Gold-catalyzed facile intramolecular rearrangement and cyclization sequence for synthesis of 2,5-dihydrofurans †

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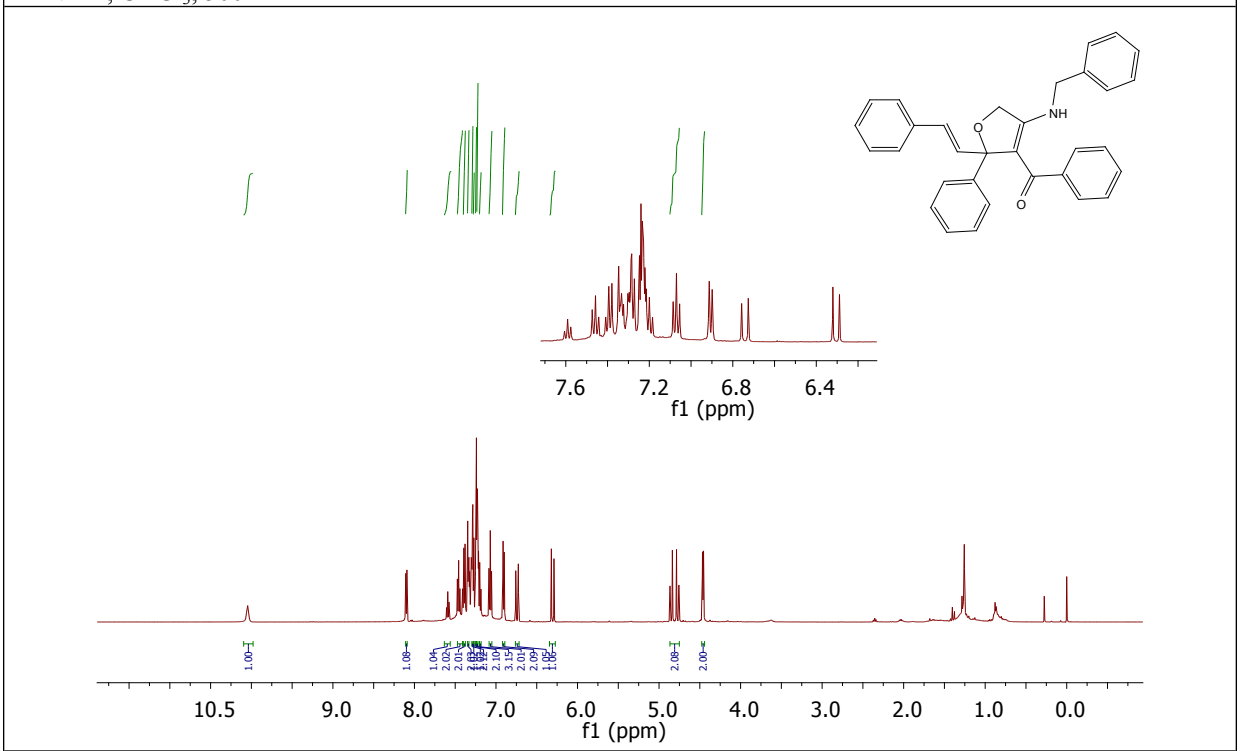
Contents:

Electronic Supplementary Material (ESI) for Chemical Communications

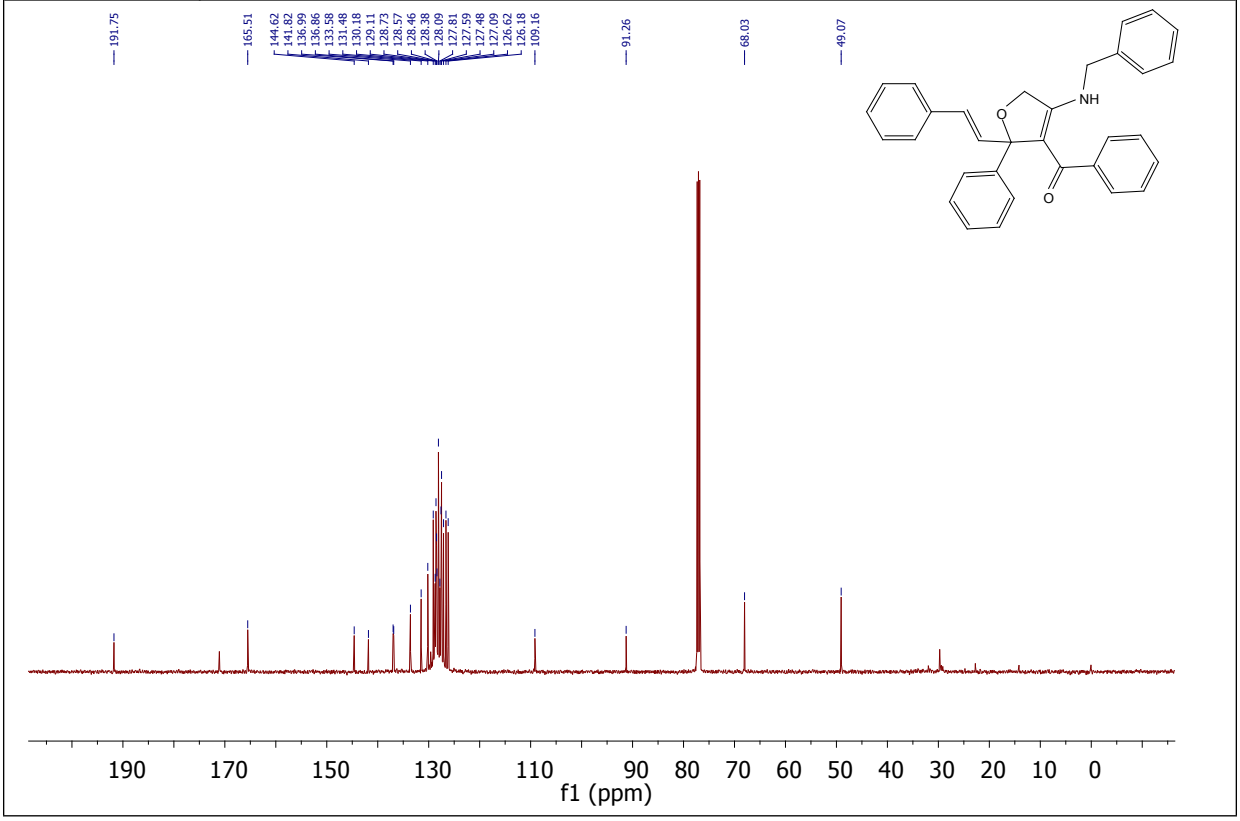
1.1 Copies of ^{1}H and ^{13}C NMR spectra (2a- 2v)

(*E*)-(4-(Benzylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3-yl)(phenyl)methanone: **2a**

^{1}HNMR, $CDCl_3$, 500MHz

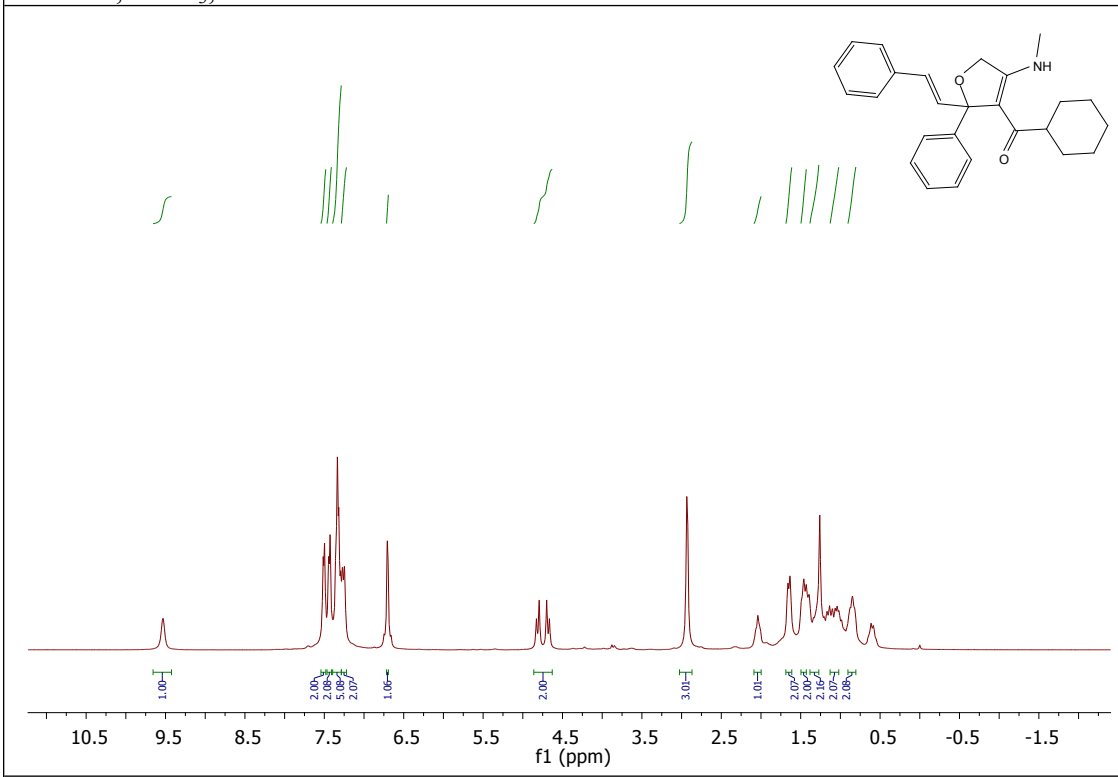


^{13}CNMR, $CDCl_3$, 125MHz

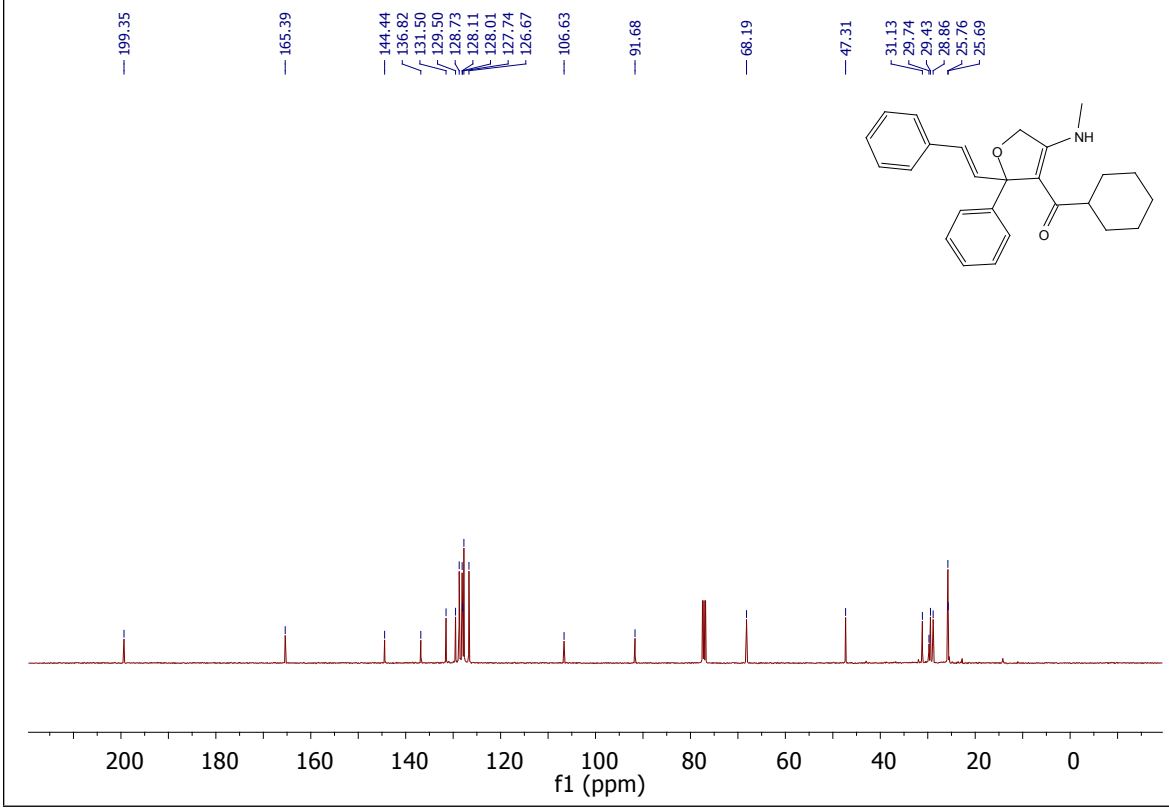


(*E*)-Cyclohexyl(4-(methylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3-yl)methanone: **2b**

^{1}HNMR, $CDCl_3$, 400MHz

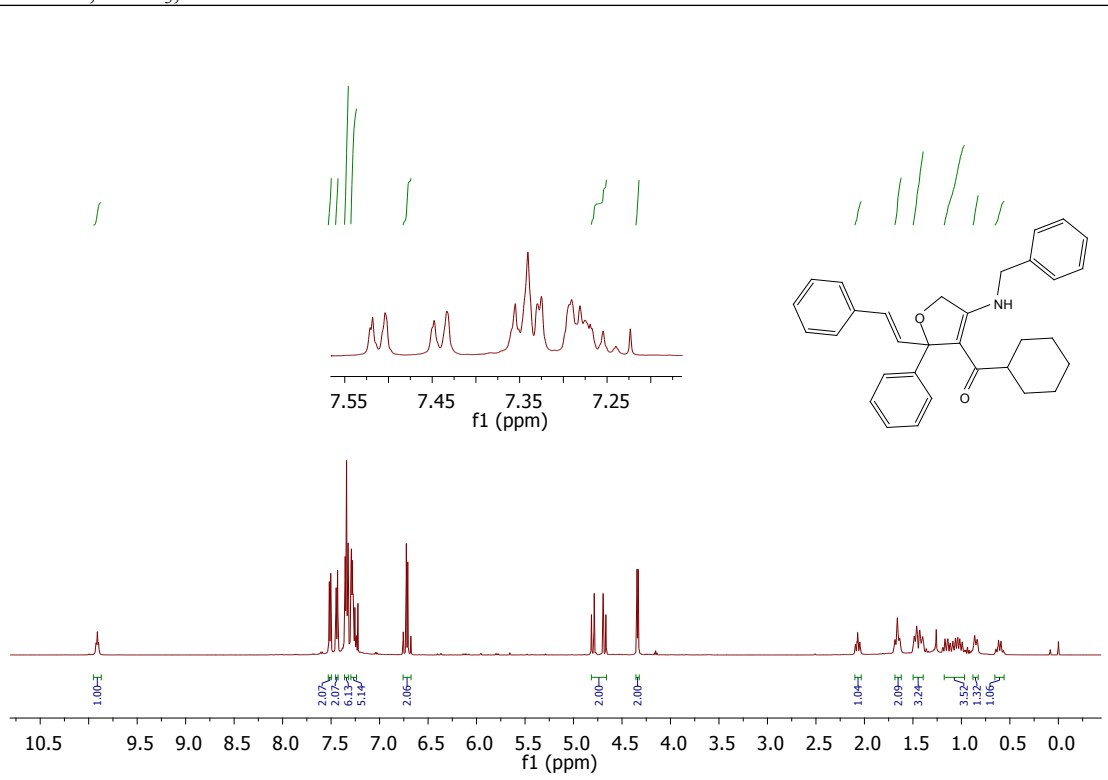


^{13}CNMR, $CDCl_3$, 100MHz

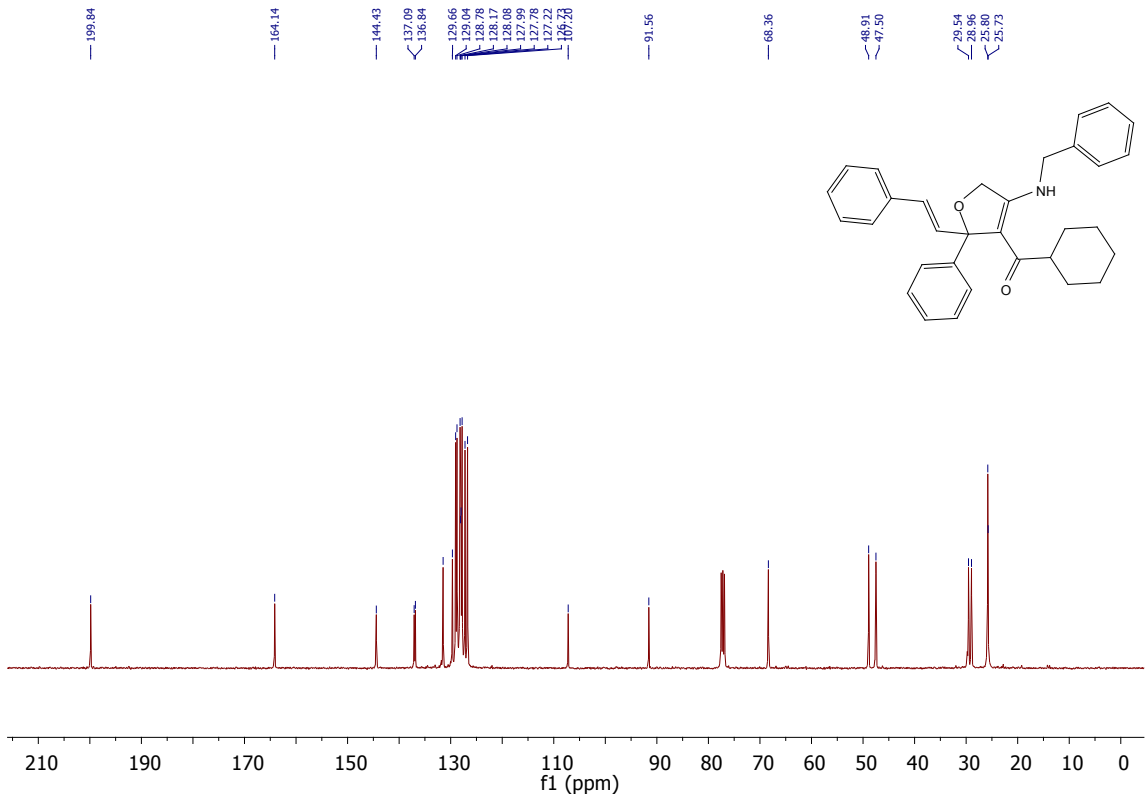


(*E*)-(4-(Benzylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3-yl)(cyclohexyl)methanone: **2c**

