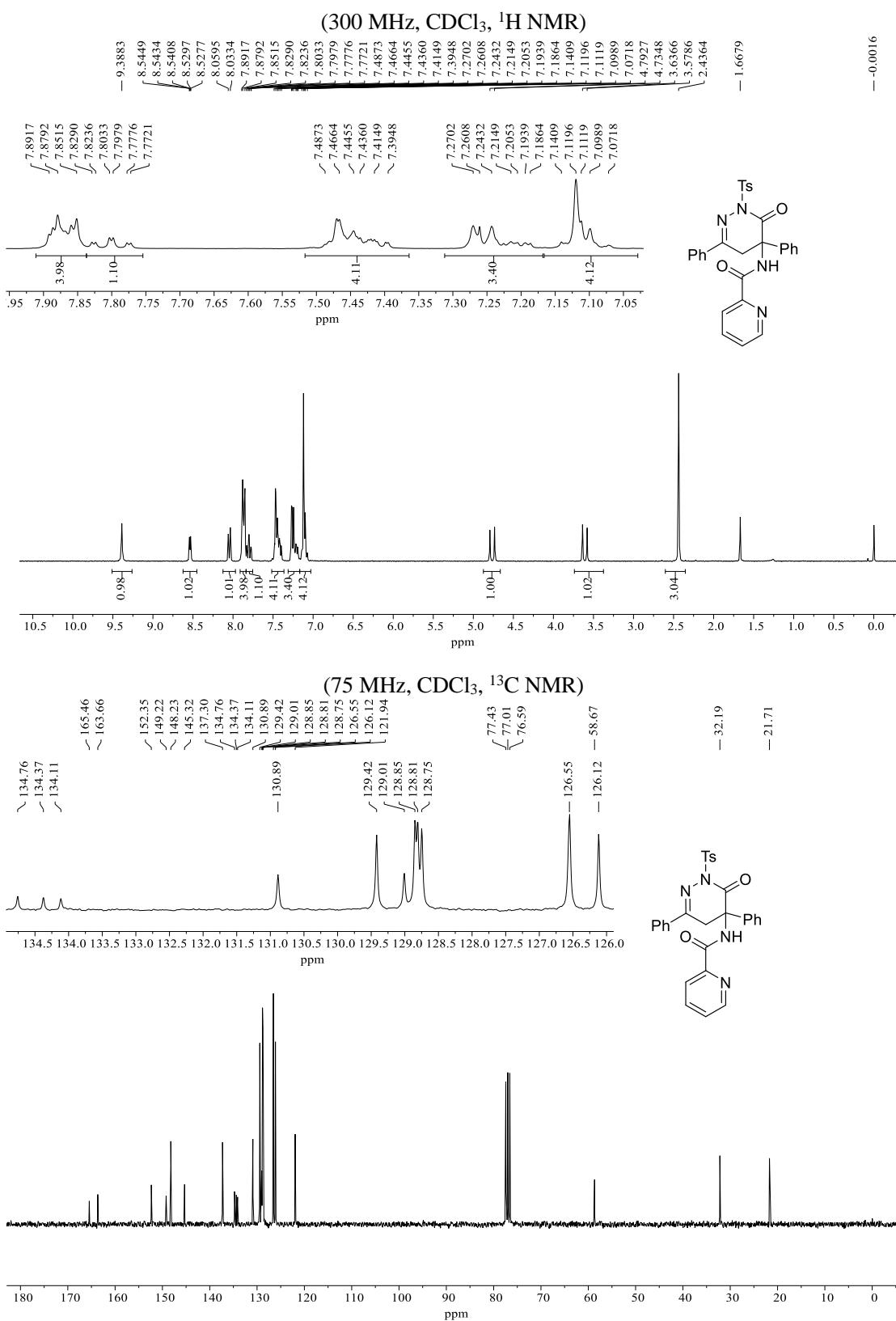
¹H NMR and ¹³C NMR spectra of 3u



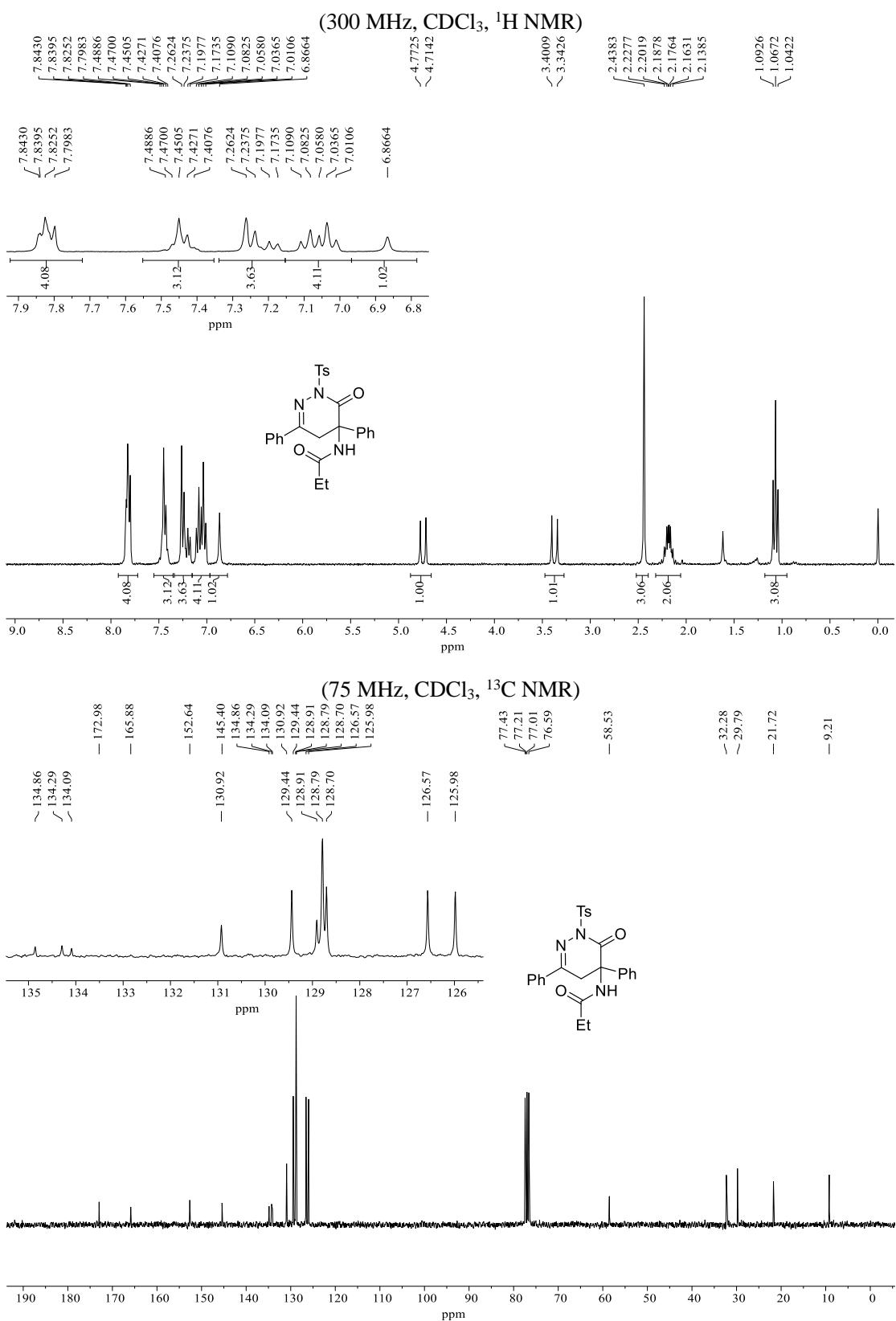
