Supporting Information

A Sequential Ugi-Smiles / Transition-Metal-Free Endo-Dig Conia-ene Cyclization: The Selective Synthesis of Saccharin Substituted 2,5-dihydropyrroles

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X-ray crystallographic analysis for product 5b

Computing details

Data collection: MAR345 dtb Program (1.24-4, 2013); cell refinement: Automar software package (3.3a, 2015); data reduction: Automar software package (3.3a, 2015); program(s) used to solve structure: SHELXT 2018/2 (Sheldrick, 2018); program(s) used to refine structure: SHELXL2016/6 (Sheldrick, 2016); molecular graphics: DIAMOND (Brandenburg, 1999); software used to prepare material for publication: PLATON (2018).

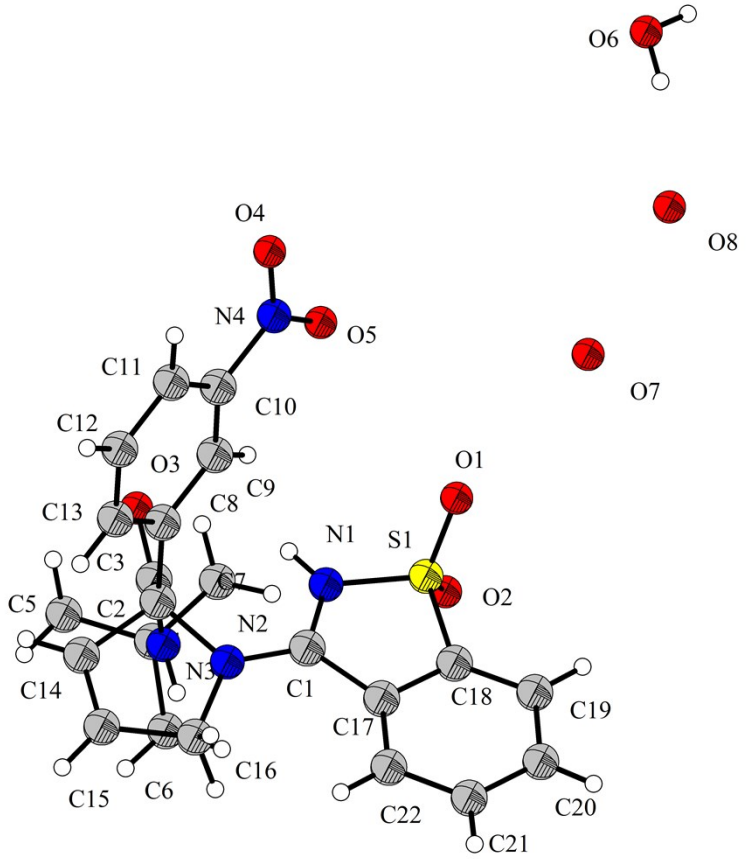


Table S1. Crystal data

$C_{22}H_{23}N_4O_5S \cdot 0.5(O) \cdot 0.5(H_2O)$	F(000)=1984
M_r = 472.51	D_x =1.275 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
a = 27.896 (6) Å	Cell parameters from 15170 reflection
b = 7.6894 (15) Å	θ = 2.7–25.0°
c = 22.958 (5) Å	μ = 0.17 mm^{-1}
β = 90.39°	*T* = 290 K
V = 4924.5 (18) Å^3	Needle, colourless
Z = 8	0.3 × 0.2 × 0.1 m

Table S2. Data collection

MAR345 Diffractometer	Radiation source: fine-focus sealed tube Mirrors monochromator Detector resolution: 0.81 pixels mm^{-1}

image–plate detector – phi oscillation scans
Absorption correction: multi-scan
R.H. Blessing, Acta Crystallogr., Sect A 1995, 51, 33-38

Tmin = 0.969, Tmax = 1.067
15163 measured reflections

4303 independent reflections
3835 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.8°$
$h = -32 \rightarrow 32$
$k = -9 \rightarrow 9$
$l = -27 \rightarrow 24$

Table S3. Refinement

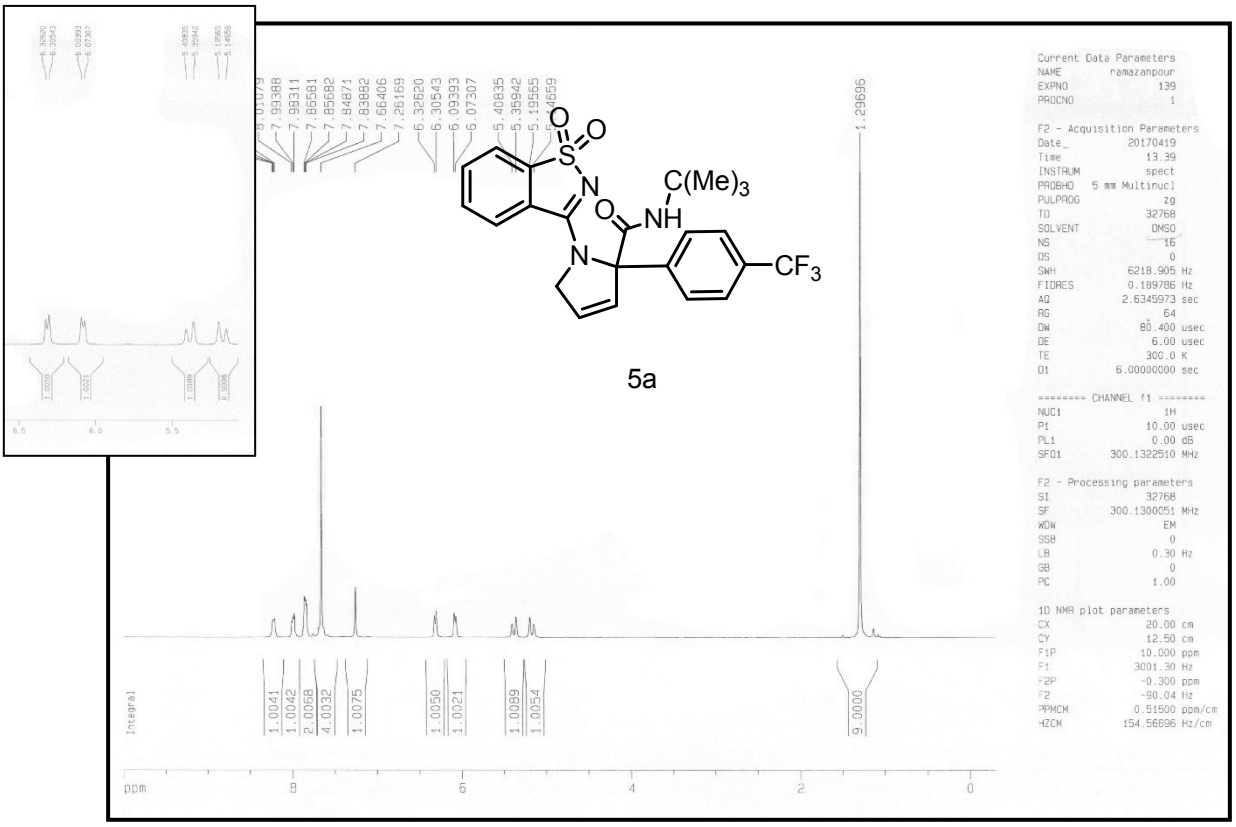
Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.181$
$S = 1.12$
4303 reflections
314 parameters
0 restraints

Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.082P)^2 + 6.4289P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