^{1}HNMR, $CDCl_3$, 500MHz

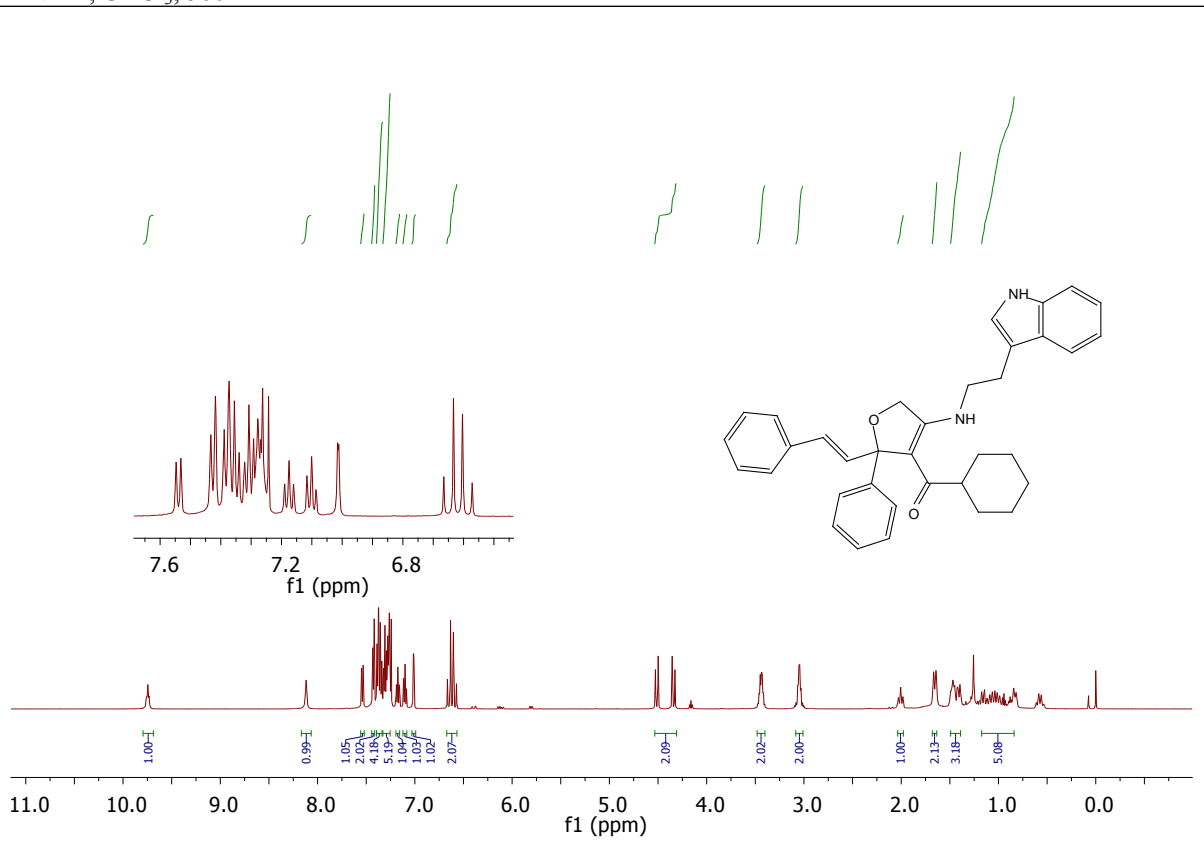


^{13}CNMR, $CDCl_3$, 100MHz

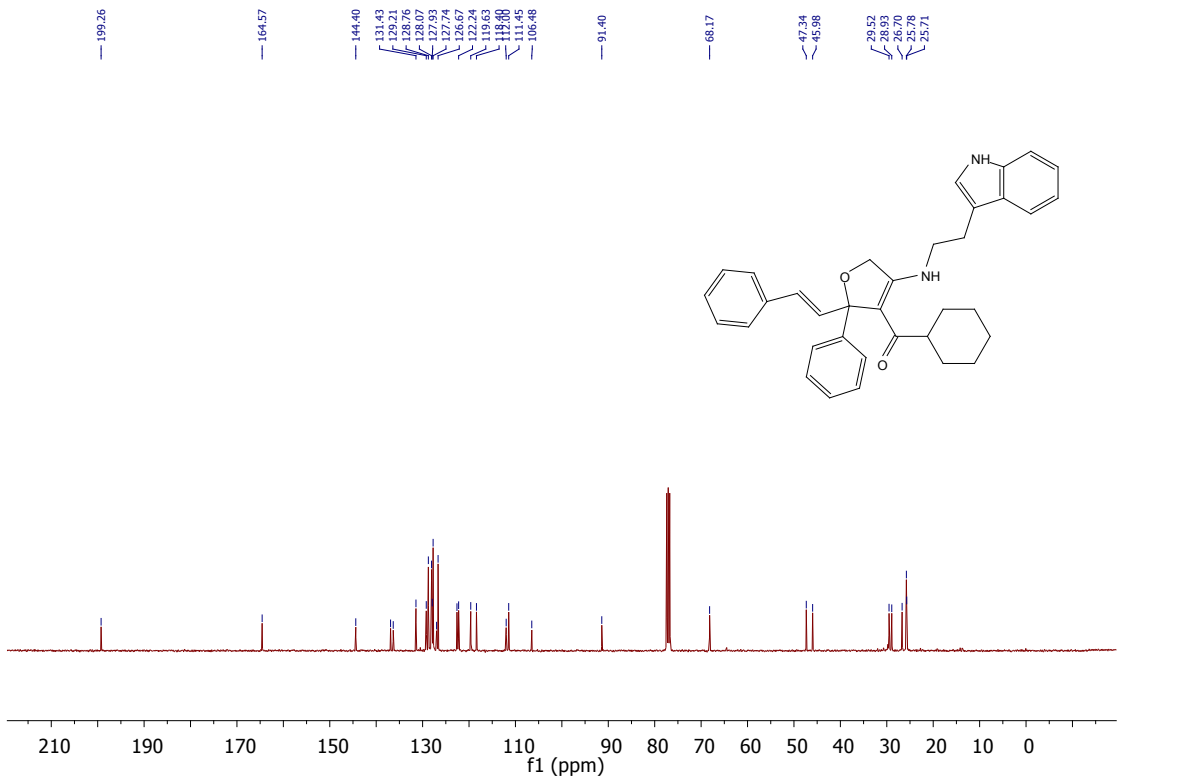


(*E*)-(4-(2-(1H-Indol-2-yl)ethylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3 yl)(cyclohexyl)methanone: **2d**

^{1}HNMR, $CDCl_3$, 500MHz

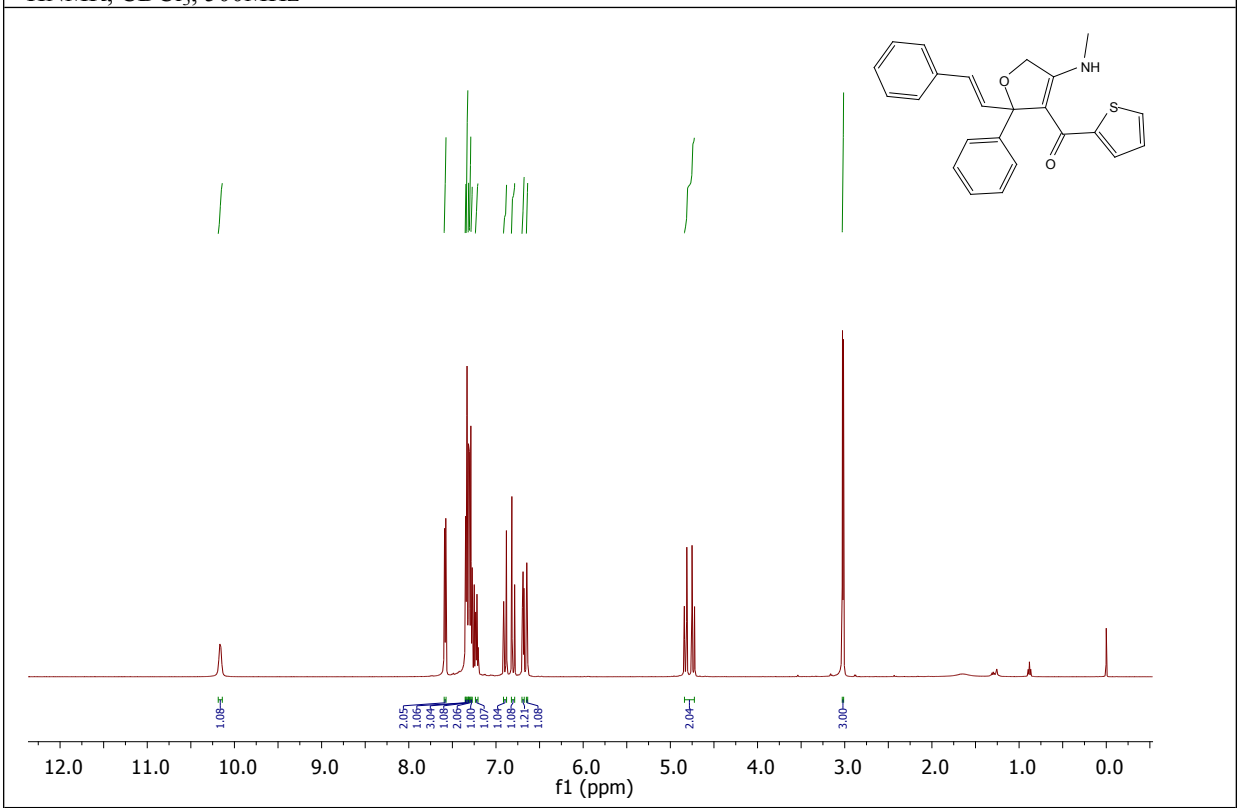


^{13}CNMR, $CDCl_3$, 100MHz

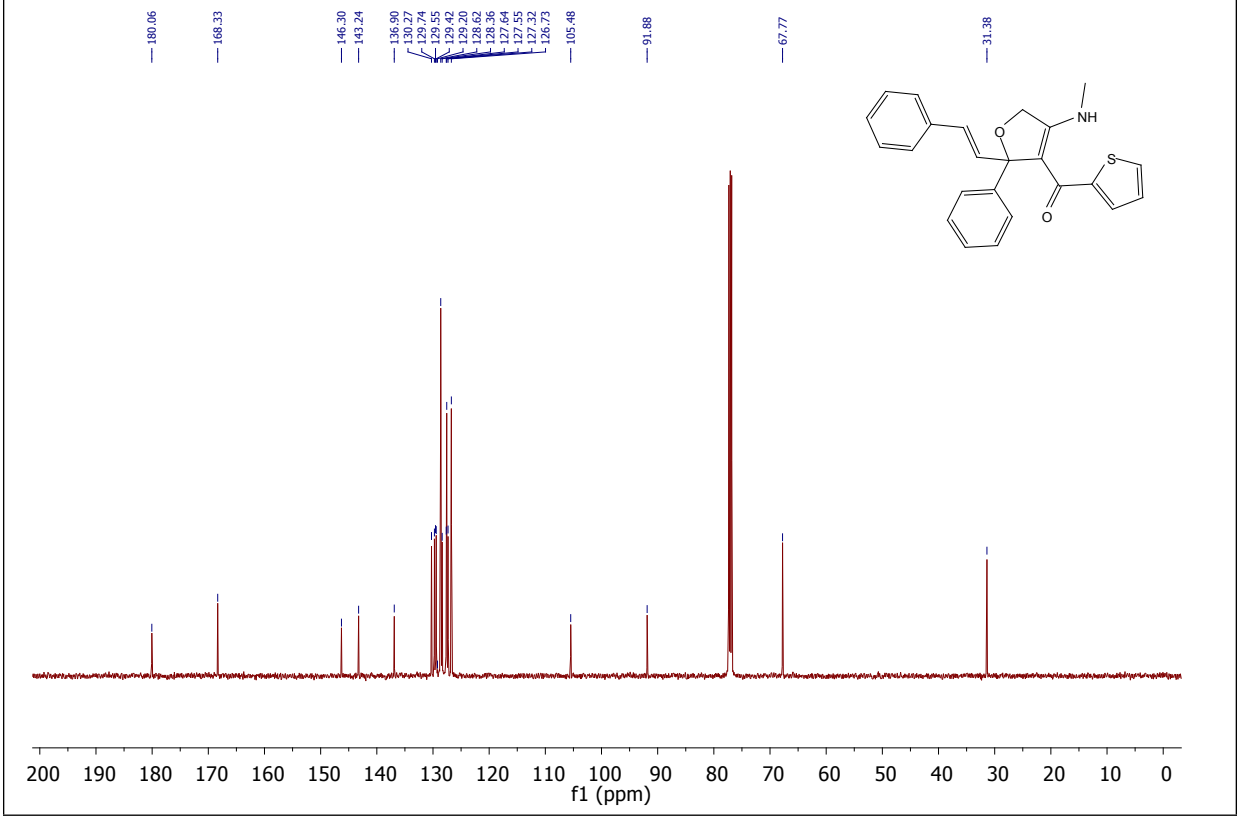


(*E*)-(4-(Methylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3-yl)(thiophen-2-yl)methanone: **2e**

^{1}HNMR, $CDCl_3$, 500MHz

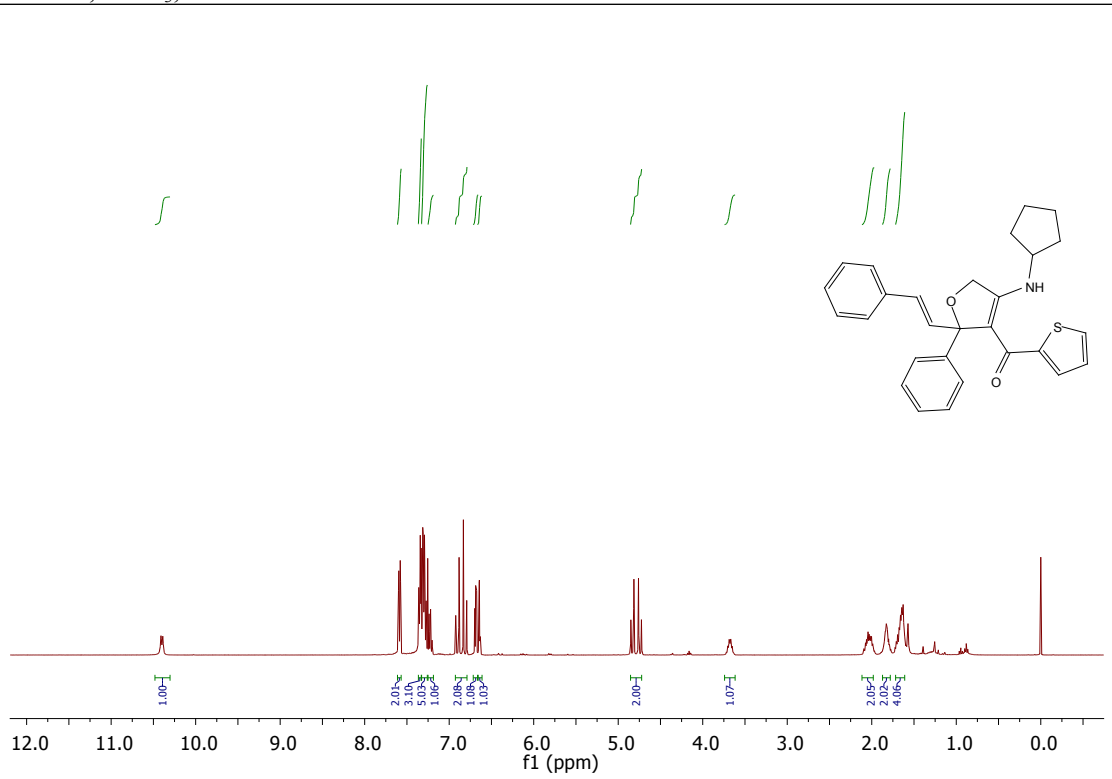


^{13}CNMR, $CDCl_3$, 125MHz

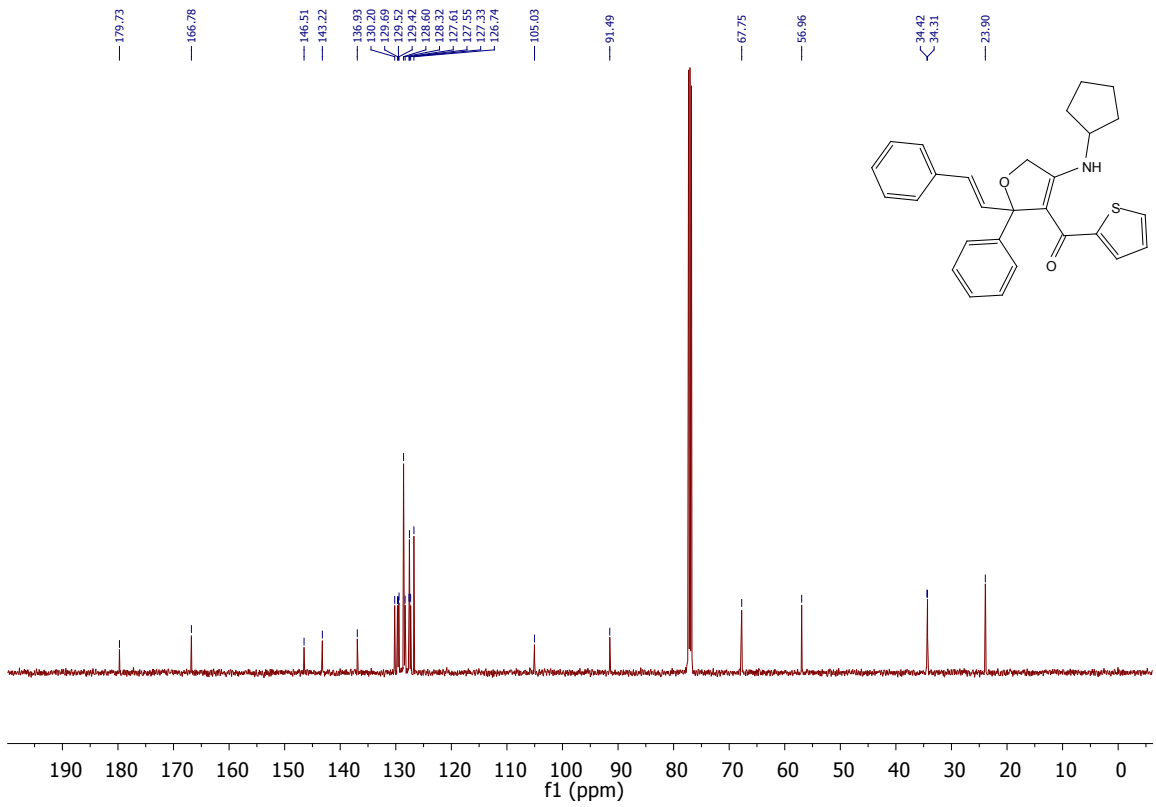


(*E*)-(4-(Cyclopentylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3-yl)(thiophen-2-yl)methanone**: 2f**