¹H NMR and ¹³C NMR spectra of **3v**



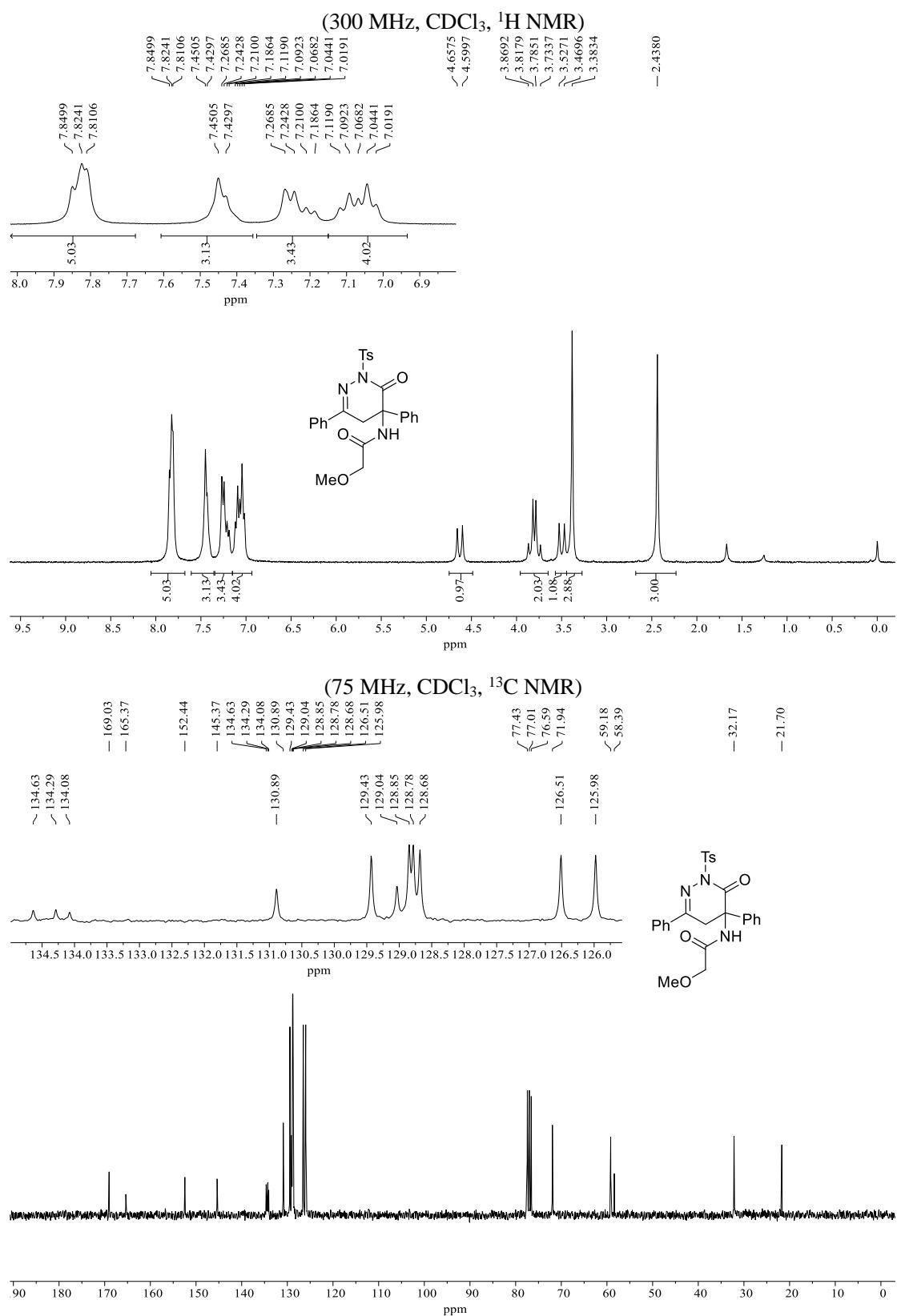