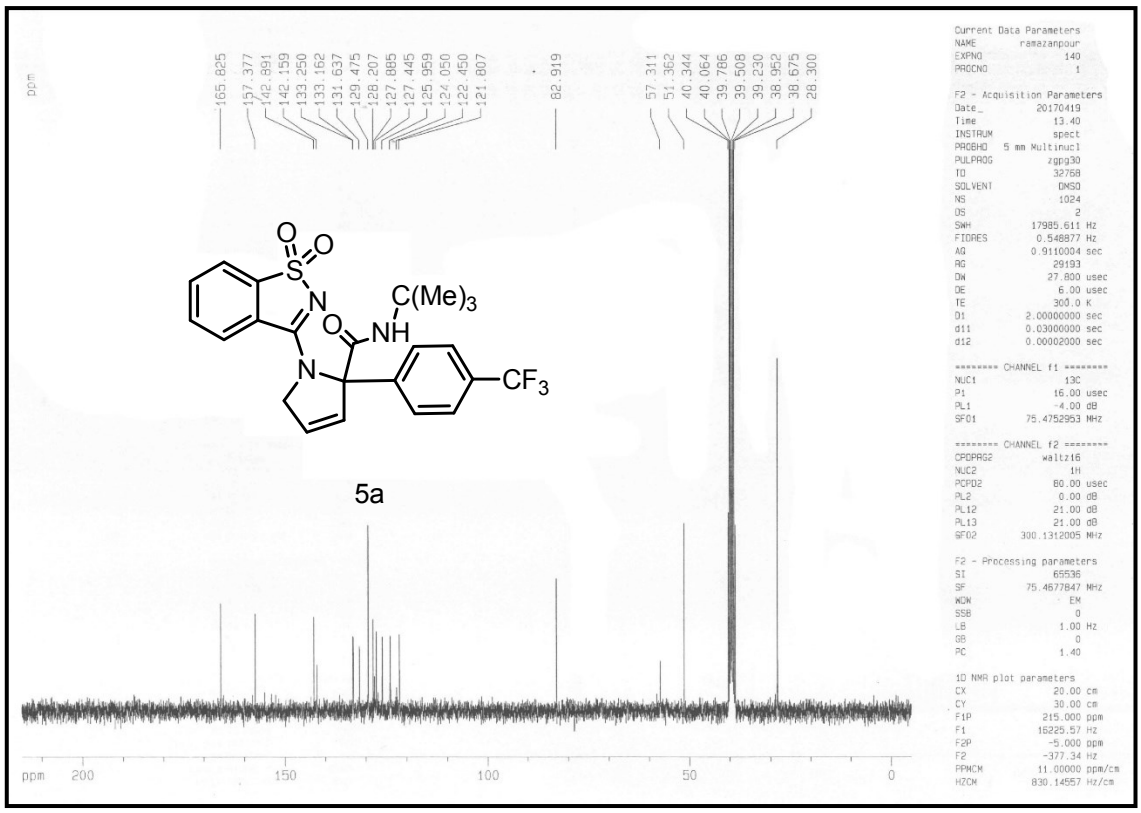
Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

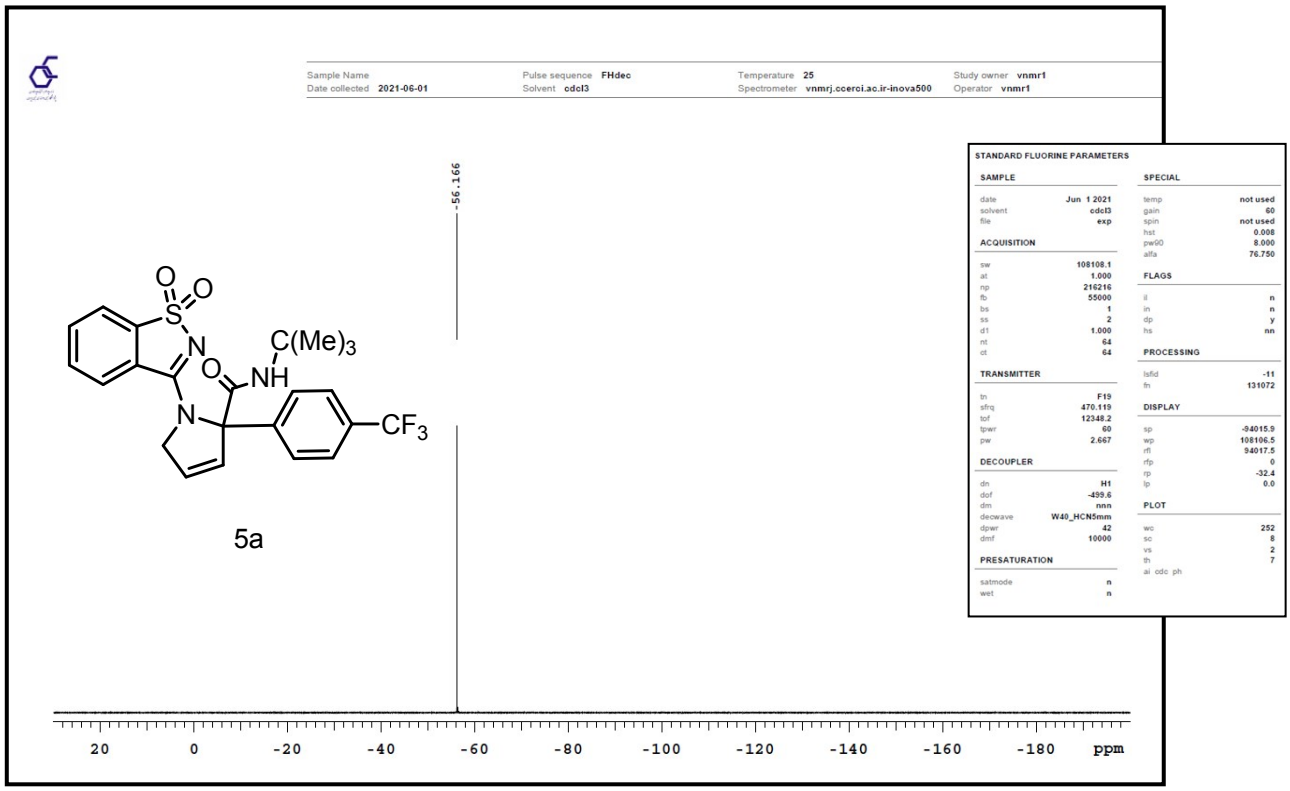
^{1}H NMR, ^{13}C NMR, ^{19}F NMR and IR Spectra



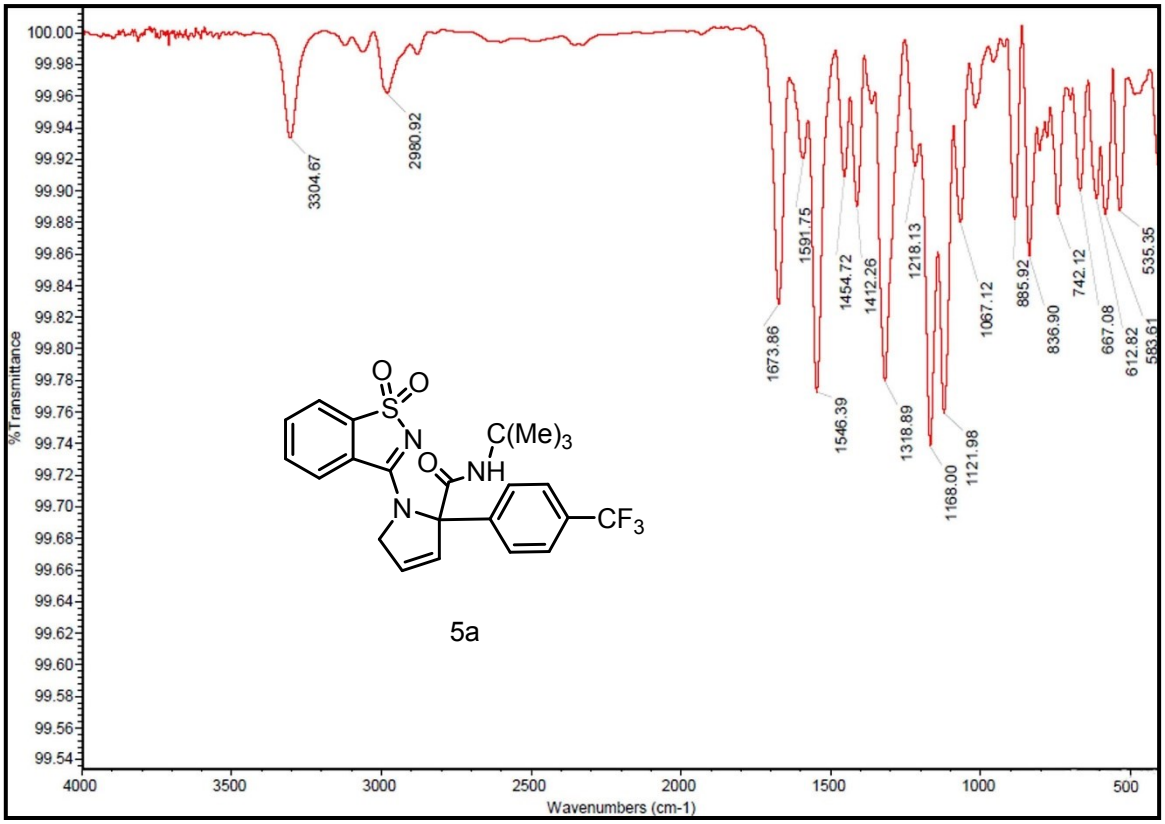
^{1}H NMR spectrum of compound **5a** (300 MHz, DMSO).