^{1}HNMR, $CDCl_3$, 400MHz

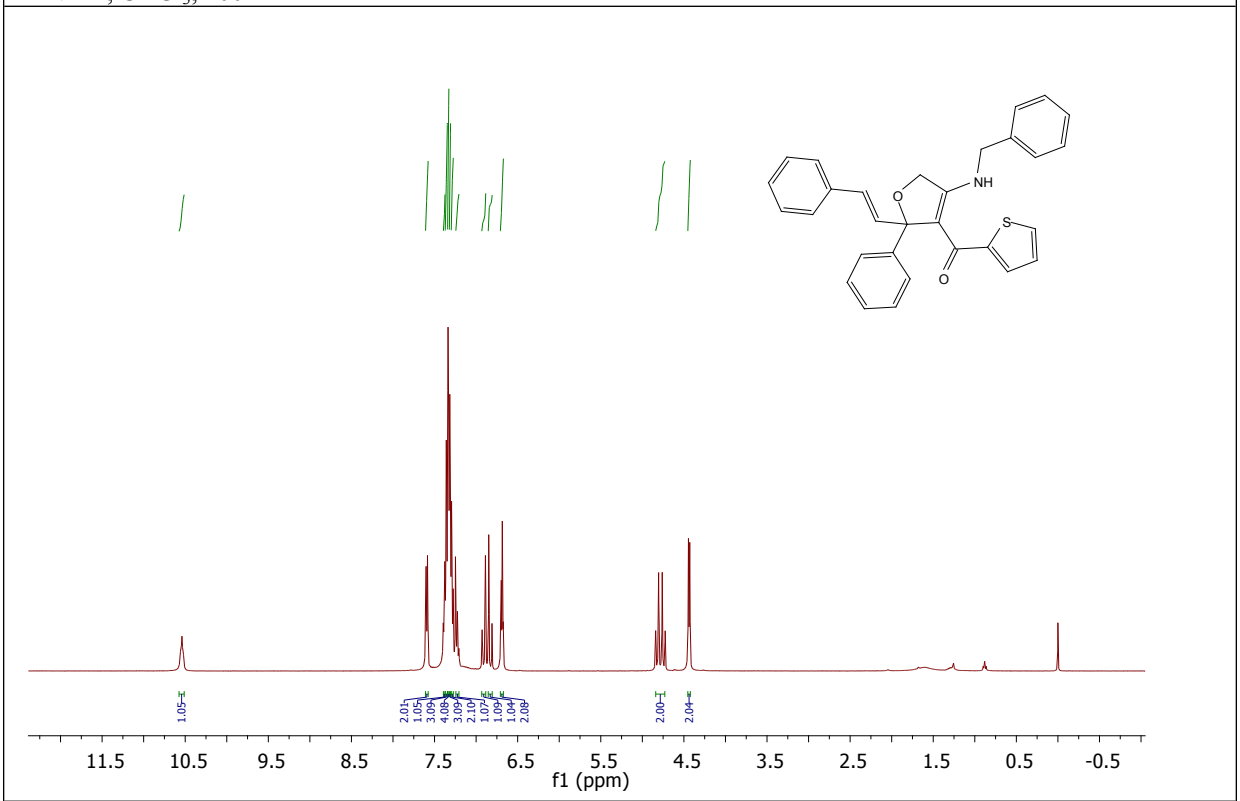


^{13}CNMR, $CDCl_3$, 125MHz

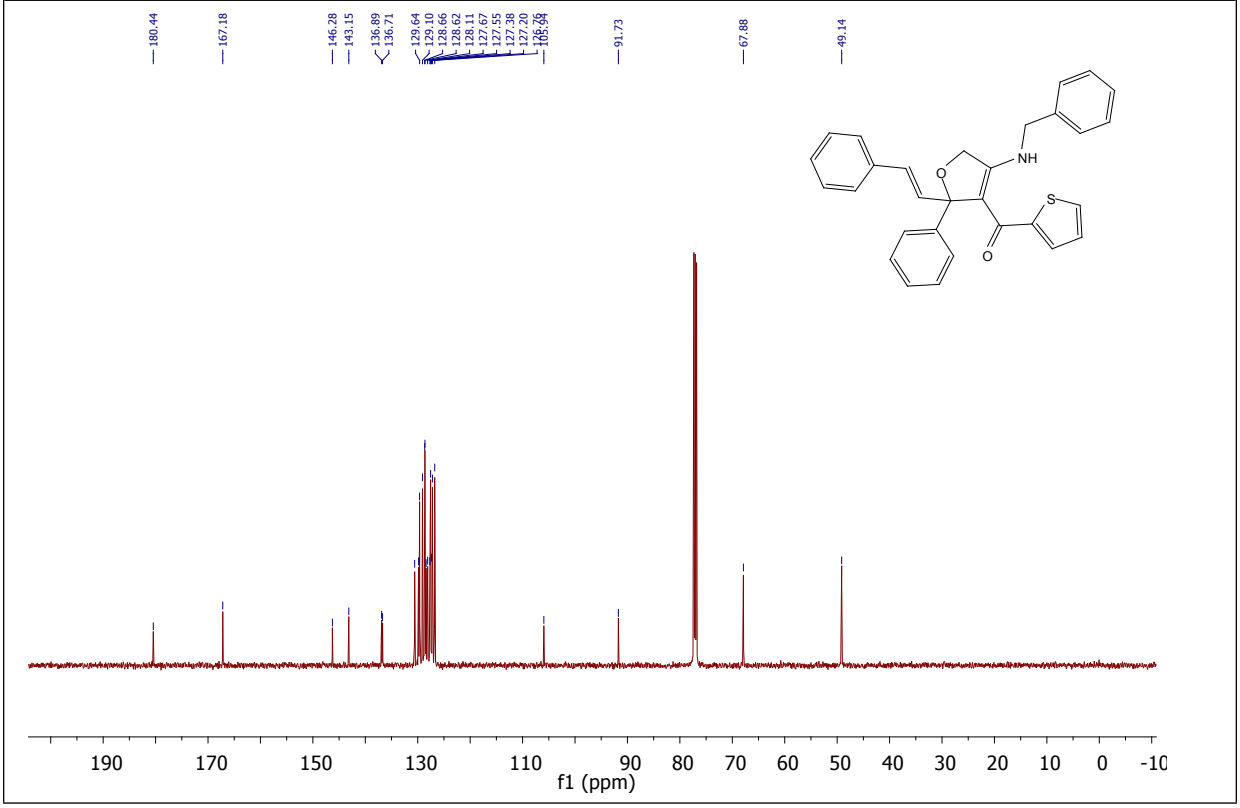


(*E*)-(4-(Benzylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3-yl)(thiophen-2-yl)methanone:**2g**

^{1}HNMR, $CDCl_3$, 400MHz

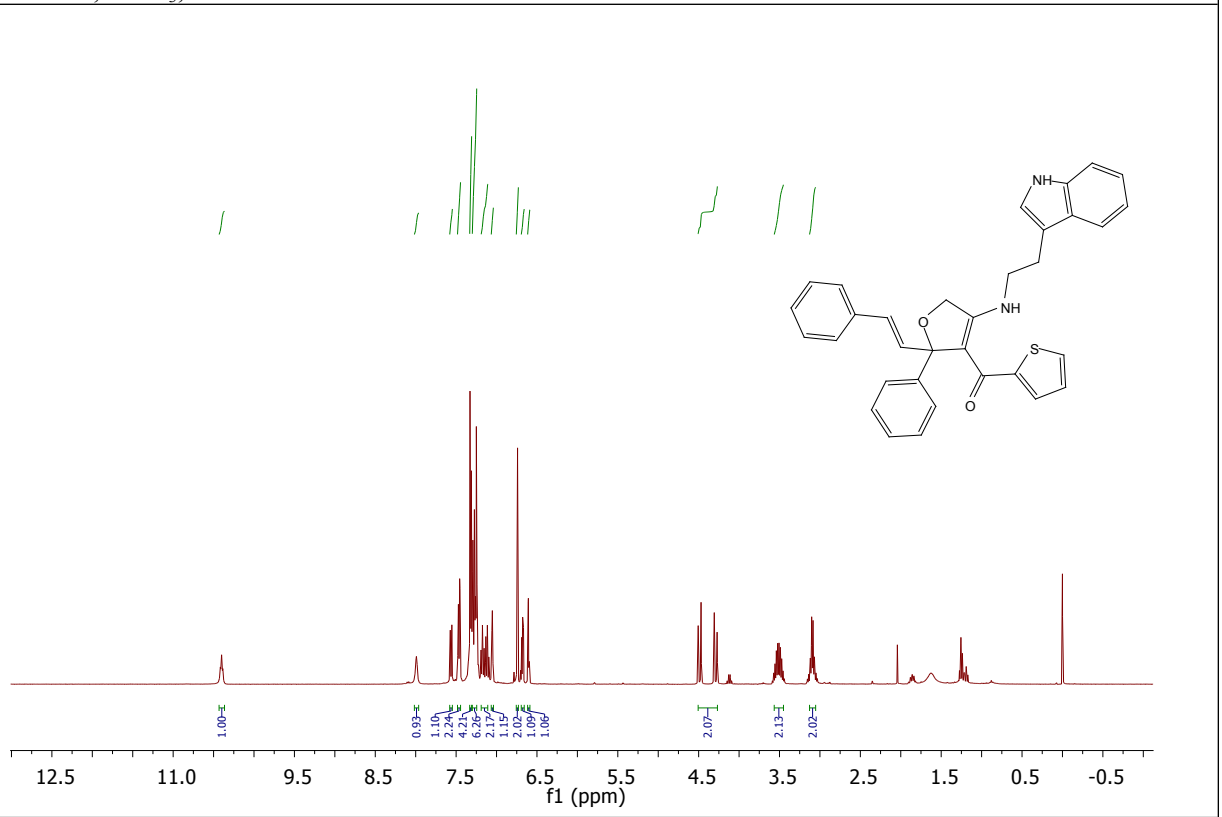


^{13}CNMR, $CDCl_3$, 125MHz

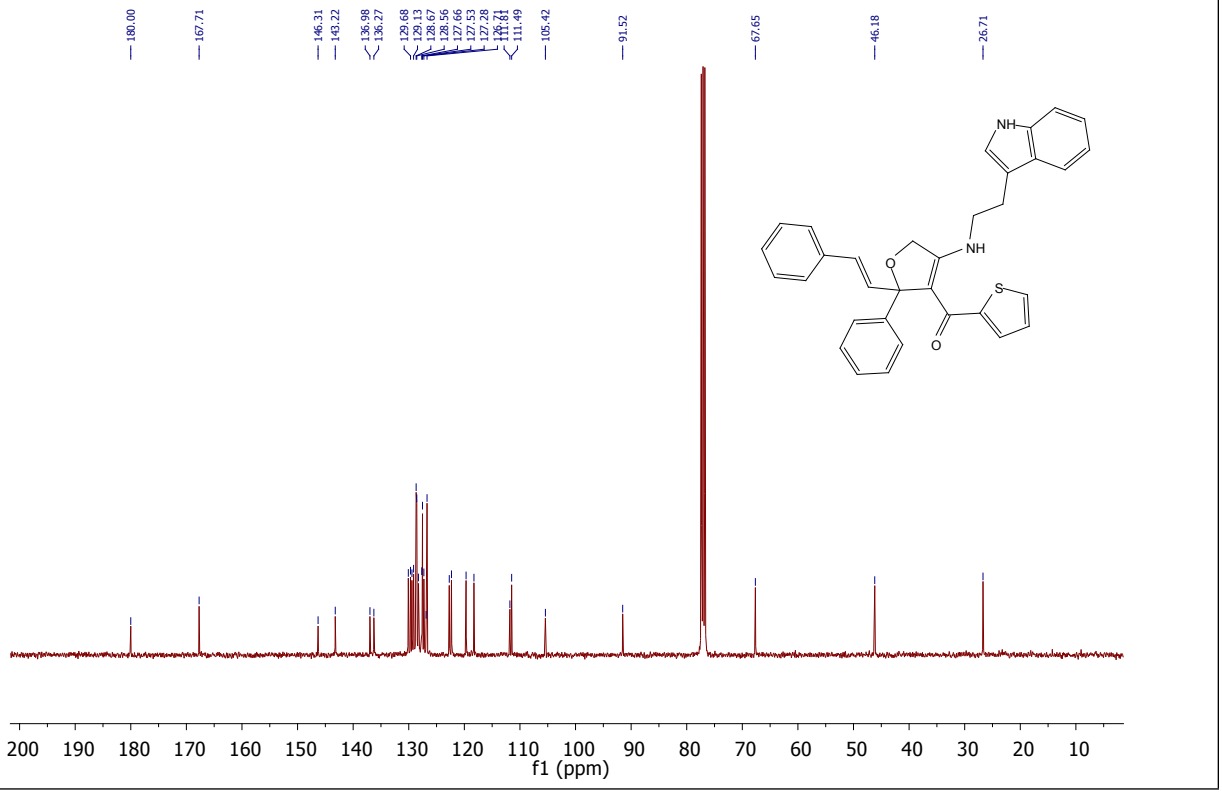


(*E*)-(4-(2-(*1H*-Indol-3-yl)ethylamino)-2-phenyl-2-styryl-2,5-dihydrofuran-3-yl)(thiophen-2-yl)methanone: **2h**