¹H NMR and ¹³C NMR spectra of 3w



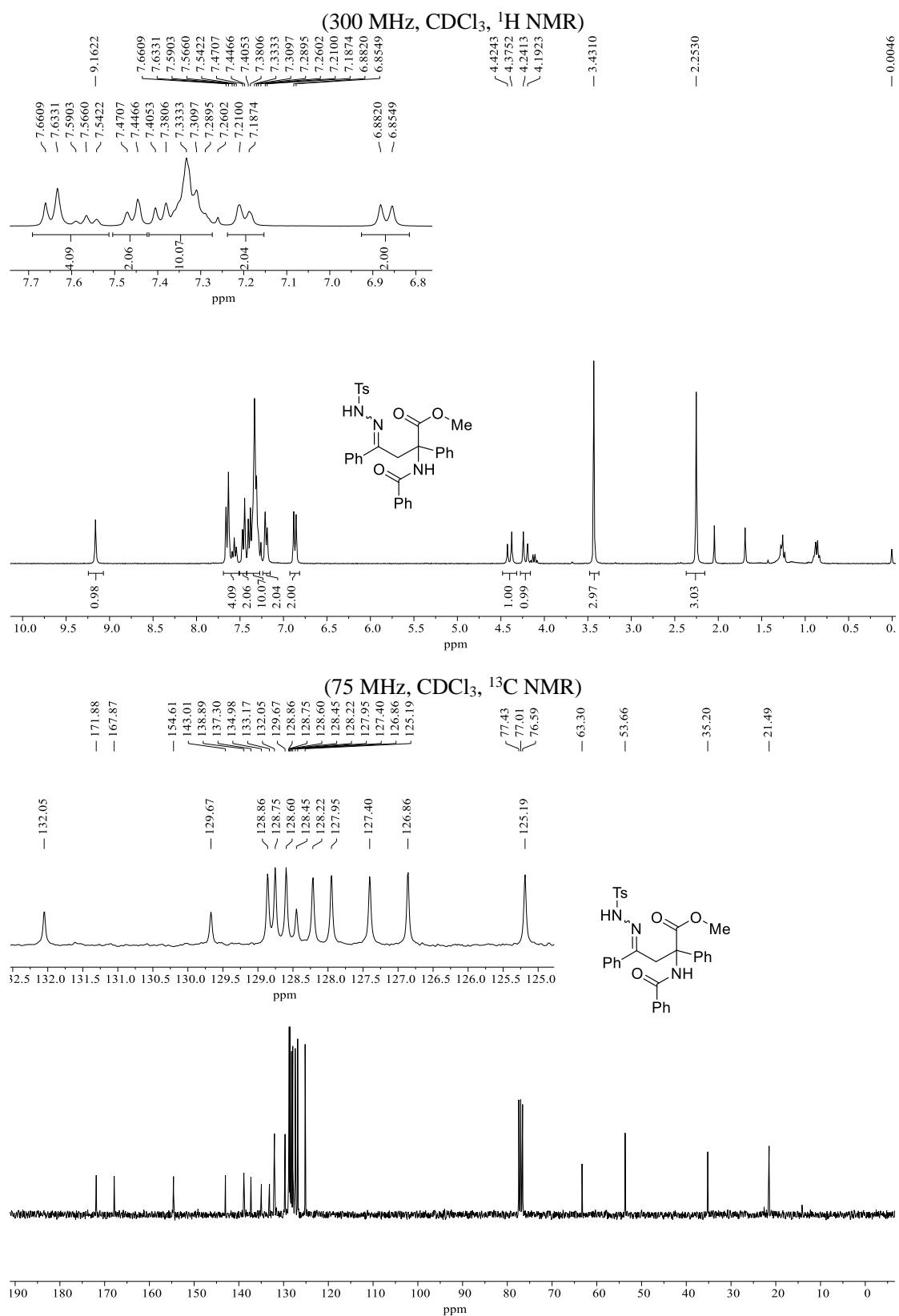
¹H NMR and ¹³C NMR spectra of **3x**