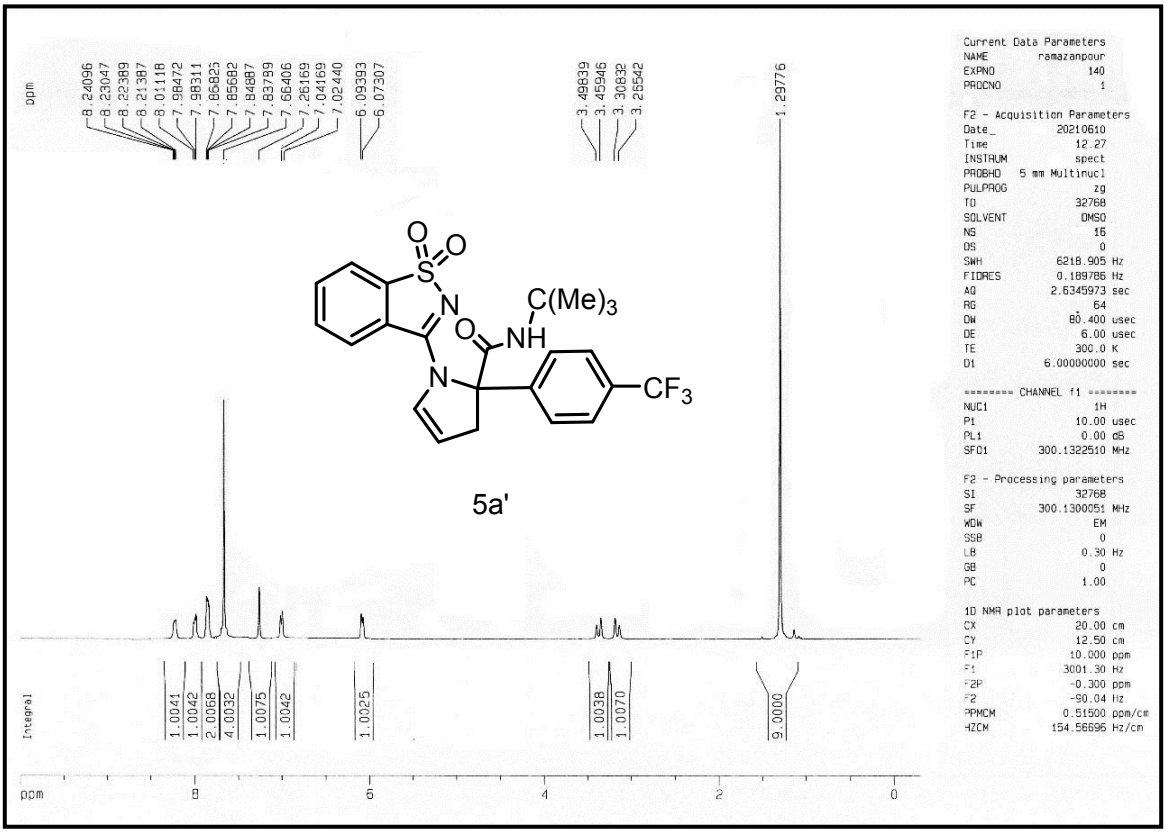
^{13}C NMR spectrum of compound **5a** (75 MHz, DMSO).



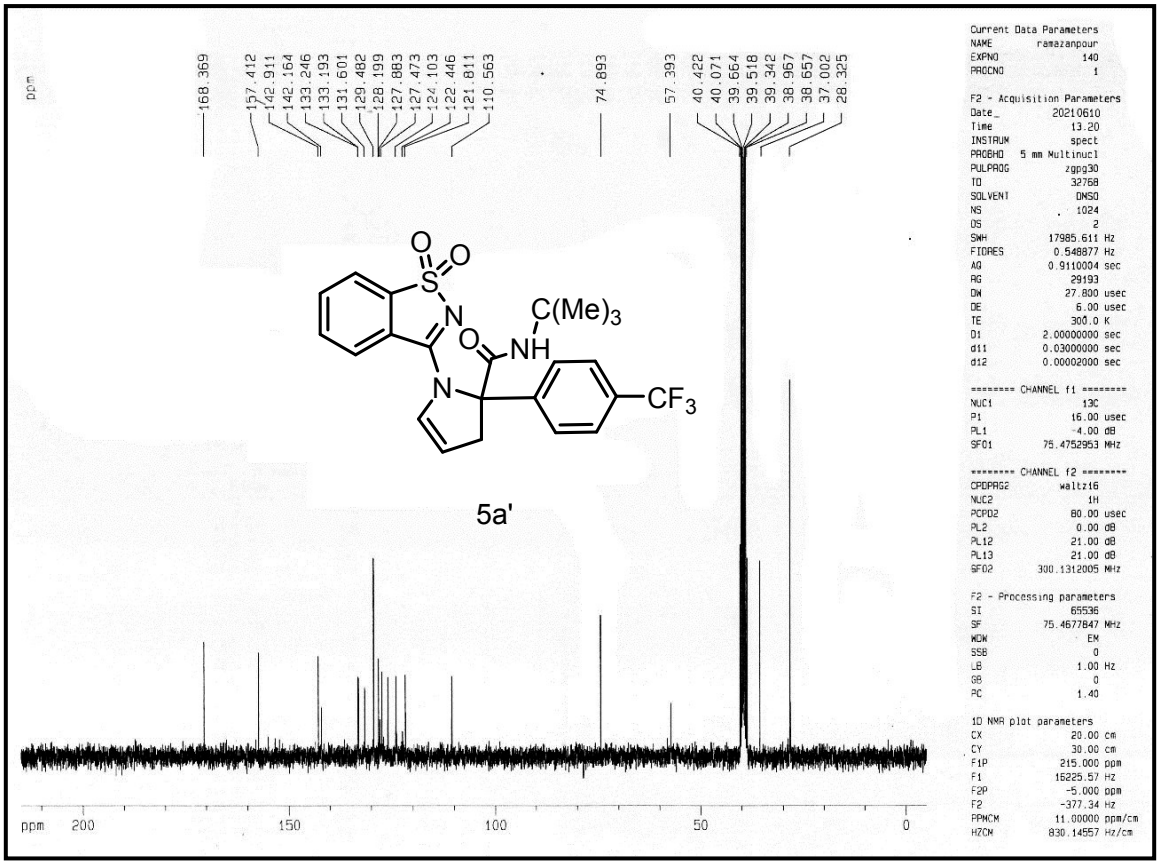
^{19}F NMR spectrum of compound **5a** (470 MHz, $CDCl_3$).