^{1}HNMR, $CDCl_3$, 400MHz

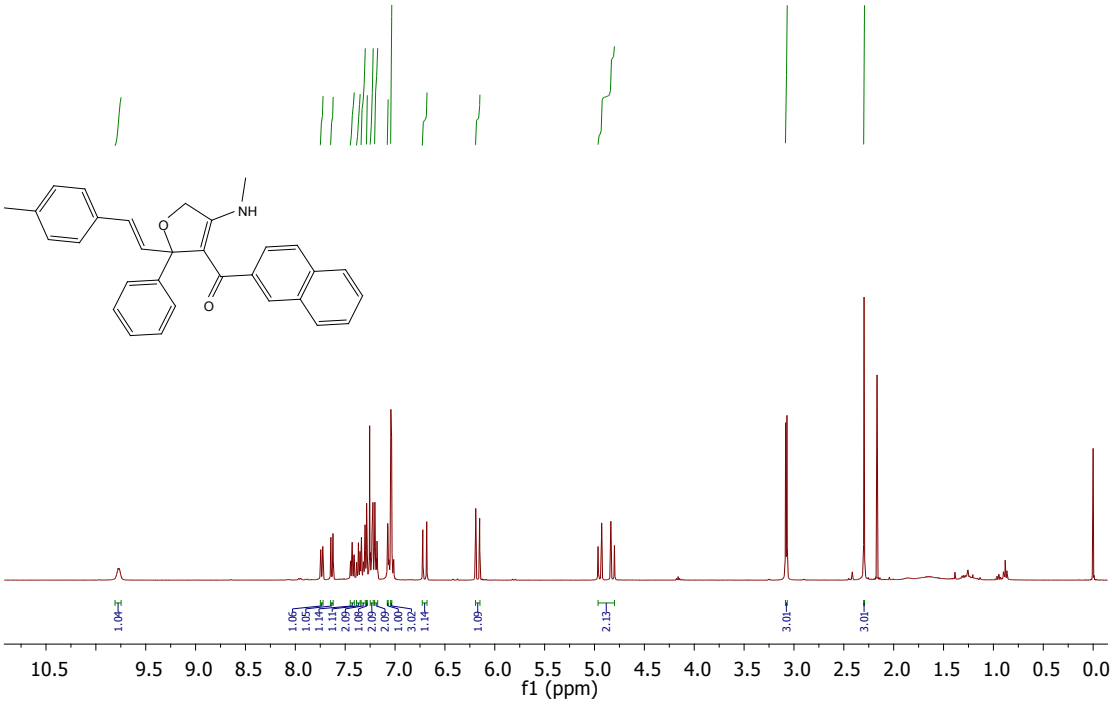


^{13}CNMR, $CDCl_3$, 100MHz

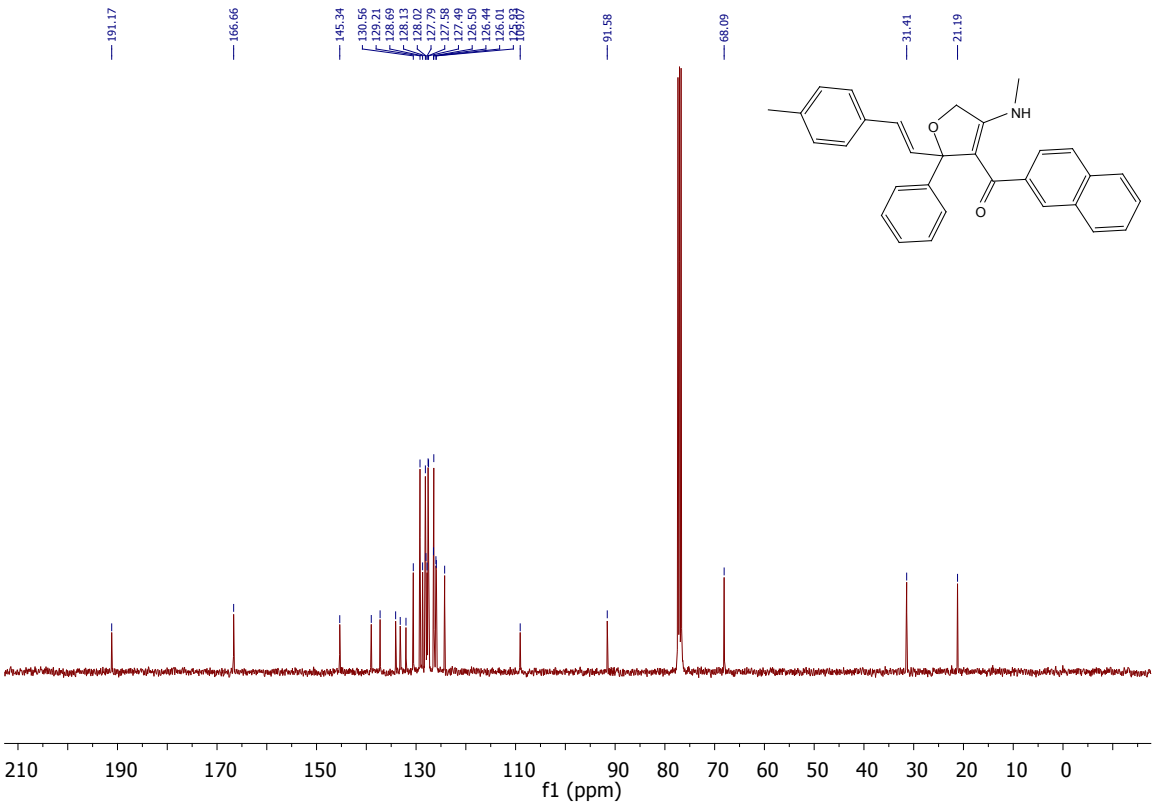


(*E*)-(4-(Methylamino)-2-styryl-2-*p*-tolyl-2,5-dihydrofuran-3-yl)(naphthalen-2-yl)methanone: **2i**

^{1}HNMR, $CDCl_3$, 400MHz

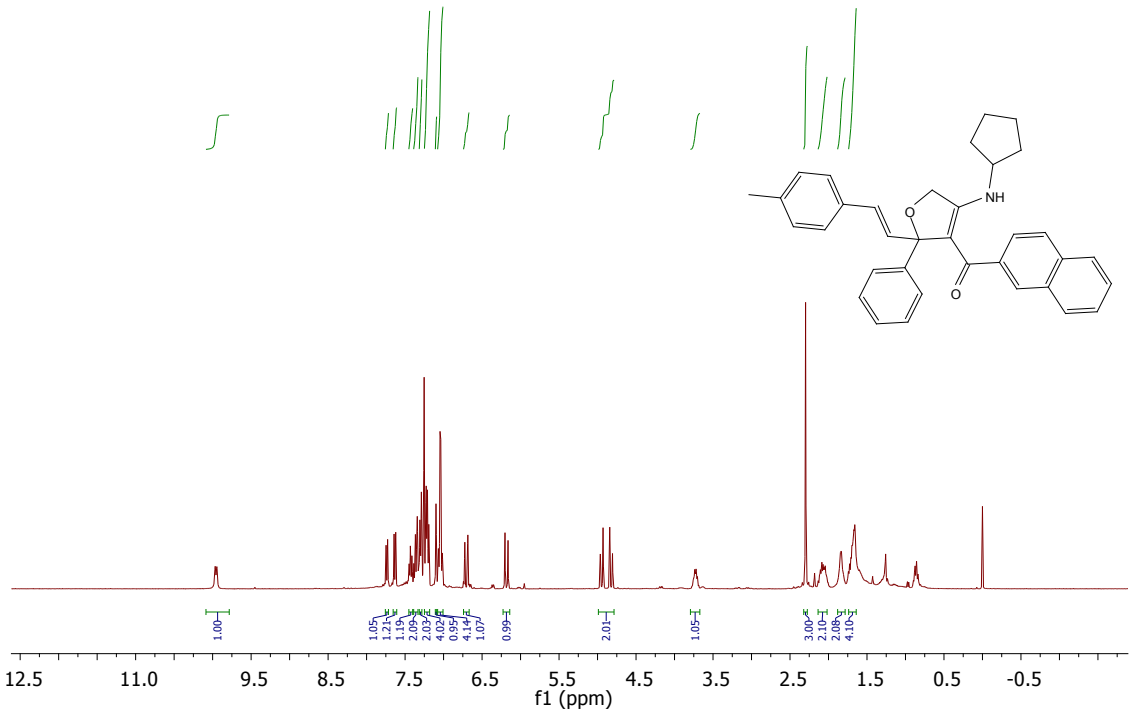


^{13}CNMR, $CDCl_3$, 100MHz

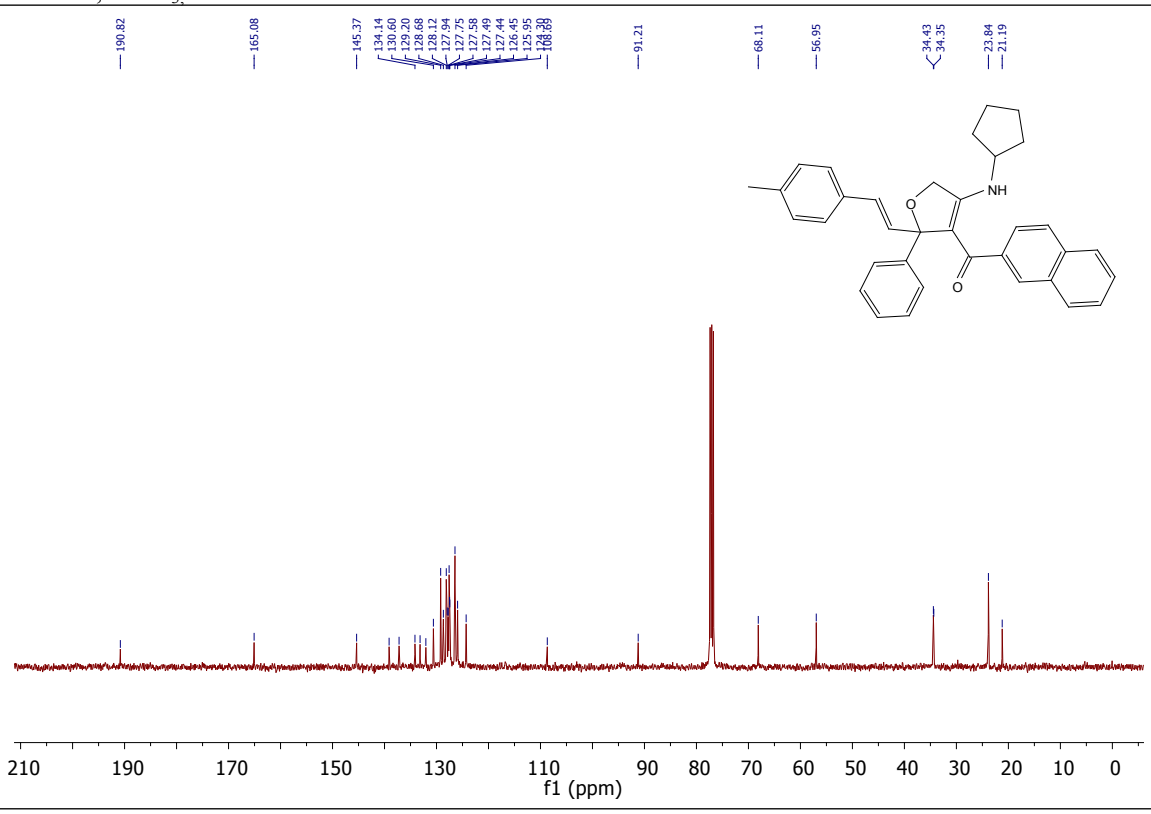


(*E*)-(4-(Cyclopentylamino)-2-styryl-2-(*p*-tolyl)-2,5-dihydrofuran-3-yl)(naphthalen-2-yl)methanone:**2j**

^{1}HNMR, $CDCl_3$, 400MHz

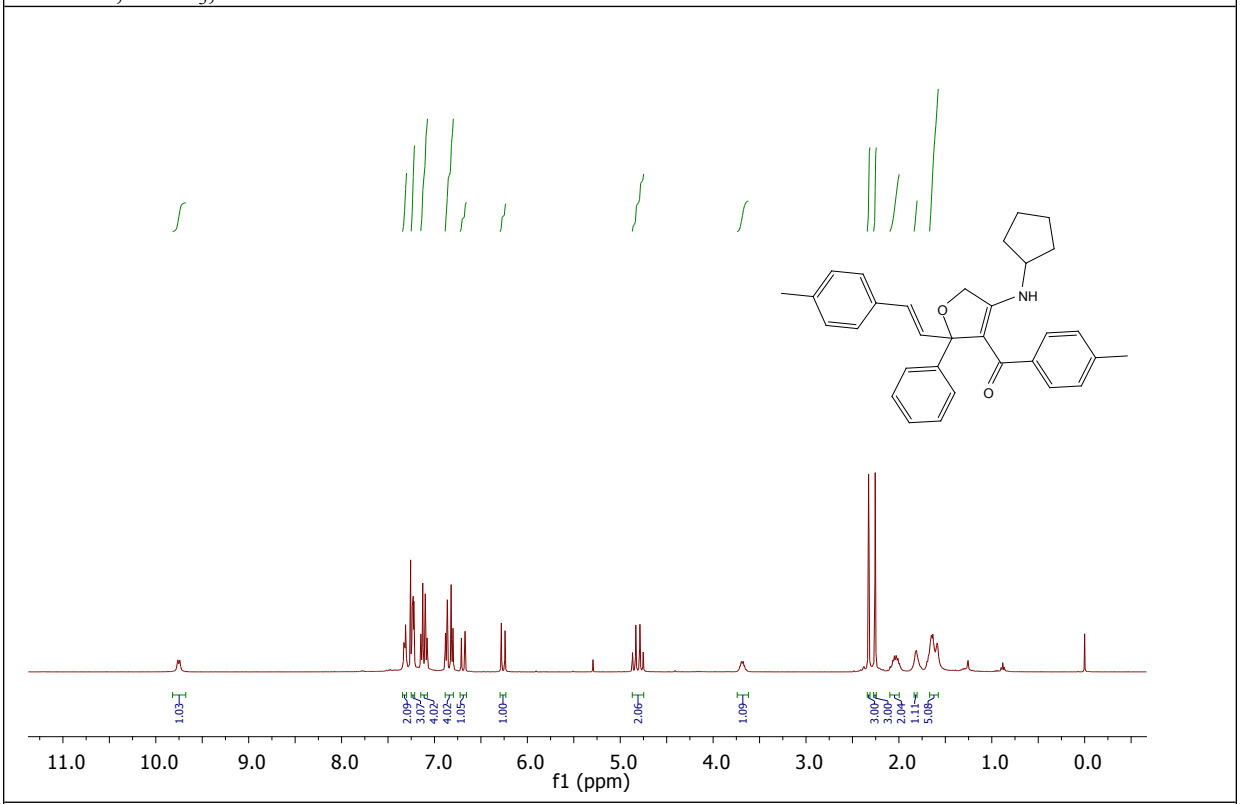


^{13}CNMR, $CDCl_3$, 100MHz

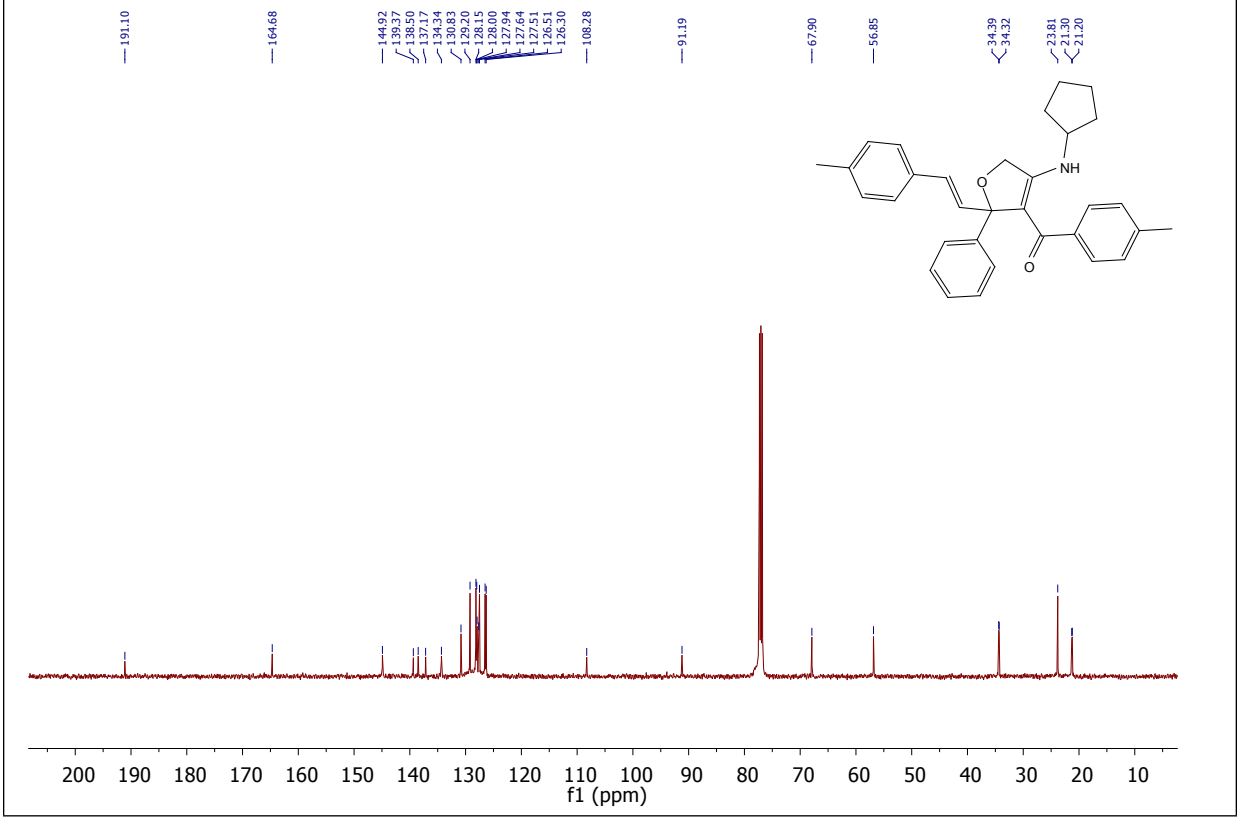


(*E*)-(4-(Cyclopentylamino)-2-styryl-2-(*p*-tolyl)-2,5-dihydrofuran-3-yl)(*p*-tolyl)methanone: **2k**