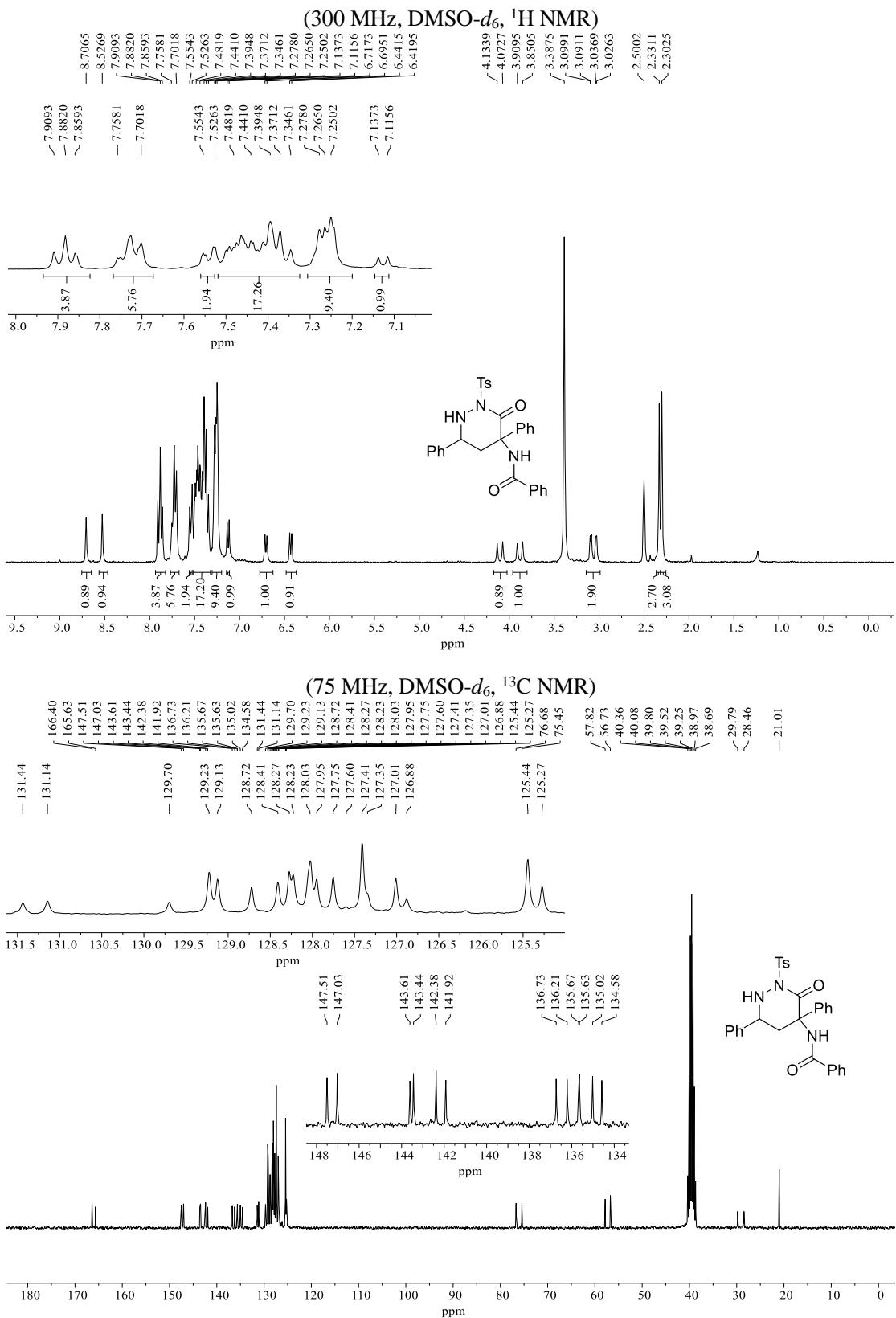
¹H NMR and ¹³C NMR spectra of **3y**



¹H NMR and ¹³C NMR spectra of **5**



¹H NMR and ¹³C NMR spectra of **6**



¹H NMR and ¹³C NMR spectra of 7