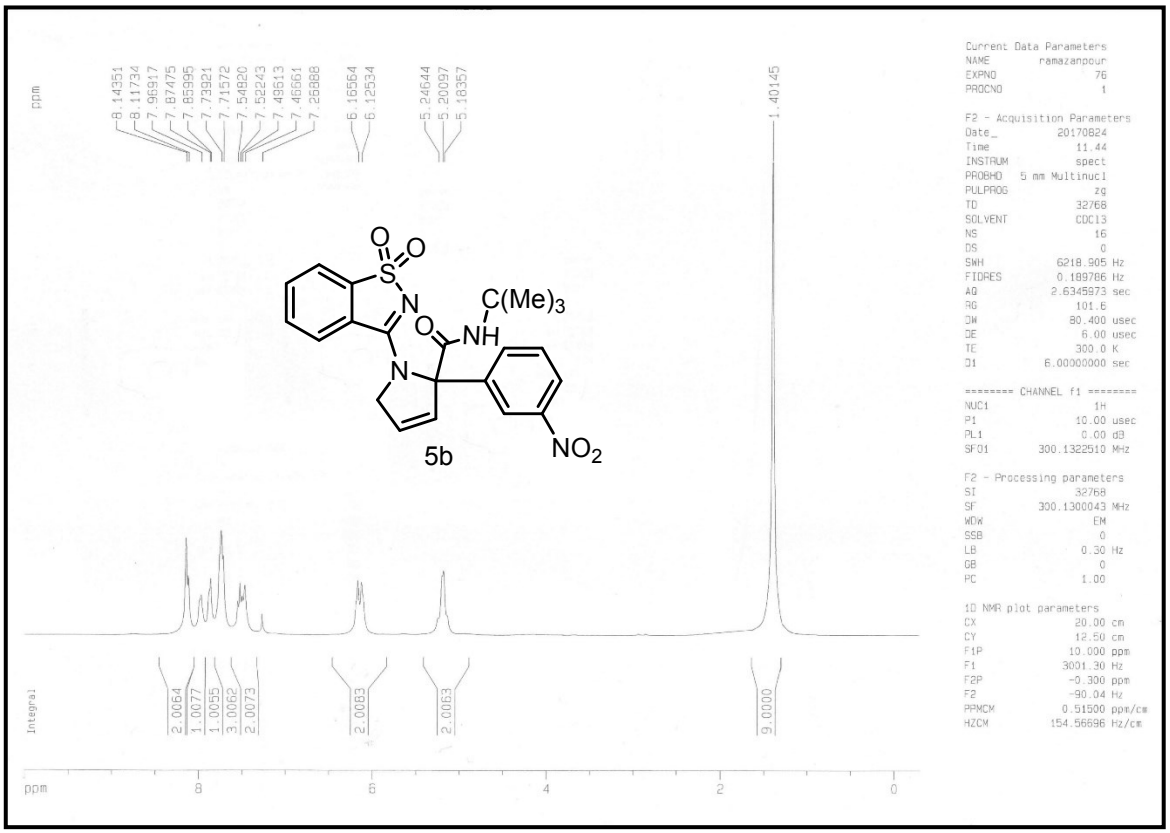
IR spectrum of compound **5a**.



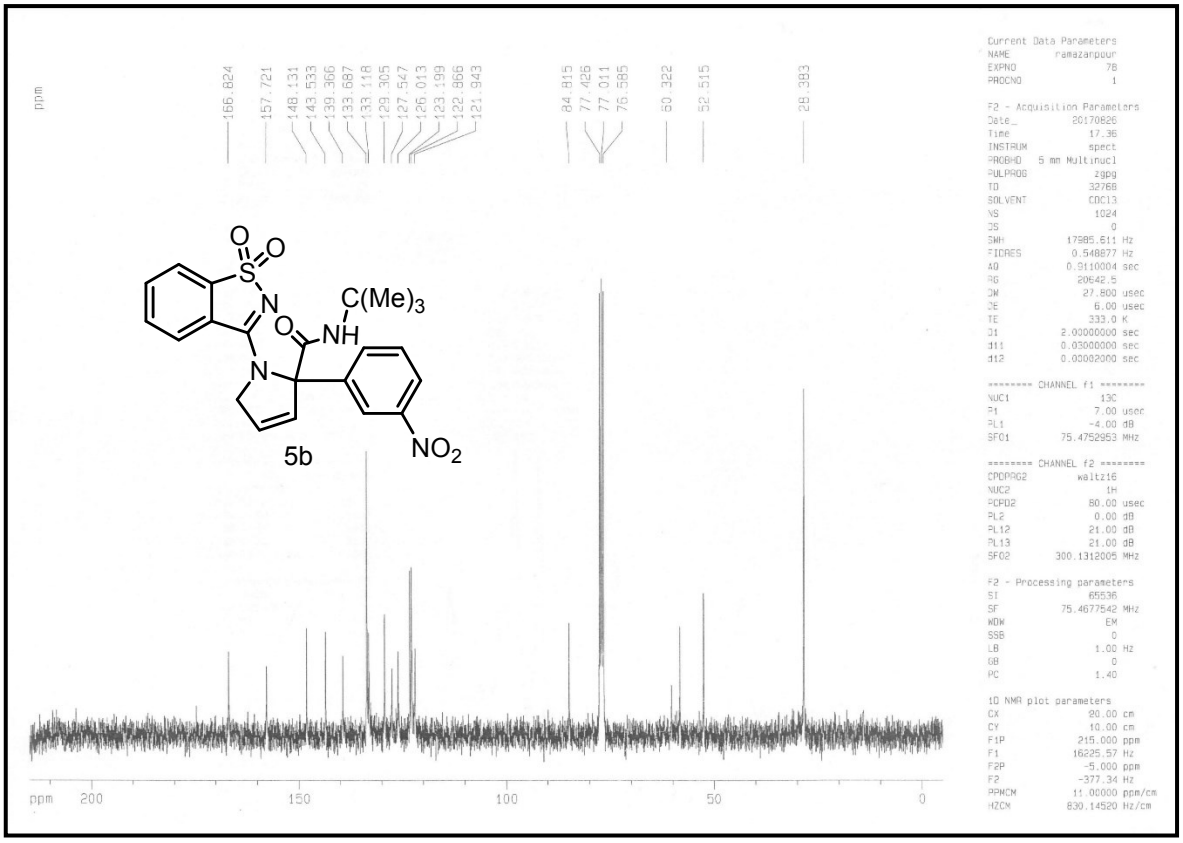
[1]H NMR spectrum of compound **5a′** (300 MHz, DMSO).



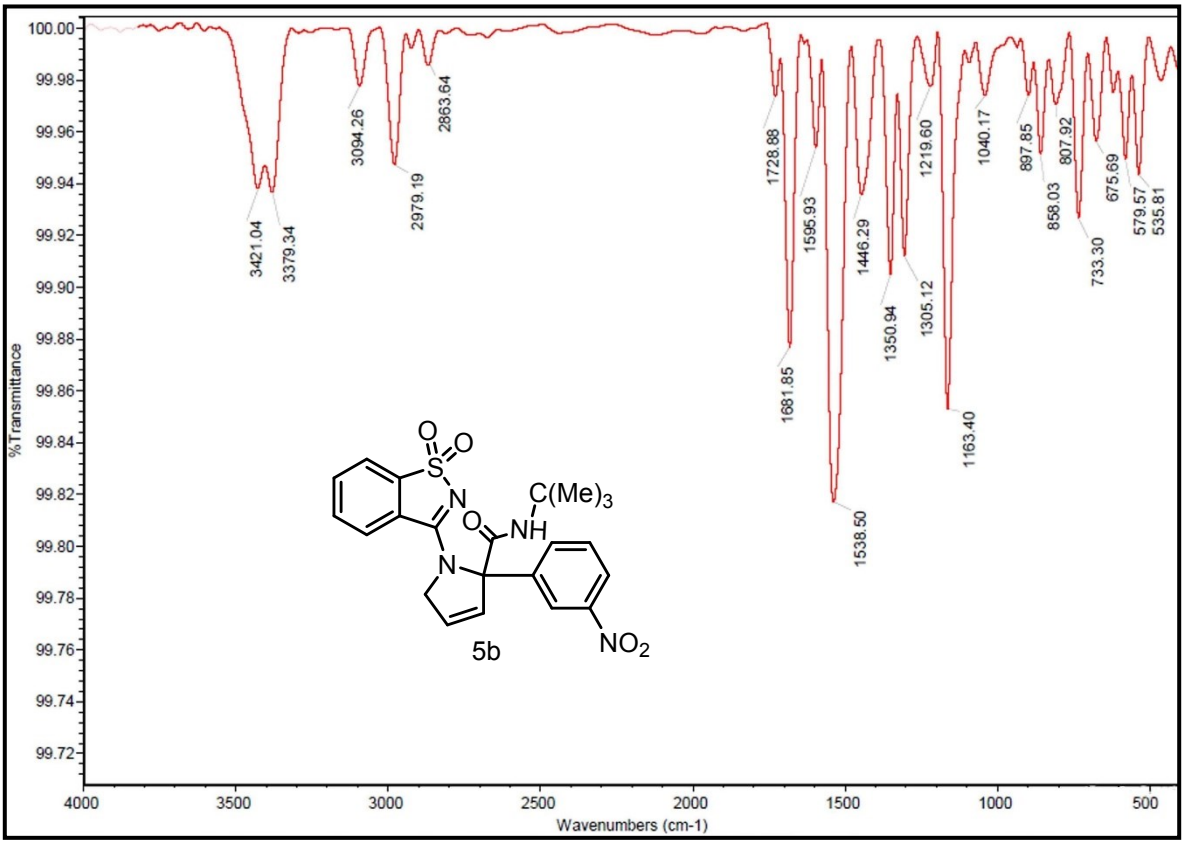
^{13}C NMR spectrum of compound **5a′** (75 MHz, DMSO).