^{1}HNMR, $CDCl_3$, 400MHz

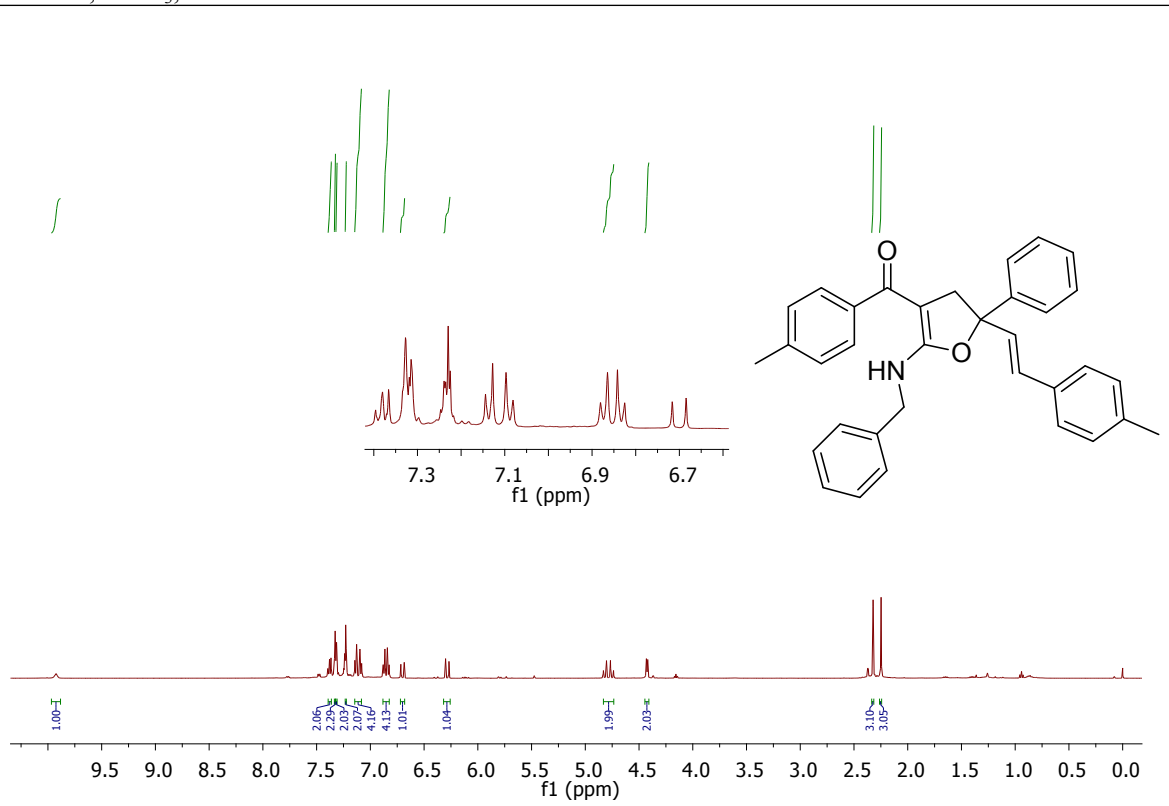


^{13}CNMR, $CDCl_3$, 100MHz

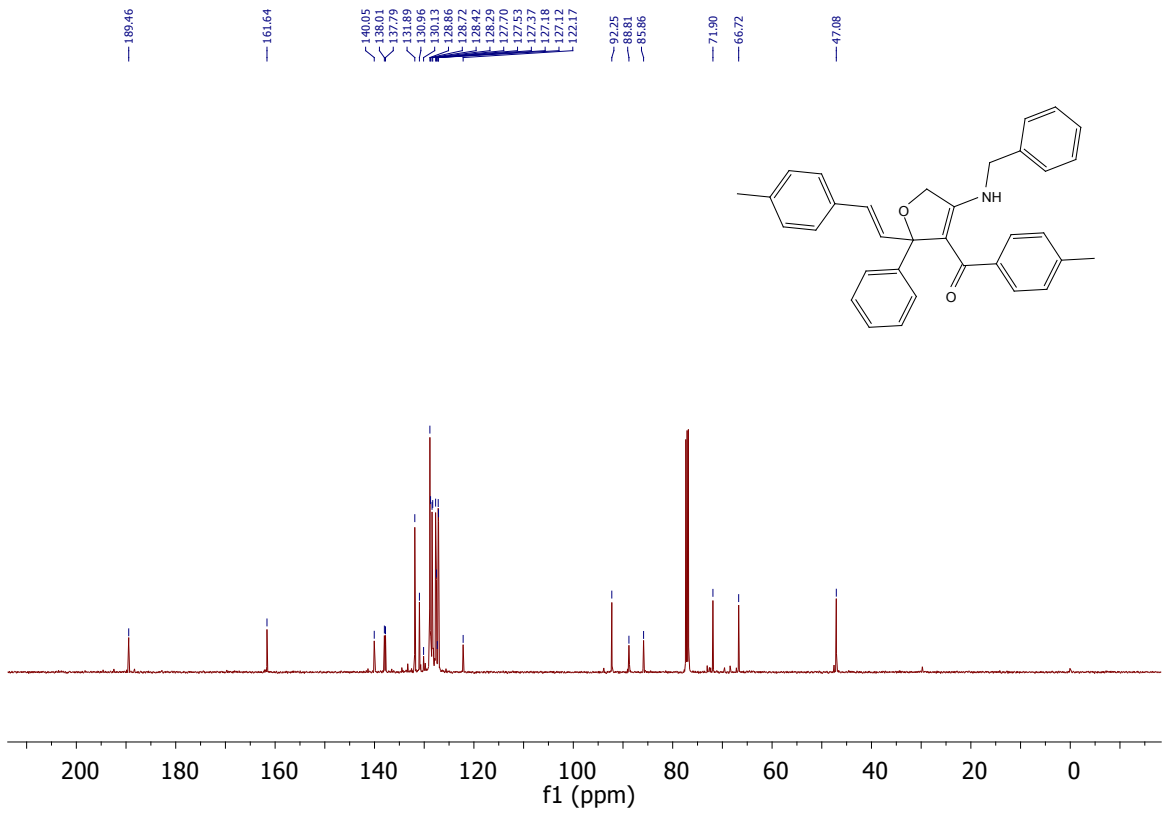


(*E*)-(4-(Benzylamino)-2-styryl-2-*p*-tolyl-2,5-dihydrofuran-3-yl)(*p*-tolyl)methanone: **2l**

^{1}HNMR, $CDCl_3$, 500MHz

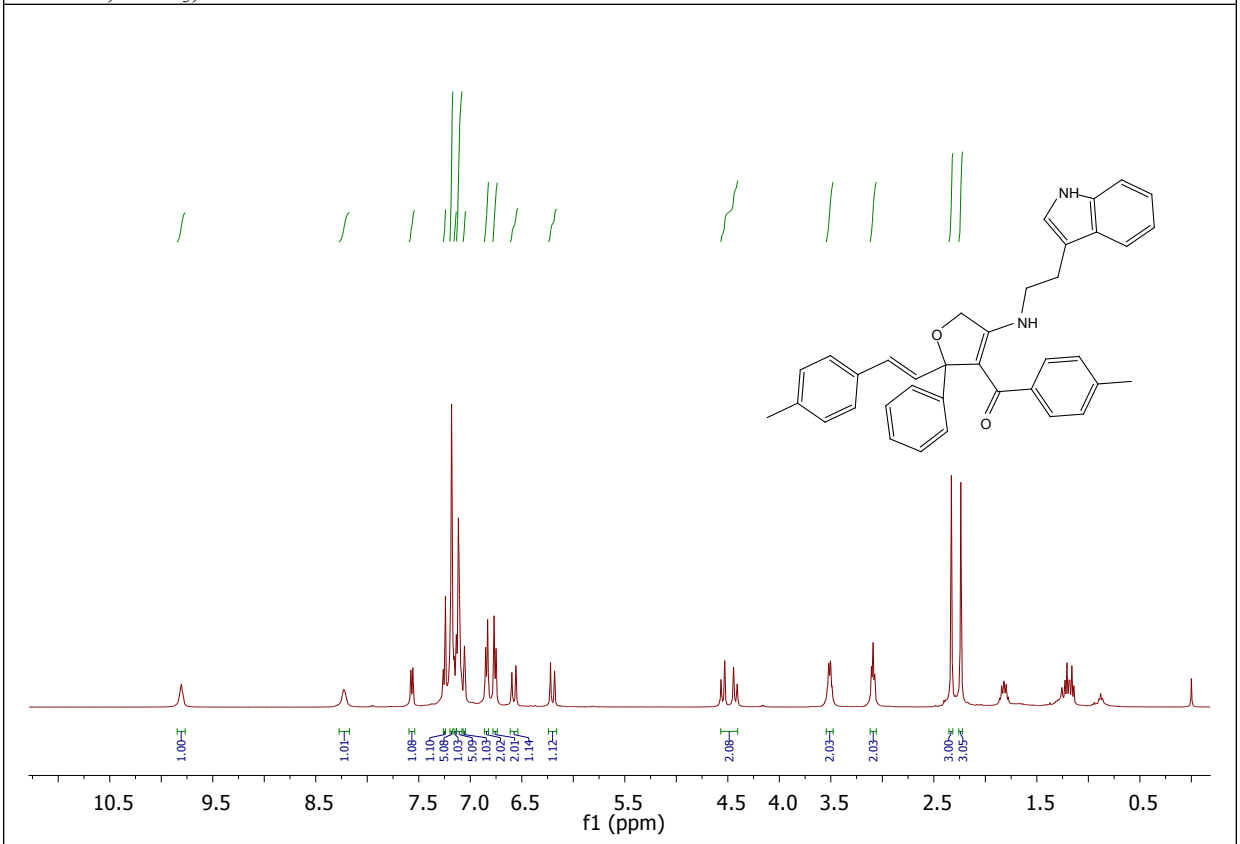


^{13}CNMR, $CDCl_3$, 125MHz

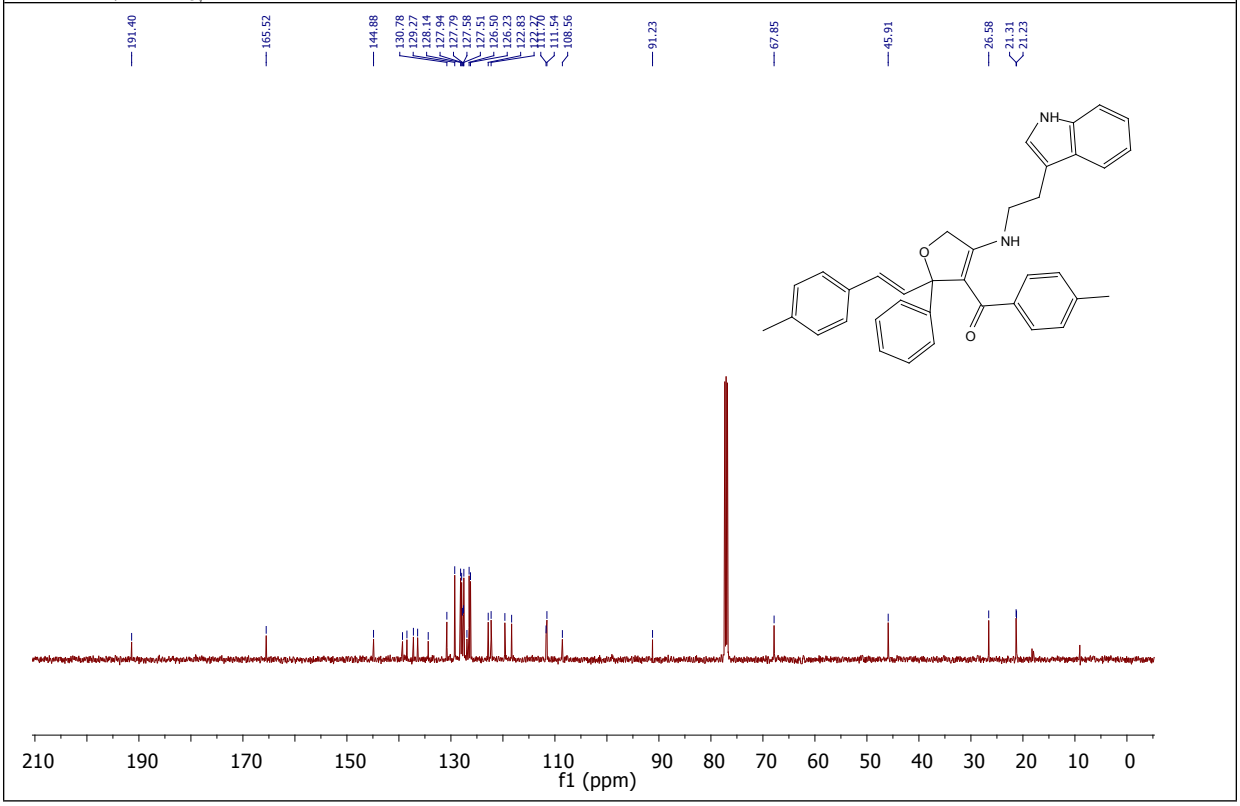


(*E*)-(4-(2-(*1H*-Indol-3-yl)ethylamino)-2-styryl-2-*p*-tolyl-2,5-dihydrofuran-3-yl)(*p*-tolyl)methanone**: 2m**

^{1}HNMR, $CDCl_3$, 400MHz

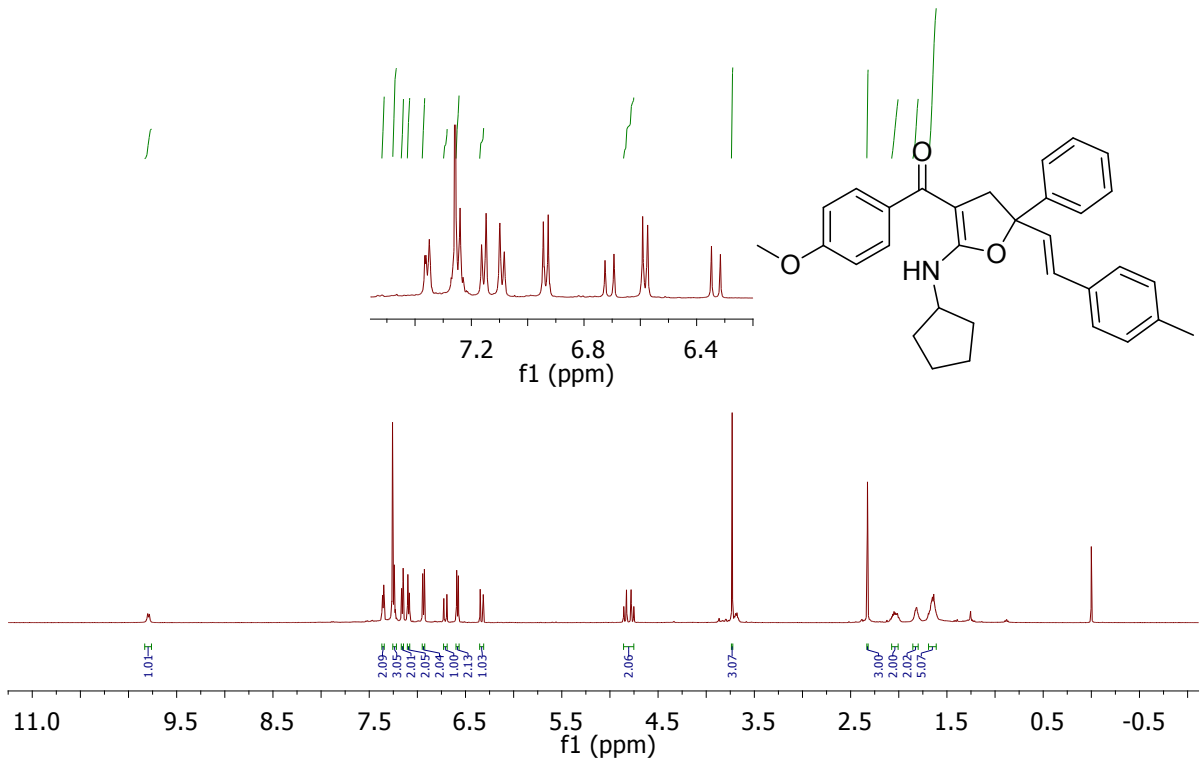


^{13}CNMR, $CDCl_3$, 125MHz

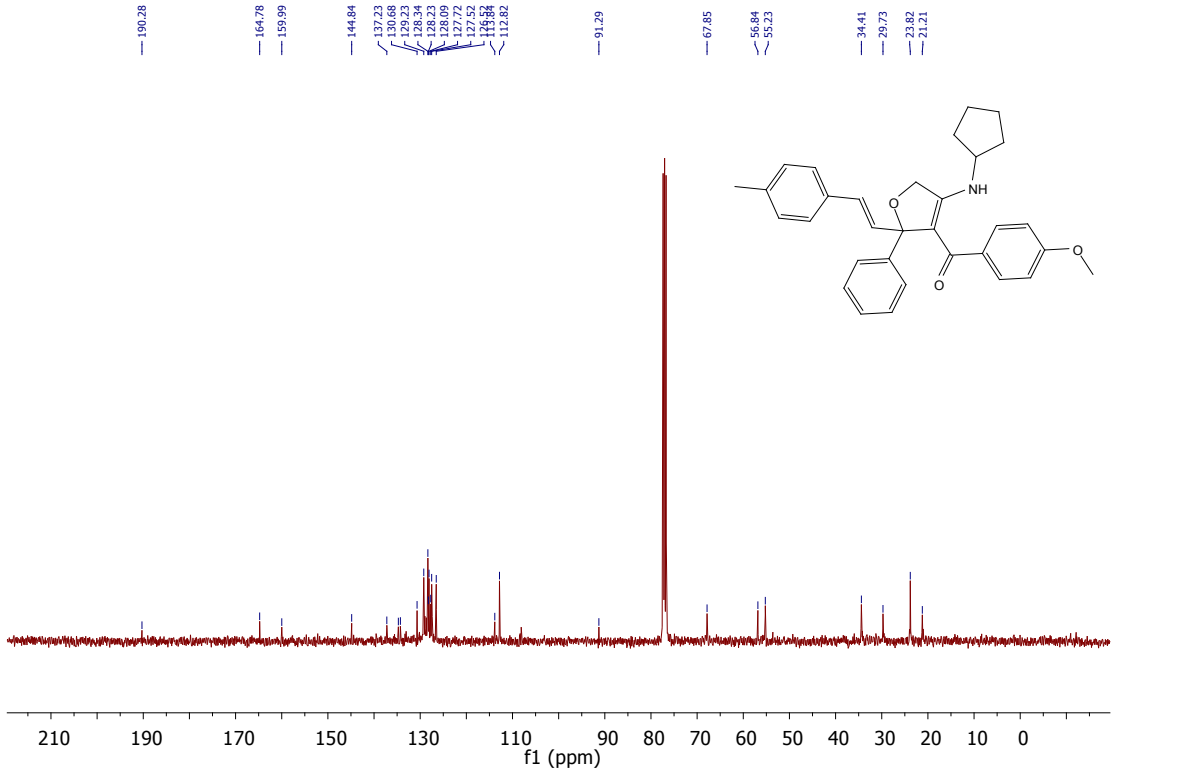


(*E*)-(4-(Cyclopentylamino)-2-styryl-2-(*p*-tolyl)-2,5-dihydrofuran-3-yl)(4-methoxyphenyl)methanone: **2n**