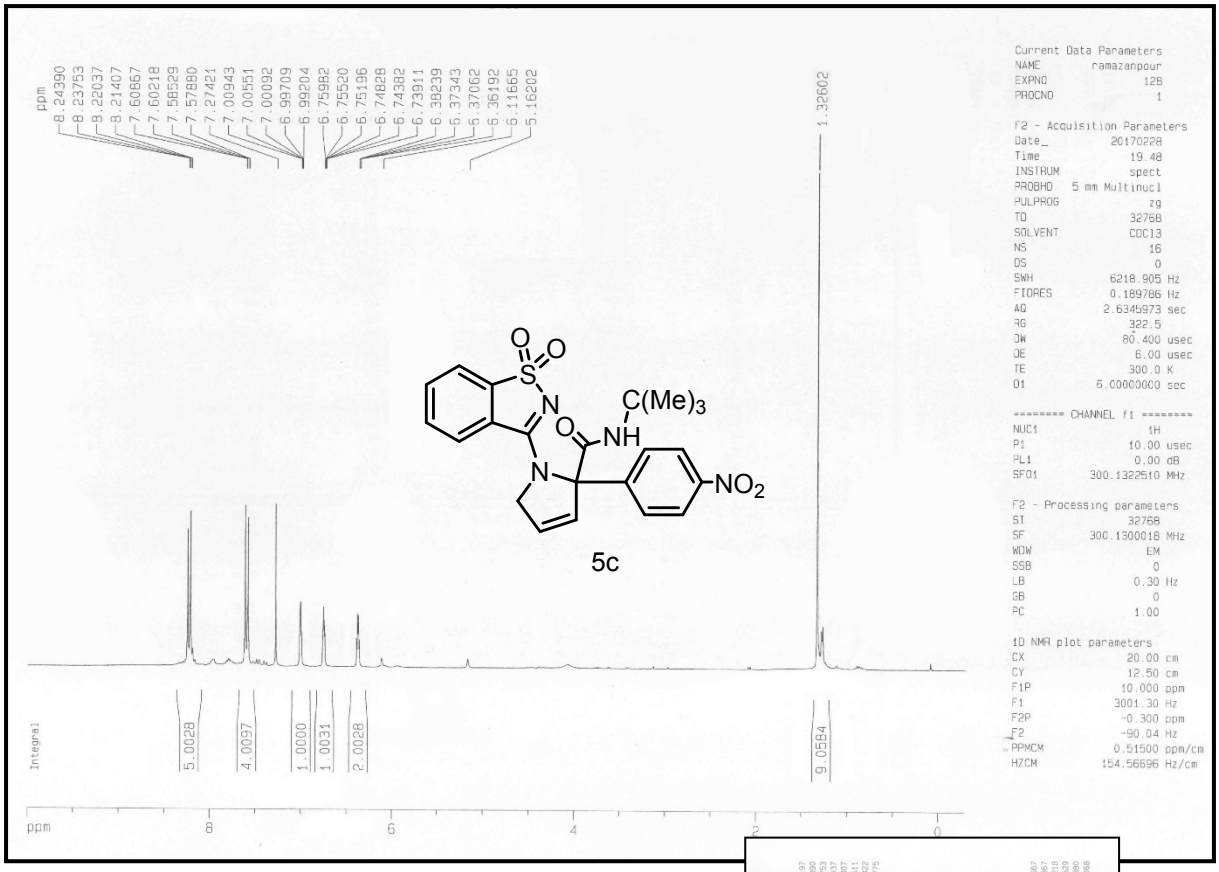
^{1}H NMR spectrum of compound **5b** (300 MHz, $CDCl_3$).

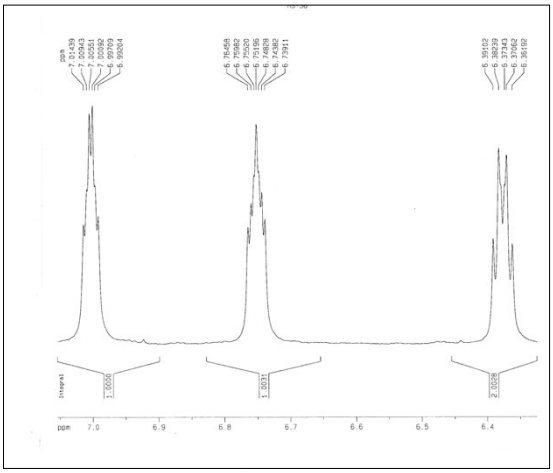


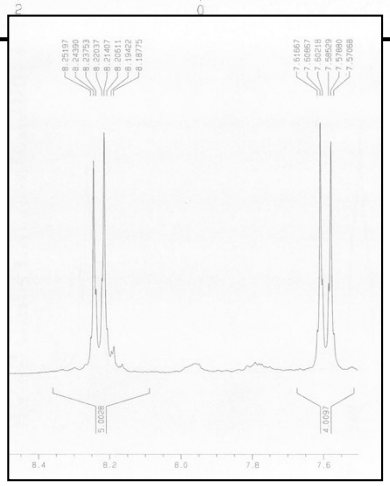
^{13}C NMR spectrum of compound **5b** (75 MHz, $CDCl_3$).



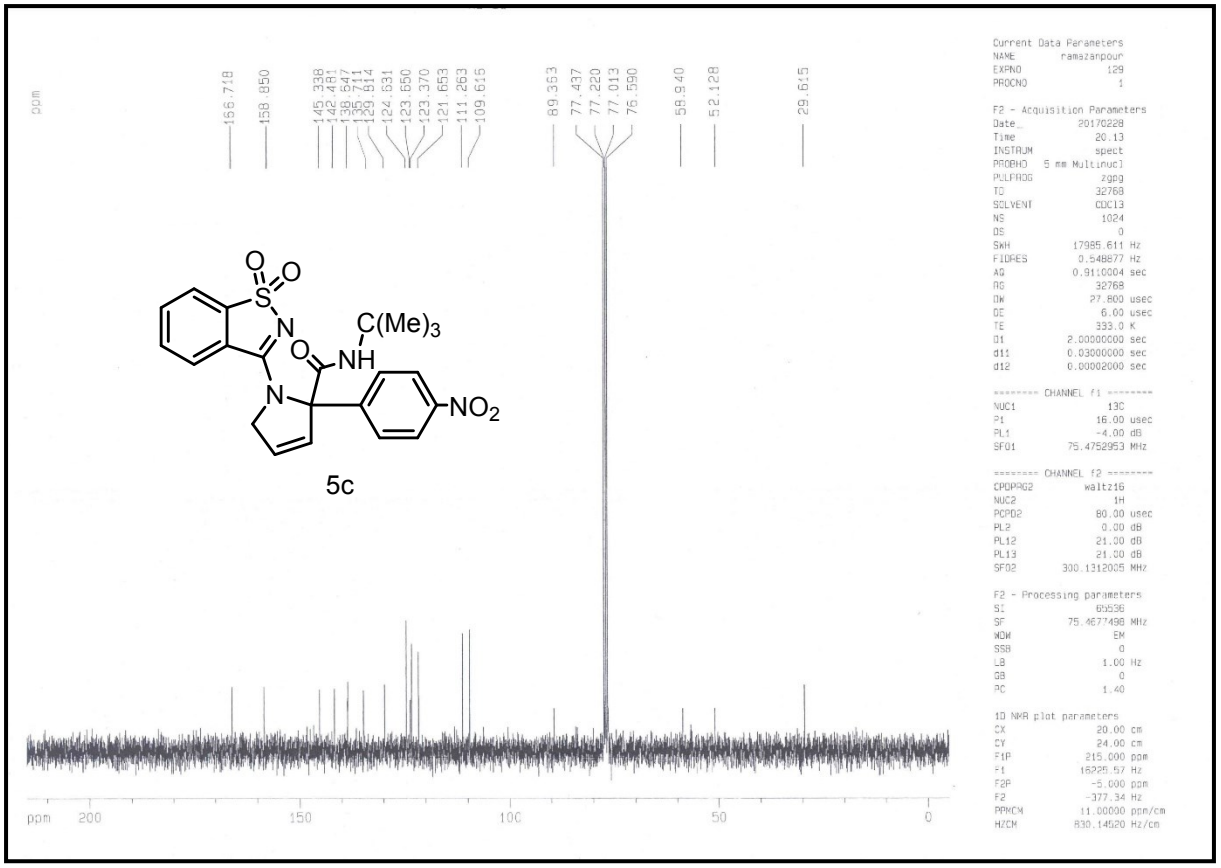
IR spectrum of compound **5b**.



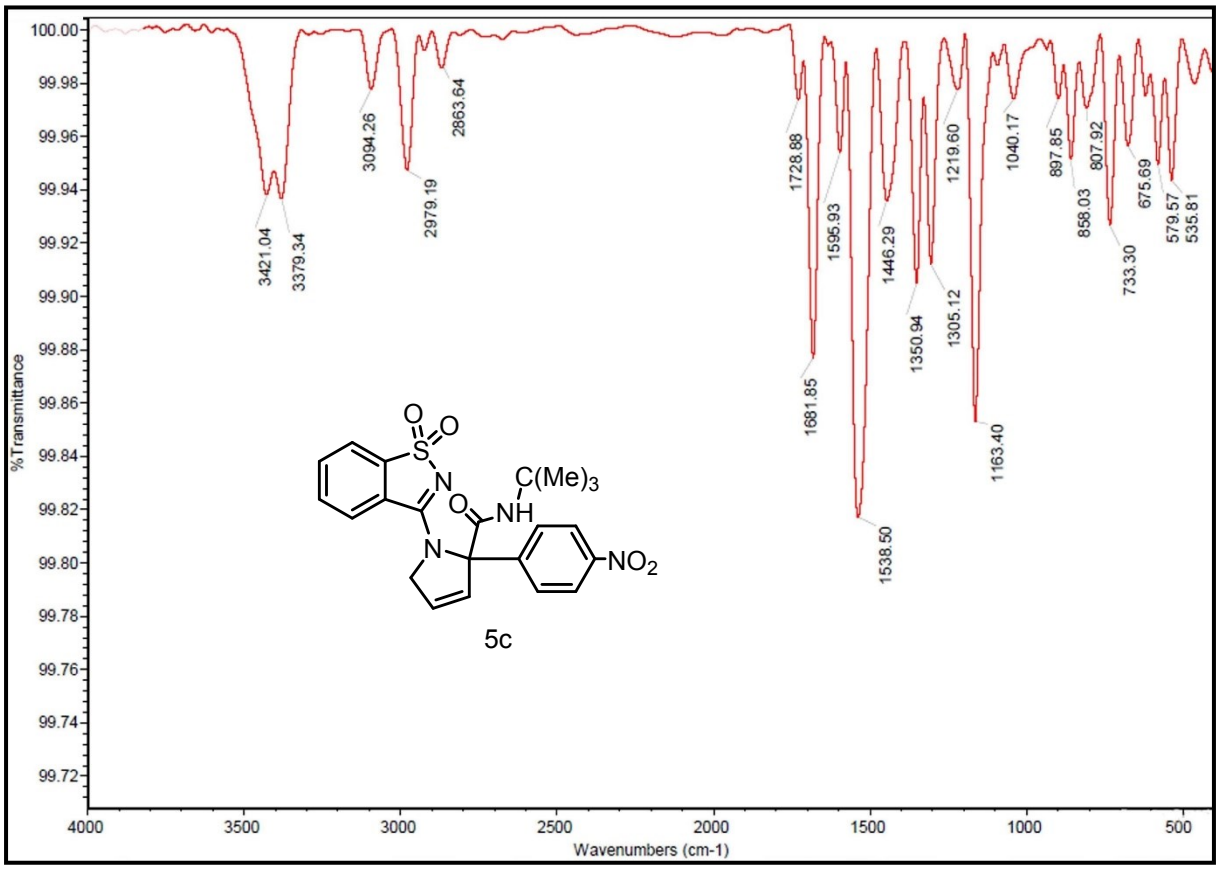




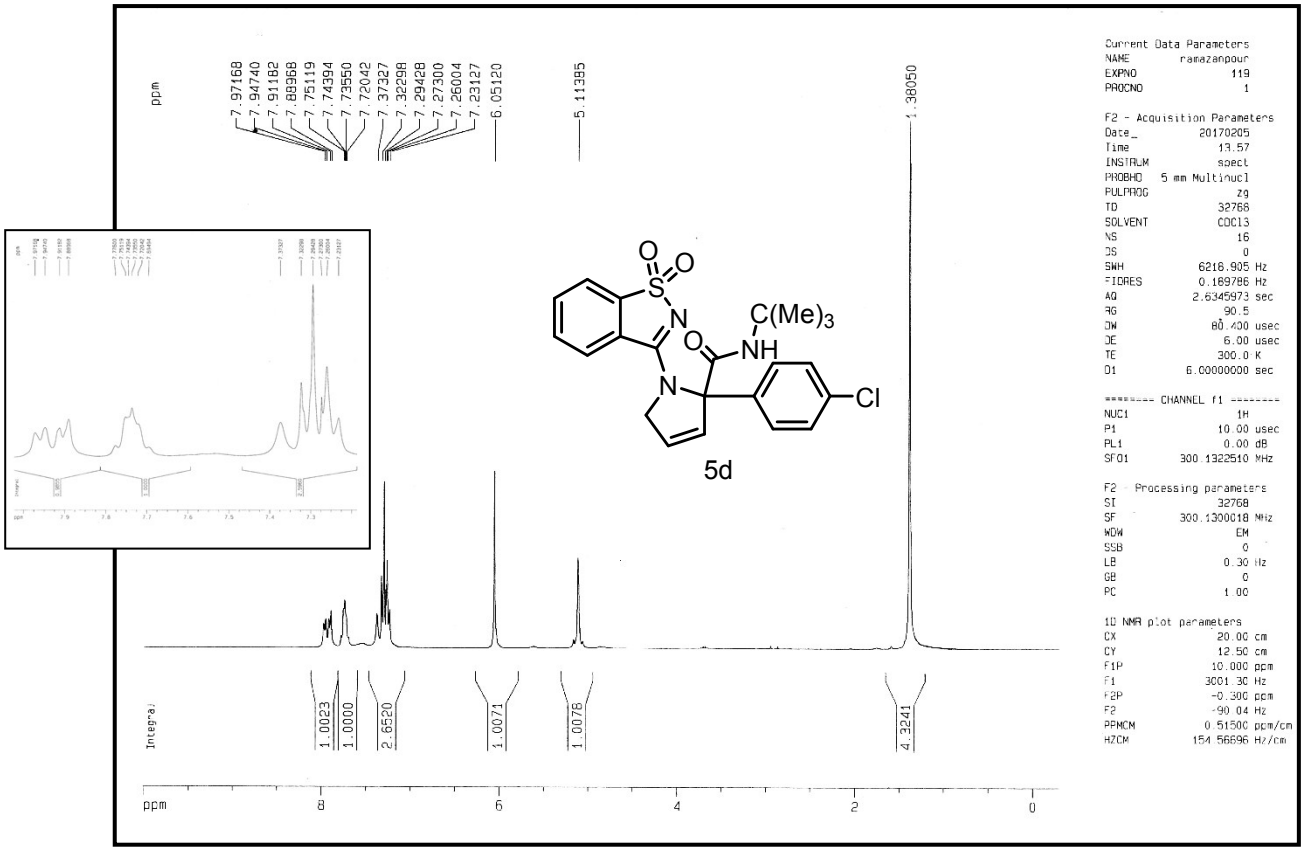
^{1}H NMR spectrum of compound **5c** (300 MHz, $CDCl_3$).