^{1}HNMR, $CDCl_3$, 500MHz

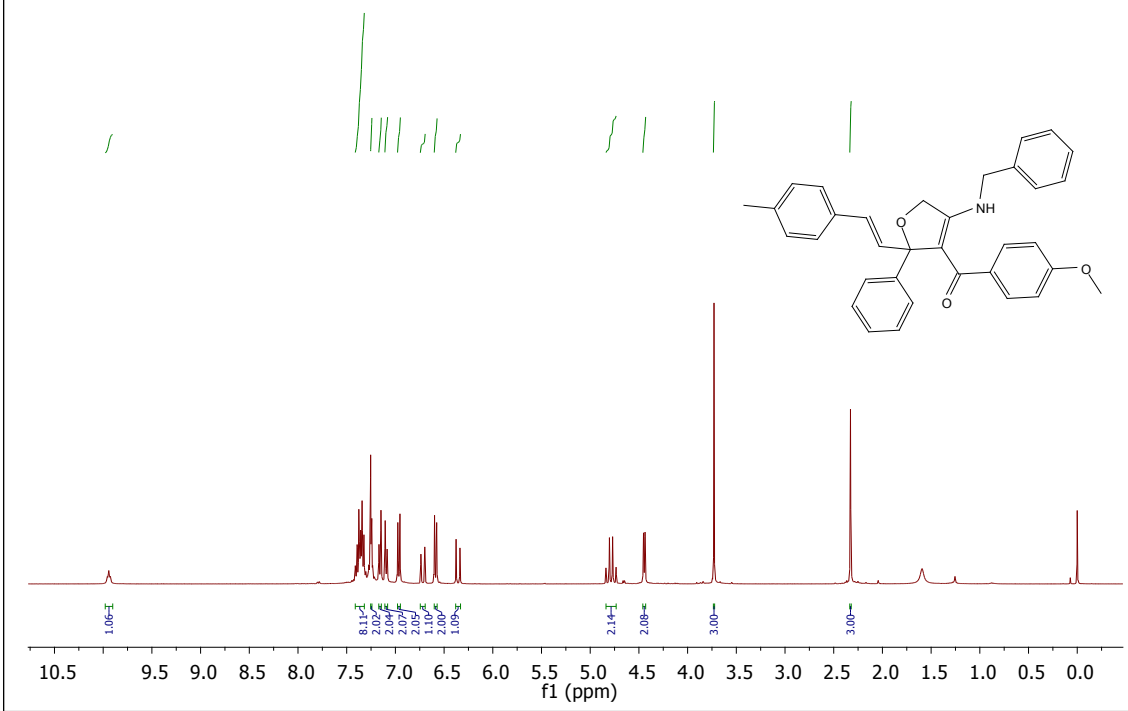


^{13}CNMR, $CDCl_3$, 100MHz

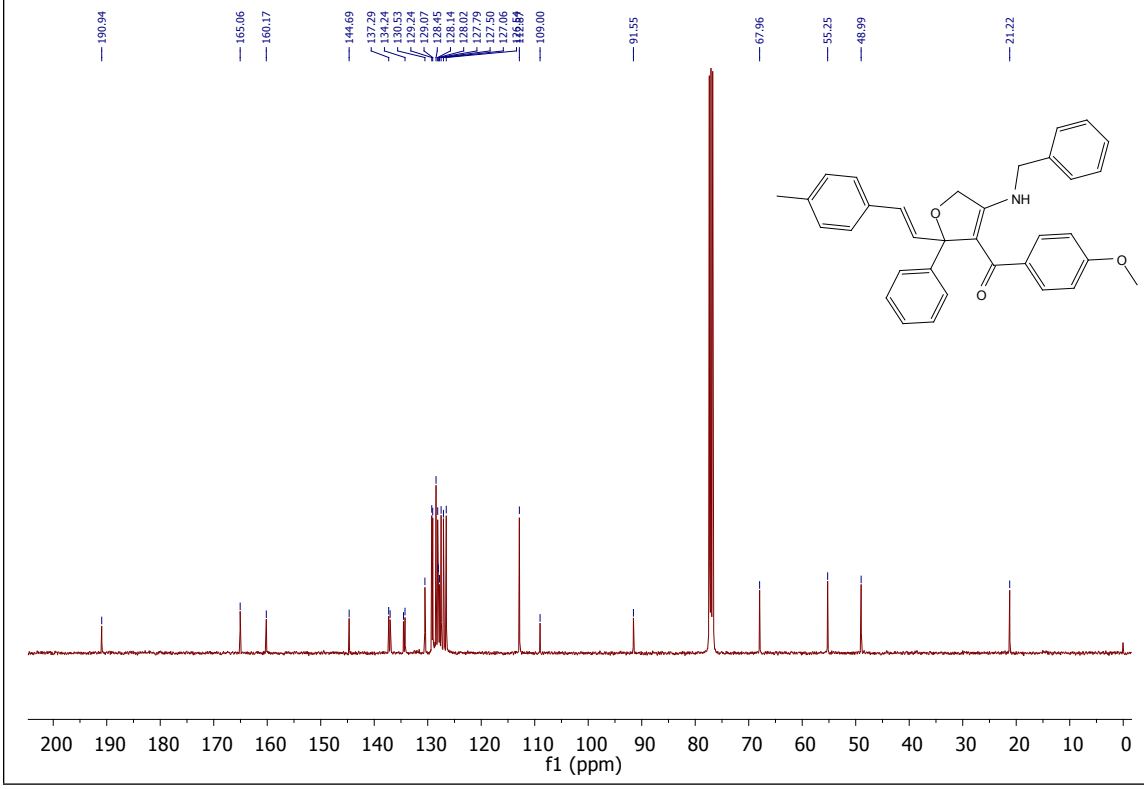


(*E*)-(4-(Benzylamino)-2-styryl-2-(*p*-tolyl)-2,5-dihydrofuran-3-yl)(4-methoxyphenyl)methanone: **2o**

^{1}HNMR, $CDCl_3$, 400MHz

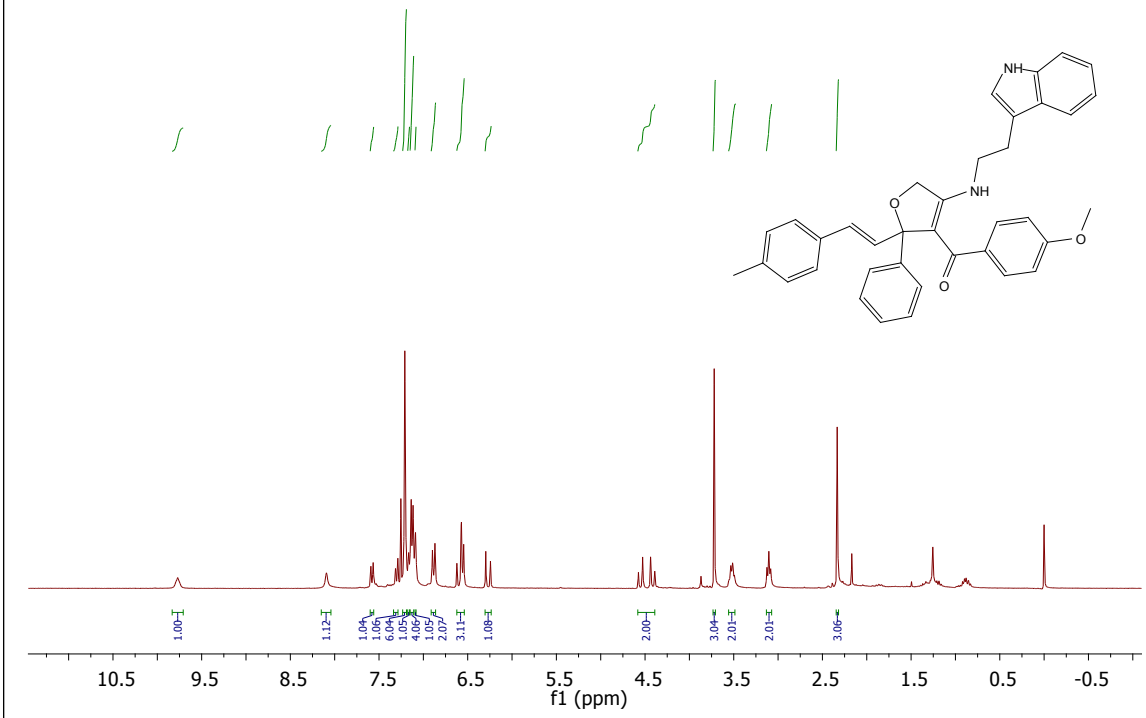


^{13}CNMR, $CDCl_3$, 100MHz

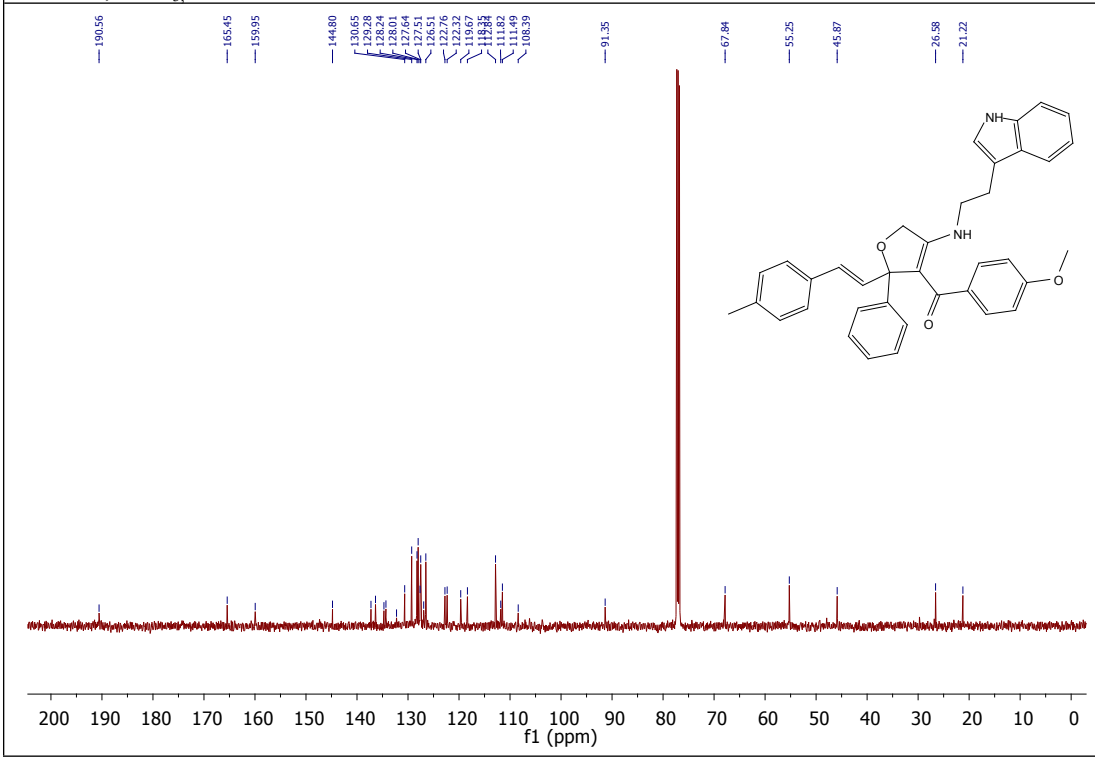


(E)-(4-(2-(*1H*-Indol-3-yl)ethylamino)-2-styryl-2-p-tolyl-2,5-dihydrofuran-3-yl)(4-methoxyphenyl)methanone: **2p:**

^{1}HNMR, $CDCl_3$, 300MHz

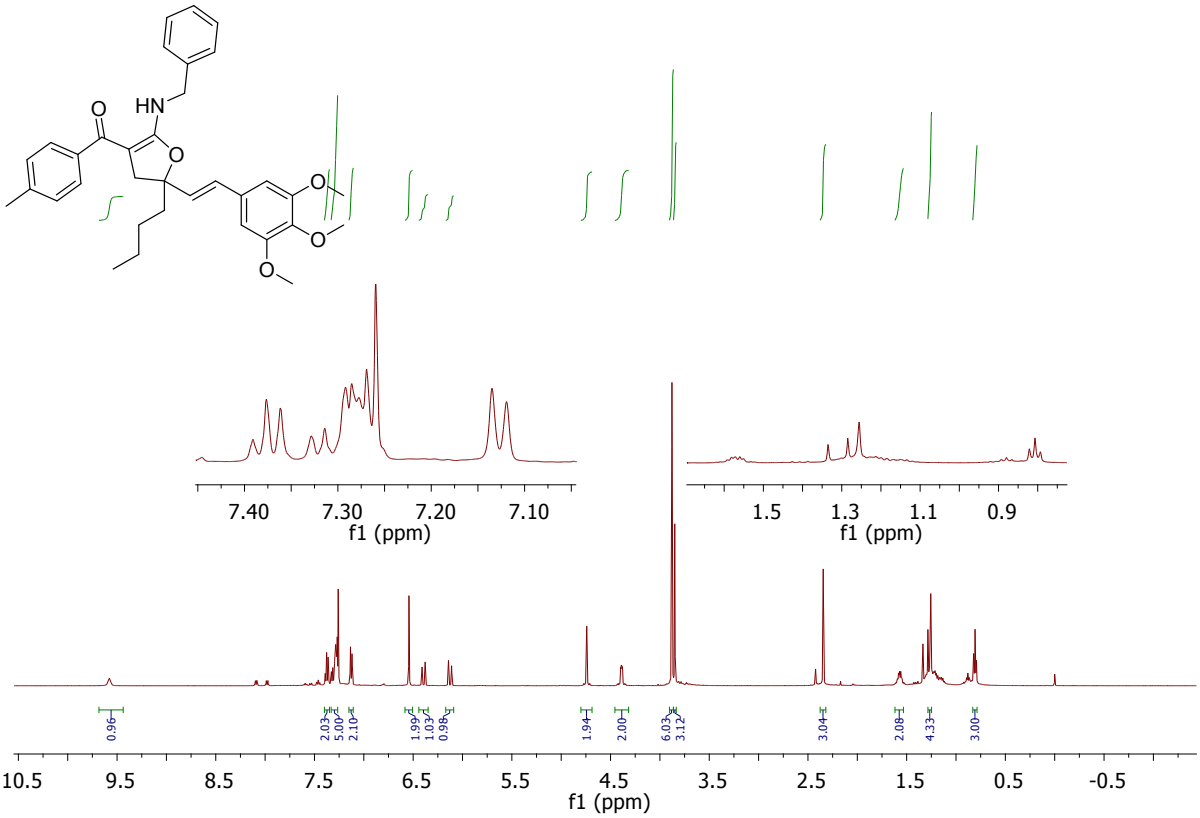


^{13}CNMR, $CDCl_3$, 125MHz

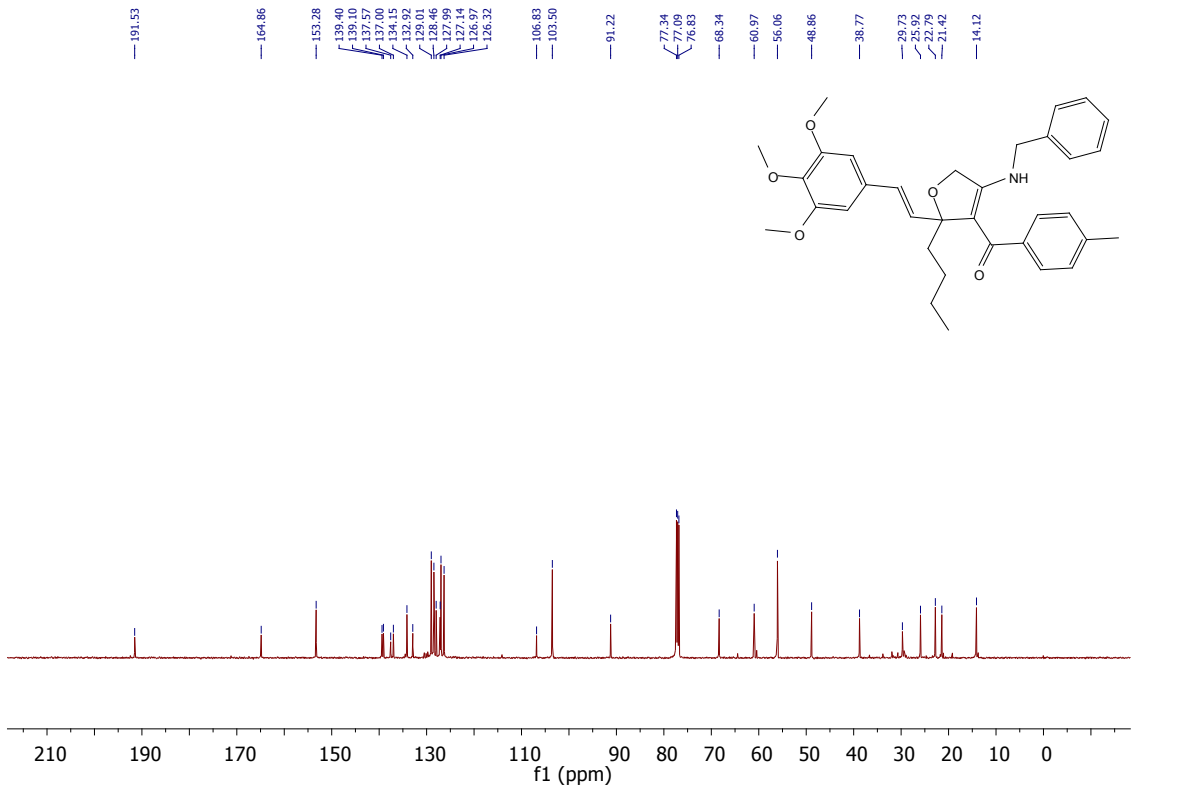


(*E*)-(4-(Benzylamino)-2-(hex-1-enyl)-2-(3,4,5-trimethoxyphenyl)-2,5-dihydrofuran-3-yl)(*p*-tolyl)methanone: **2q**