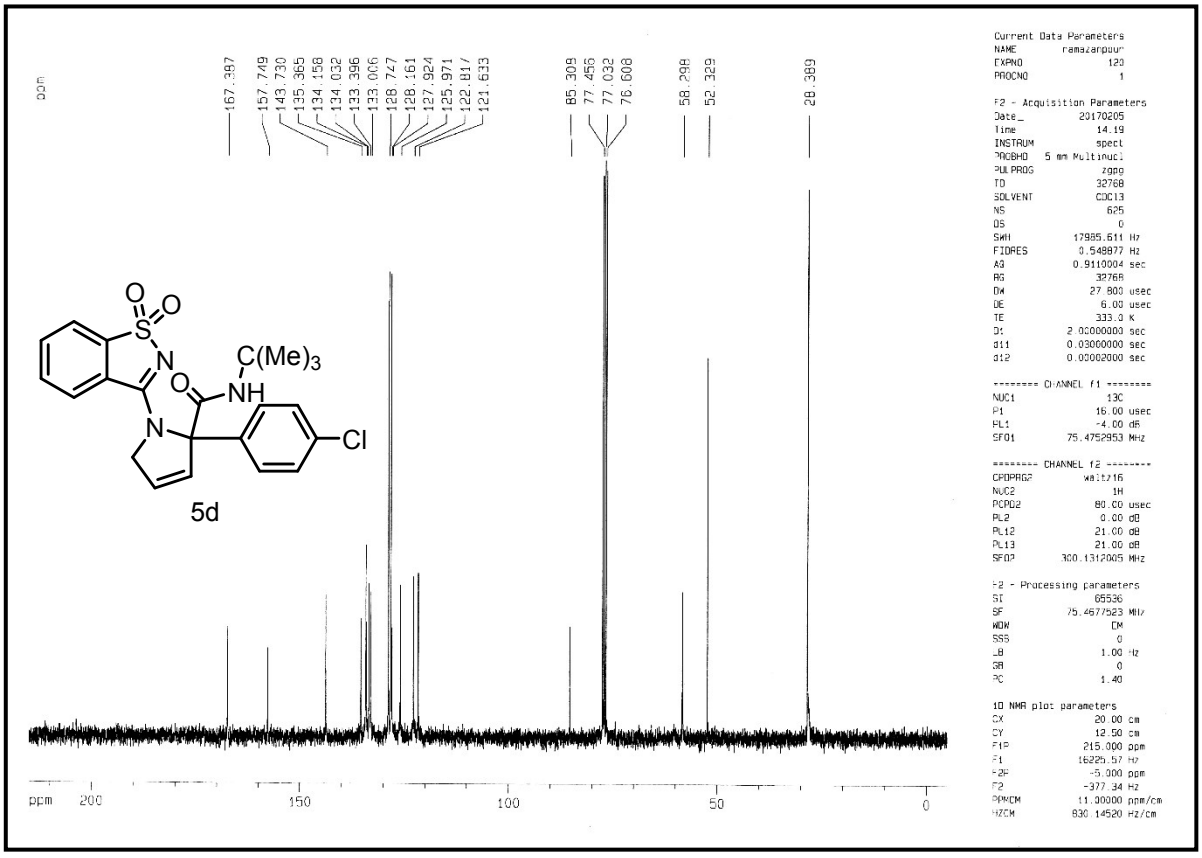
^{13}C NMR spectrum of compound **5c** (75 MHz, $CDCl_3$).



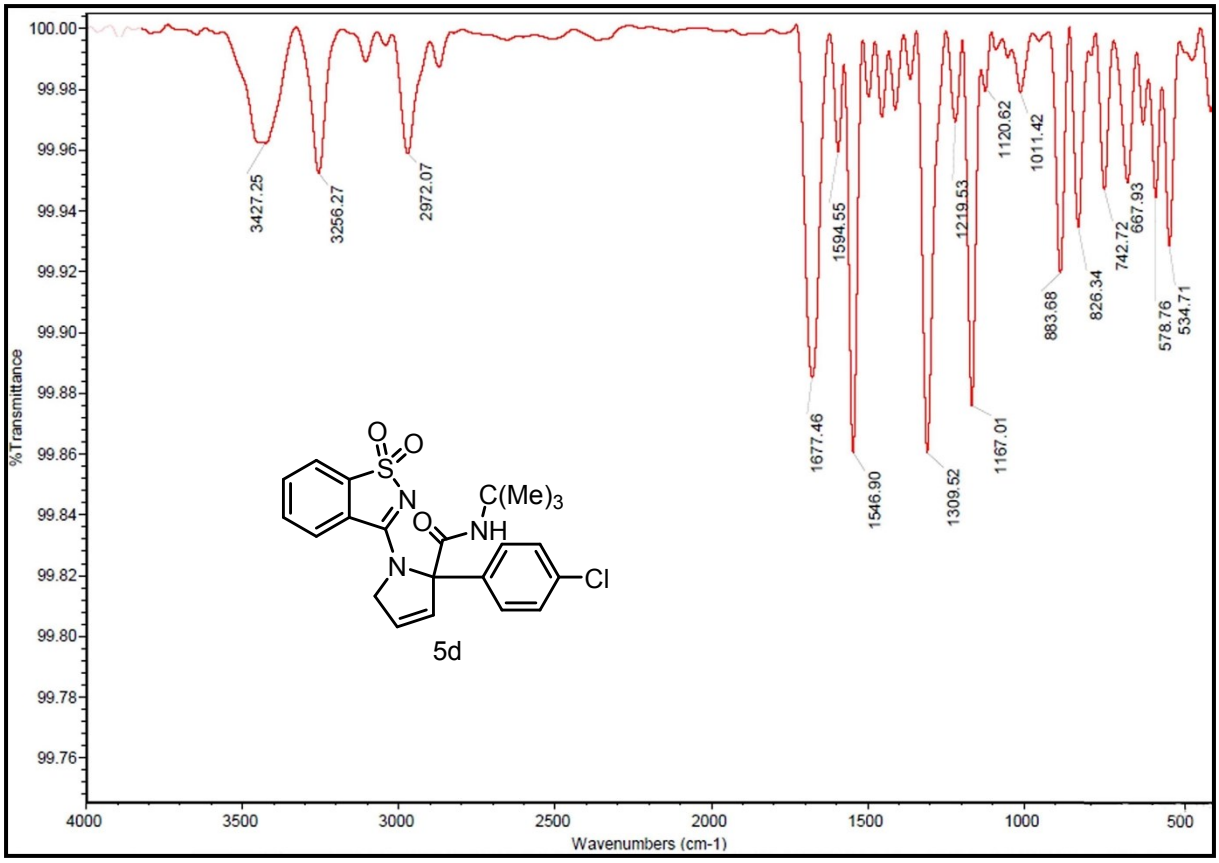
IR spectrum of compound **5c**.



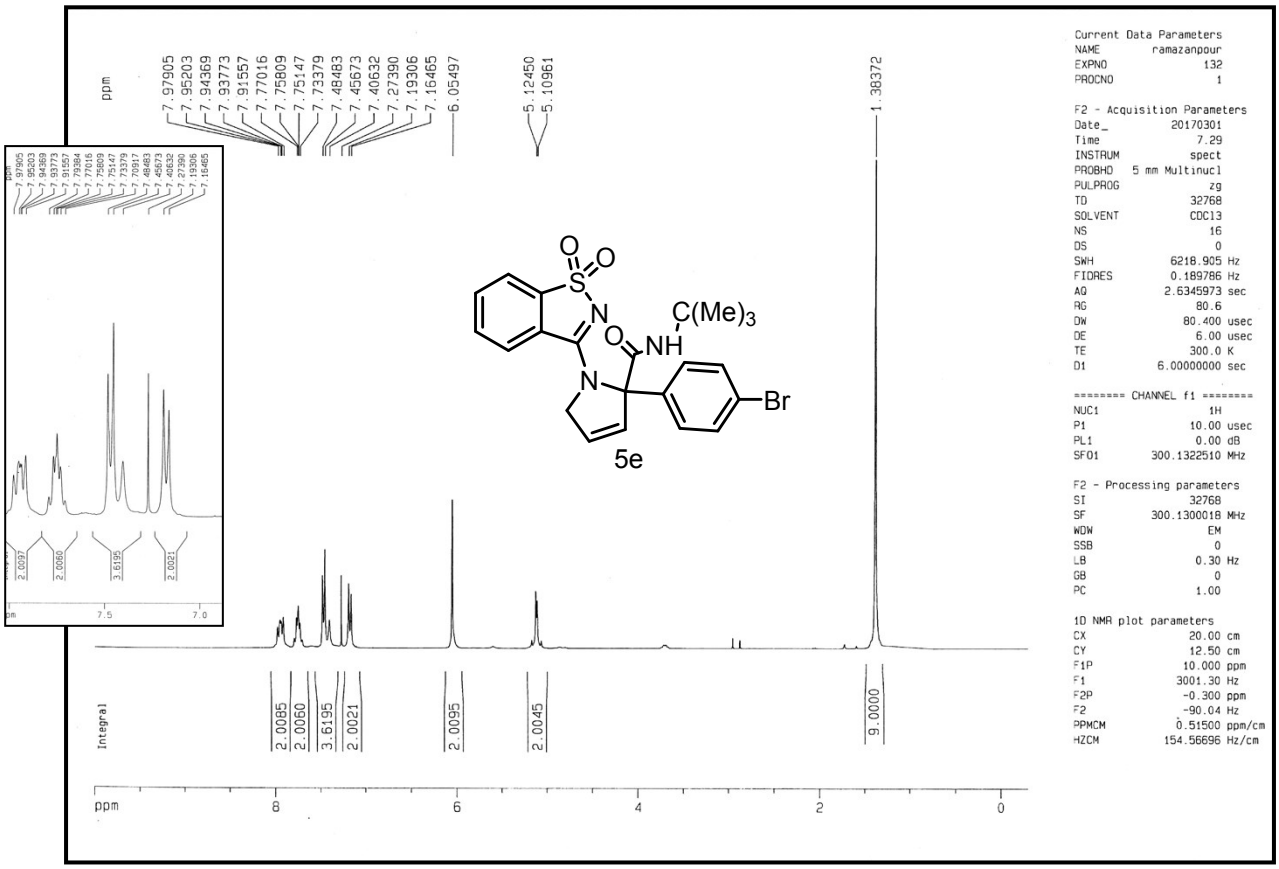
^{1}H NMR spectrum of compound **5d** (300 MHz, $CDCl_3$).