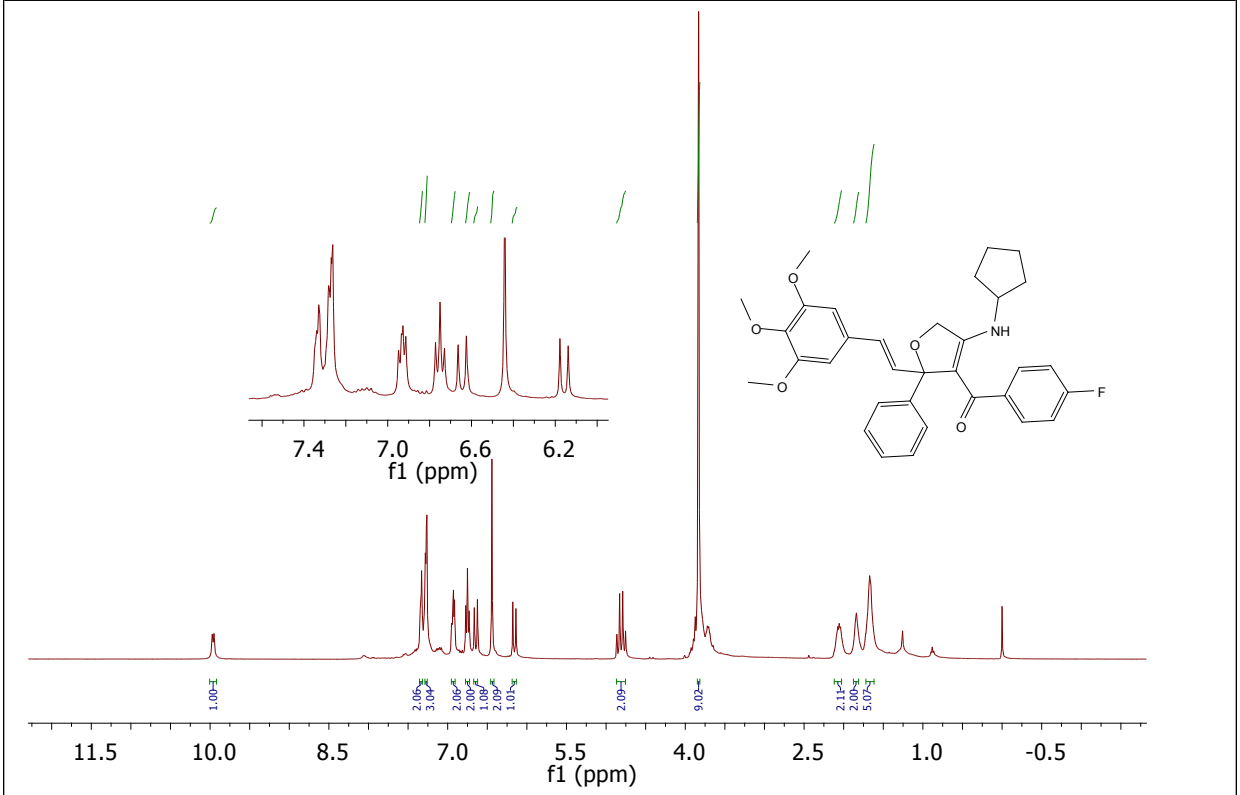
^{1}HNMR, $CDCl_3$, 500MHz



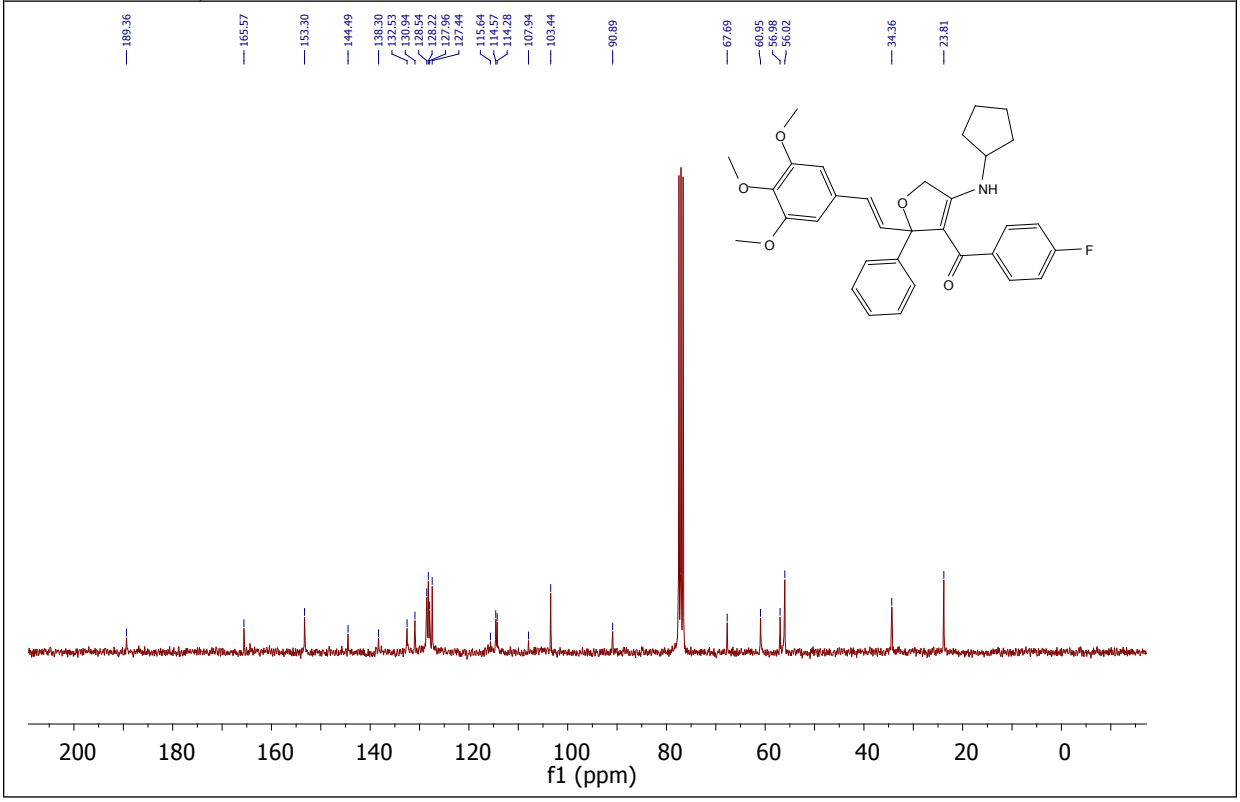
^{13}CNMR, $CDCl_3$, 125MHz



(*E*)-(4-(Cyclopentylamino)-2-styryl-2-(3,4,5-trimethoxyphenyl)-2,5-dihydrofuran-3-yl)(4-fluorophenyl)methanone: **2r**, ^{1}HNMR, $CDCl_3$, 400MHz

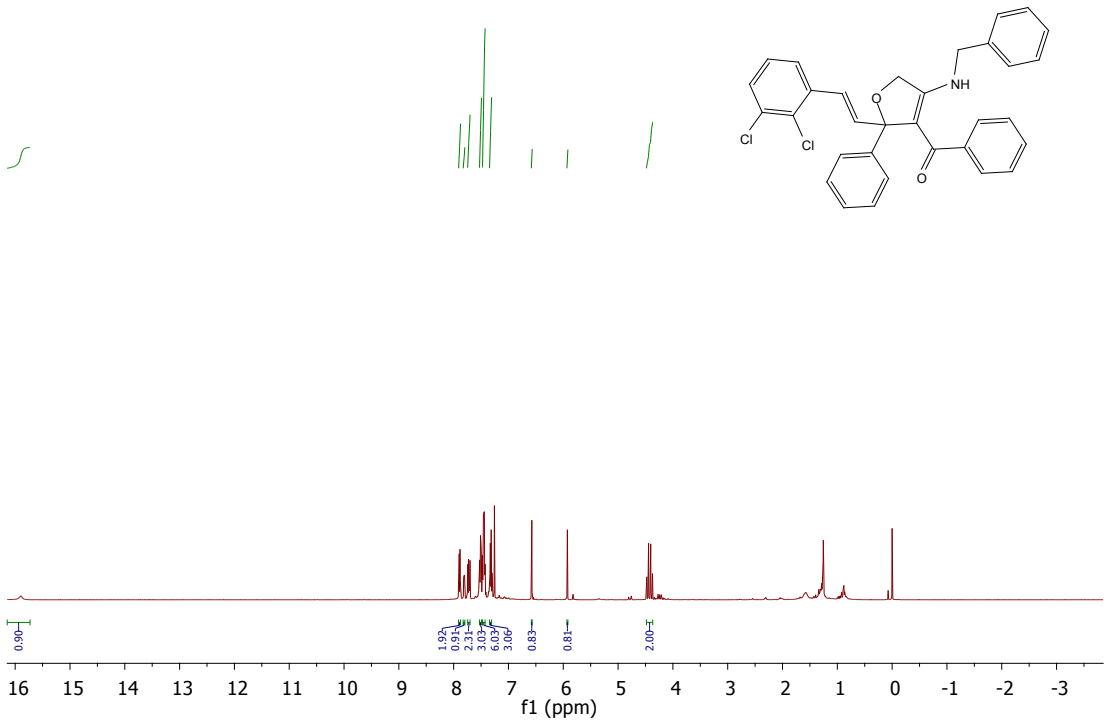


^{13}CNMR, $CDCl_3$, 75MHz

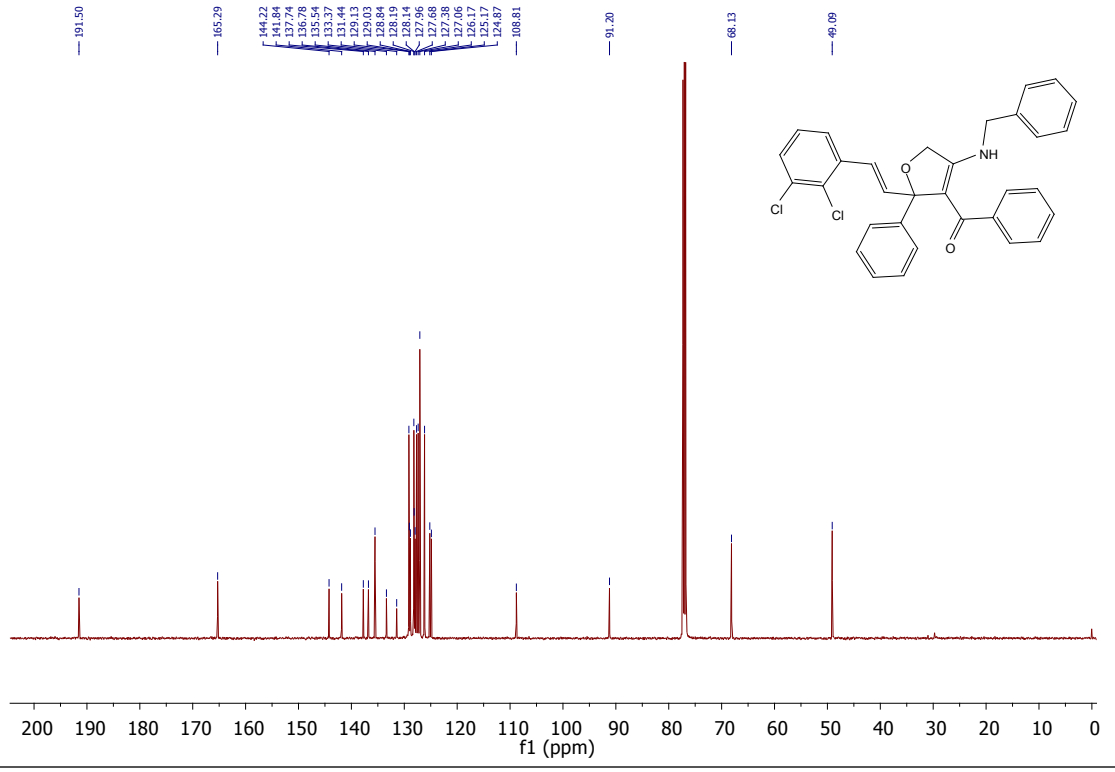


(*E*)-(4-(Benzylamino)-2-(2,3-dichlorophenyl)-2-styryl-2,5-dihydrofuran-3-yl)(phenyl)methanone: **2s**

^{1}HNMR, $CDCl_3$, 500MHz

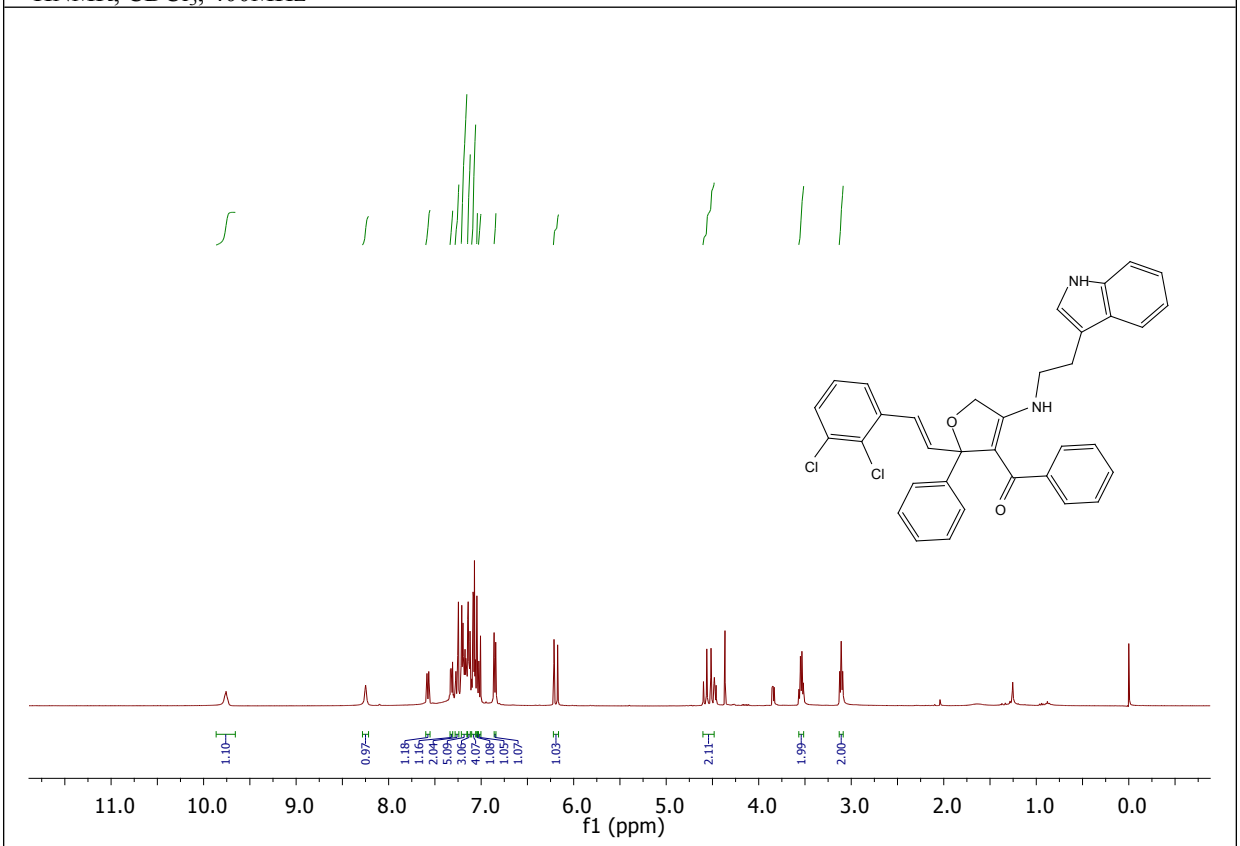


^{13}CNMR, $CDCl_3$, 125MHz

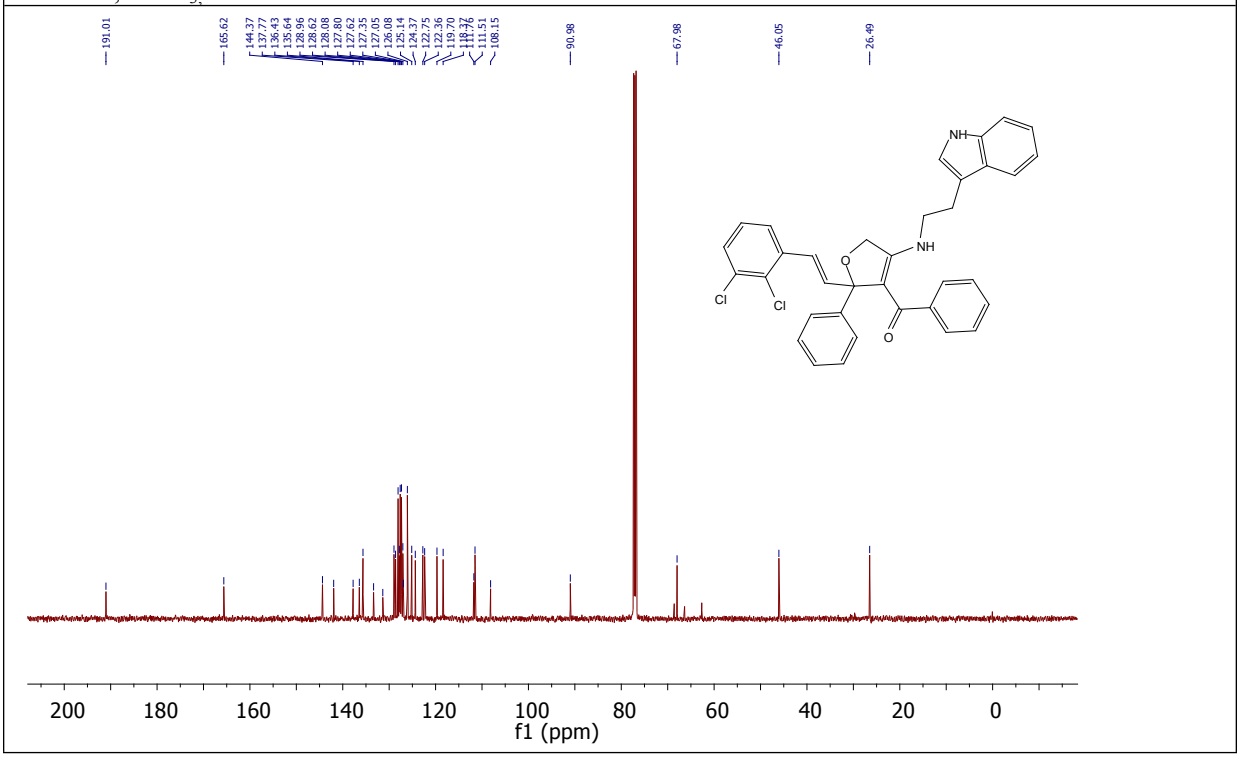


(*E*)-(4-(2-(*1H*-Indol-2-yl)ethylamino)-2-(2,3-dichlorophenyl)-2-styryl-2,5-dihydrofuran-3-yl)(phenyl)methanone: **2t**

^{1}HNMR, $CDCl_3$, 400MHz

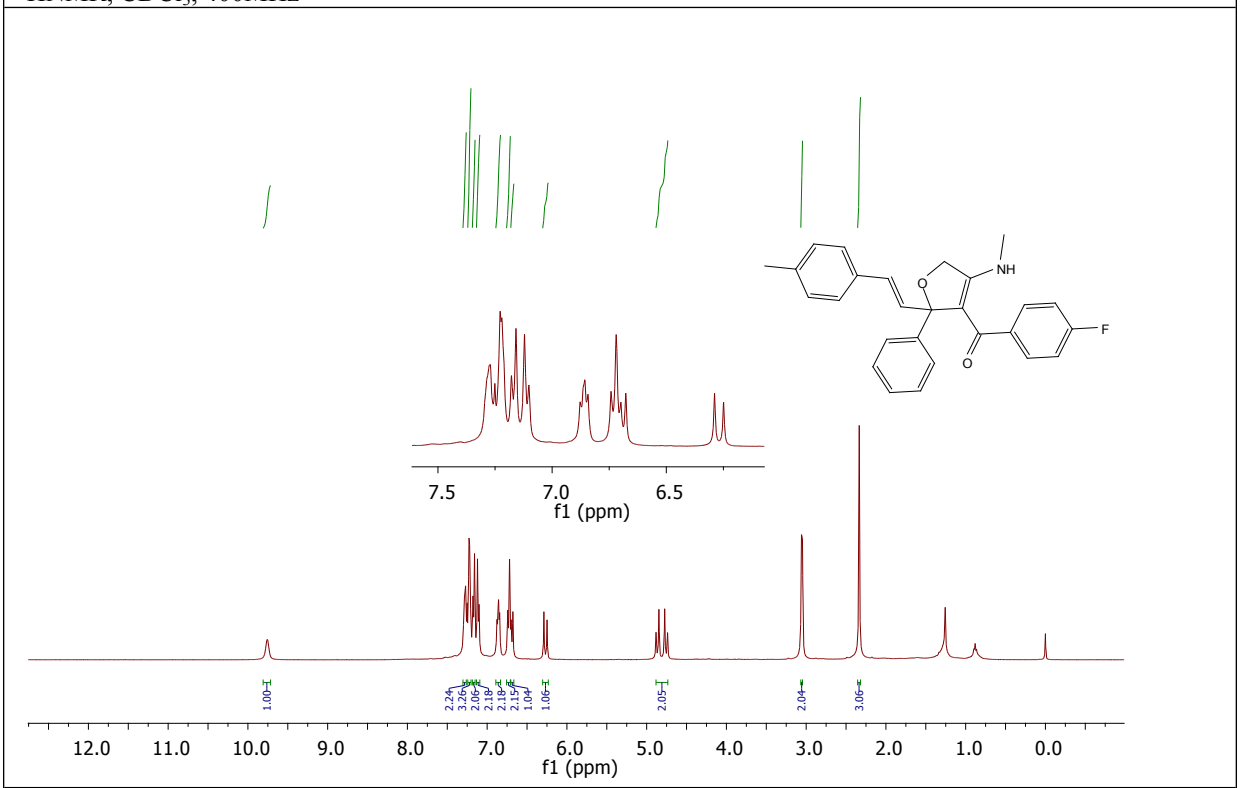


^{13}CNMR, $CDCl_3$, 125MHz

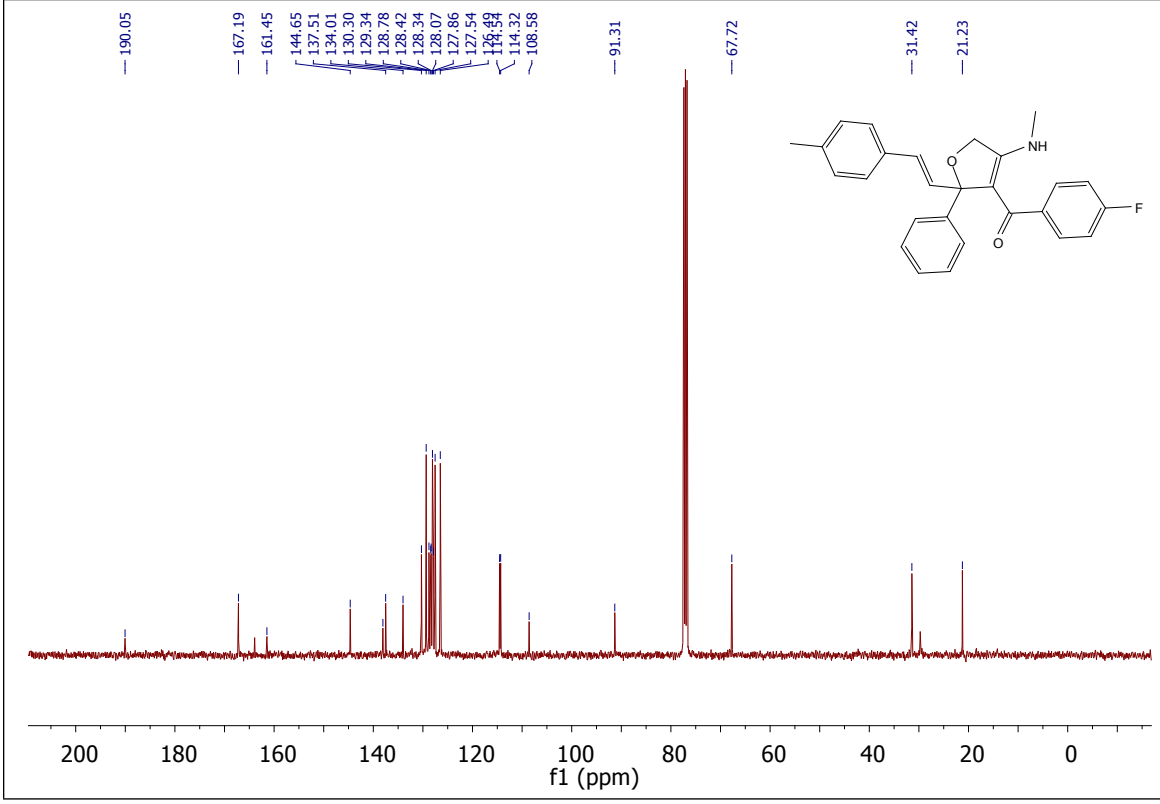


(*E*)-(4-Fluorophenyl)(4-(methylamino)-2-styryl-2-p-tolyl-2,5-dihydrofuran-3-yl)methanone: **2u**

^{1}HNMR, $CDCl_3$, 400MHz

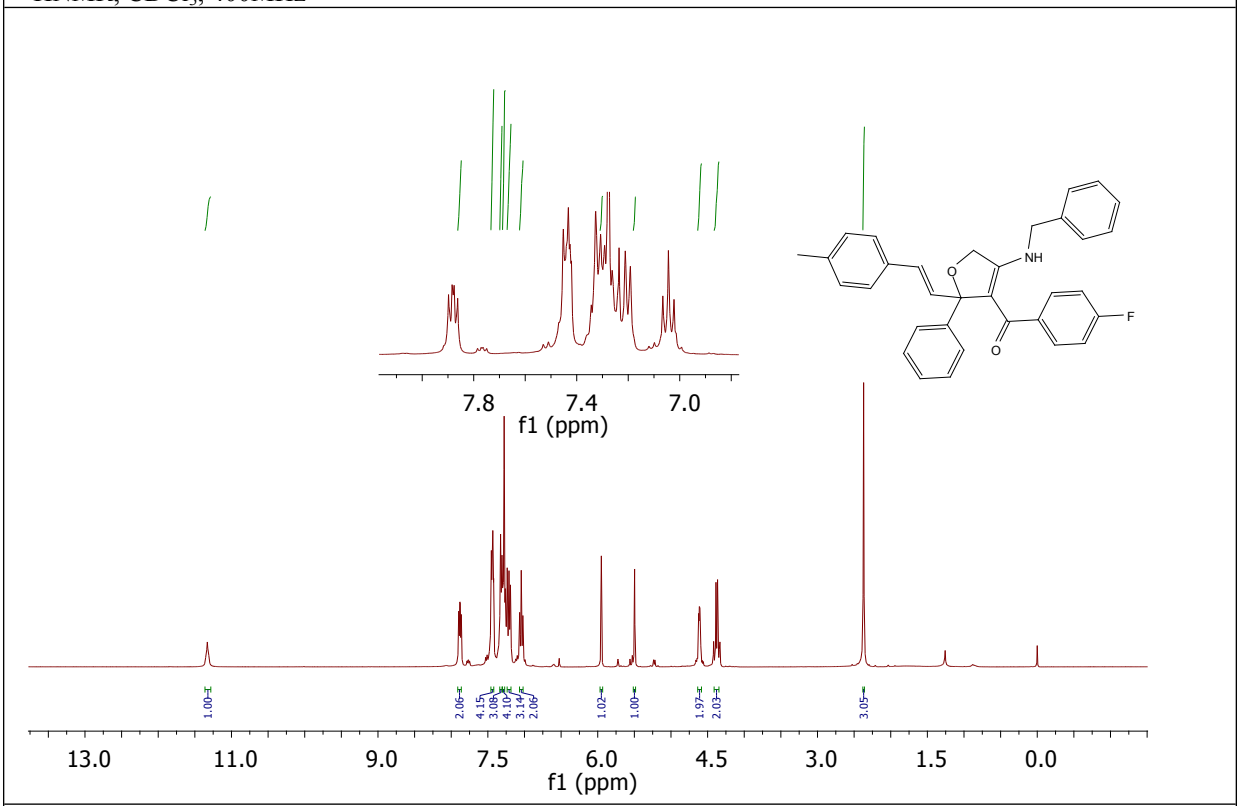


^{13}CNMR, $CDCl_3$, 100MHz

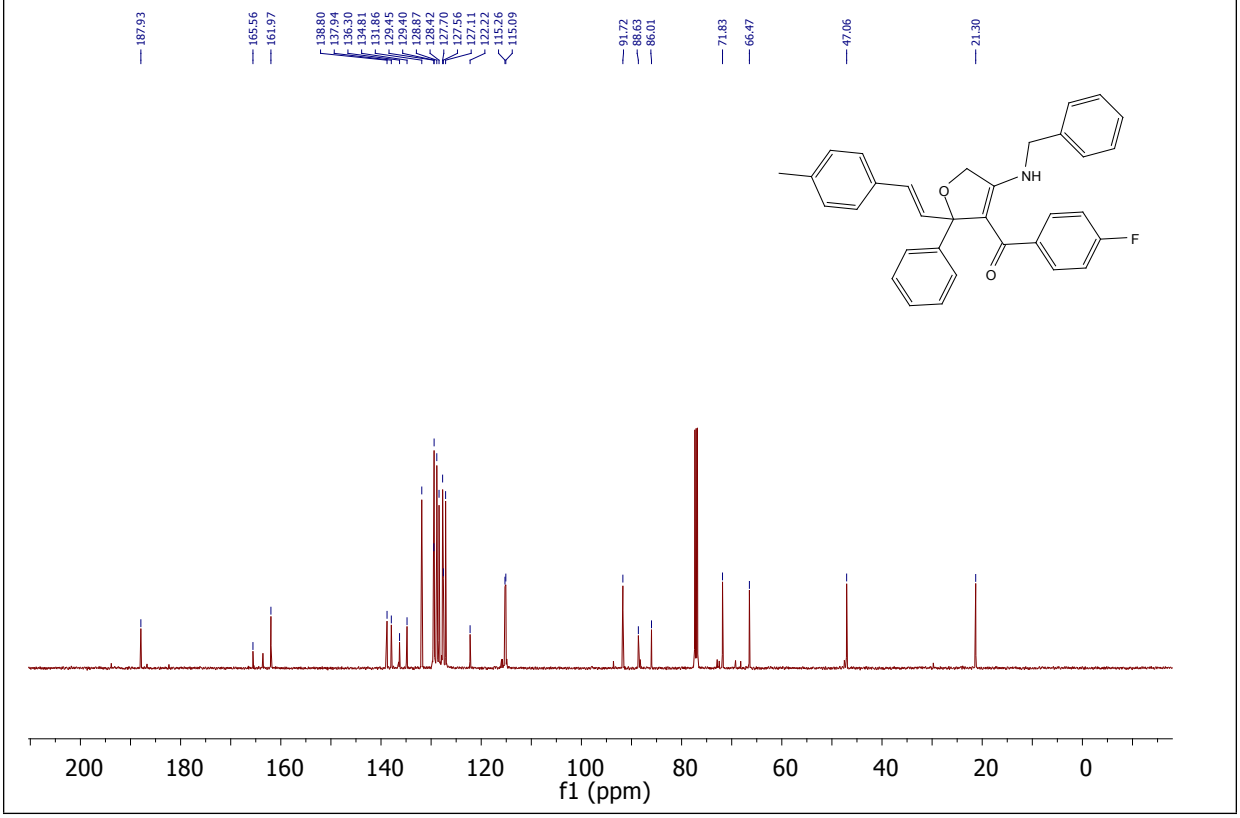


(*E*)-(4-(Benzylamino)-2-styryl-2-p-tolyl-2,5-dihydrofuran-3-yl)(4-fluorophenyl)methanone: **2v**

^{1}HNMR, $CDCl_3$, 400MHz



^{13}CNMR, $CDCl_3$, 125MHz



1.2 X-ray crystallography data of 2e

X-ray data for the compounds were collected at room temperature using a Bruker Smart Apex CCD diffractometer with graphite monochromated MoKα radiation (λ=0.71073Å) with ω-scan method.[1] Preliminary lattice parameters and orientation matrices were obtained from four sets of frames.

Integration and scaling of intensity data were accomplished using SAINT program.[1] The structure was solved by direct methods using SHELXS and refinement was carried out by full-matrix least-squares technique using SHELXL.[2] Anisotropic displacement parameters were included for all non-hydrogen atoms.The N-bound H atomwas located in difference Fourier maps and their positions and isotropic displacement parameters were refined. All other H atoms were positioned geometrically and treated as riding on their parent C atoms [C-H = 0.93-0.97 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H or 1.2U_{eq}(c) for other H atoms]. The methyl groups were allowed to rotate but not to tip.

Crystal Data for $C_{24}H_{21}NO_2S$ (M =387.48 g/mol): triclinic, space group P-1 (no. 2), a = 8.1000(10) Å, b = 9.6228(12) Å, c = 14.7780(18) Å, α = 98.898(2)°, β = 99.647(2)°, γ = 113.901(2)°, V = 1006.0(2) Å^3, Z = 2, T = 294.15 K, μ(MoKα) = 0.180 mm^{-1}, *Dcalc* = 1.279 g/cm^3, 10001 reflections measured (4.778° ≤ 2Θ ≤ 49.996°), 3555 unique (R_{int} = 0.0381, R_{sigma} = 0.0478) which were used in all calculations. The final R_1 was 0.0653 (I > 2σ(I)) and wR_2 was 0.1667 (all data). CCDC1904334 contains supplementary Crystallographic data for the structure. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0) 1223 336 033; email: deposit@ccdc.cam.ac.uk].

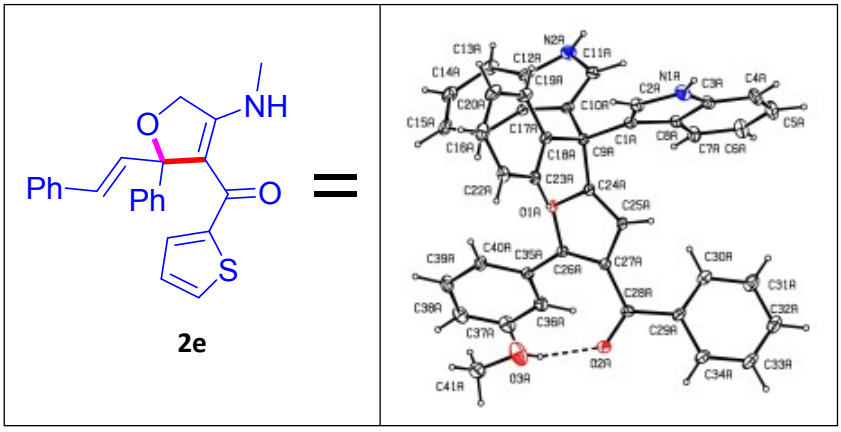


Fig.1. A view of BG51, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary radii.

1. Bruker (2001). SAINT (Version 6.28a) & SMART (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
2. Sheldrick G. M. (2015) Acta Crystallogr C71: 3-8.