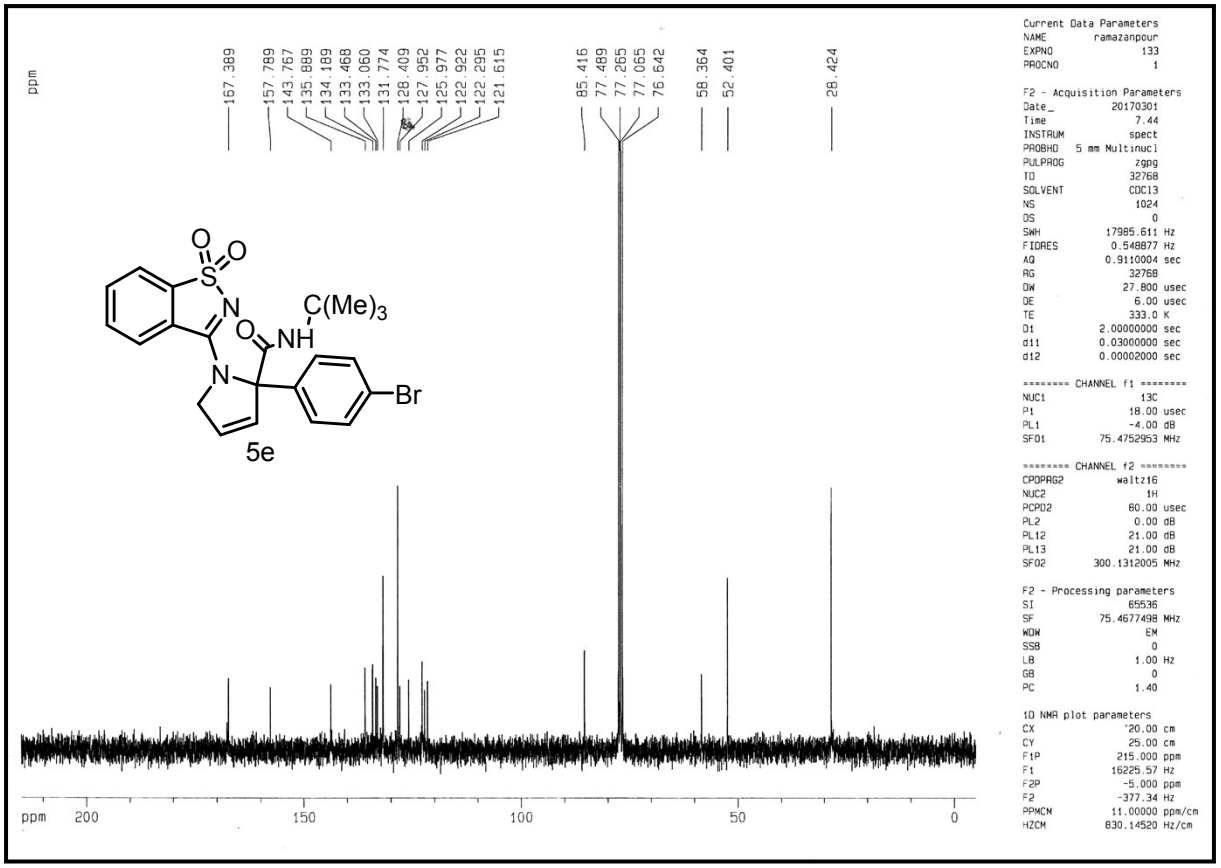
^{13}C NMR spectrum of compound **5d** (75 MHz, $CDCl_3$).



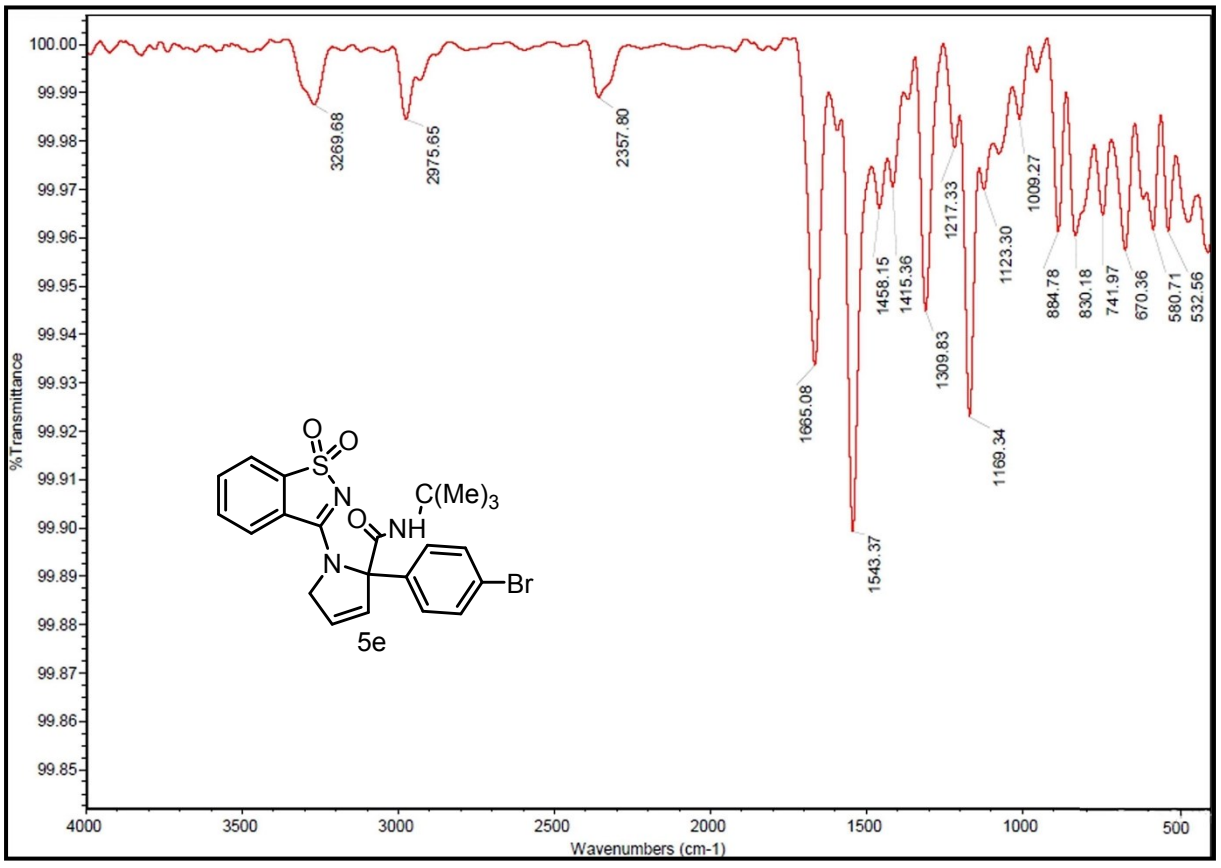
IR spectrum of compound **5d**.



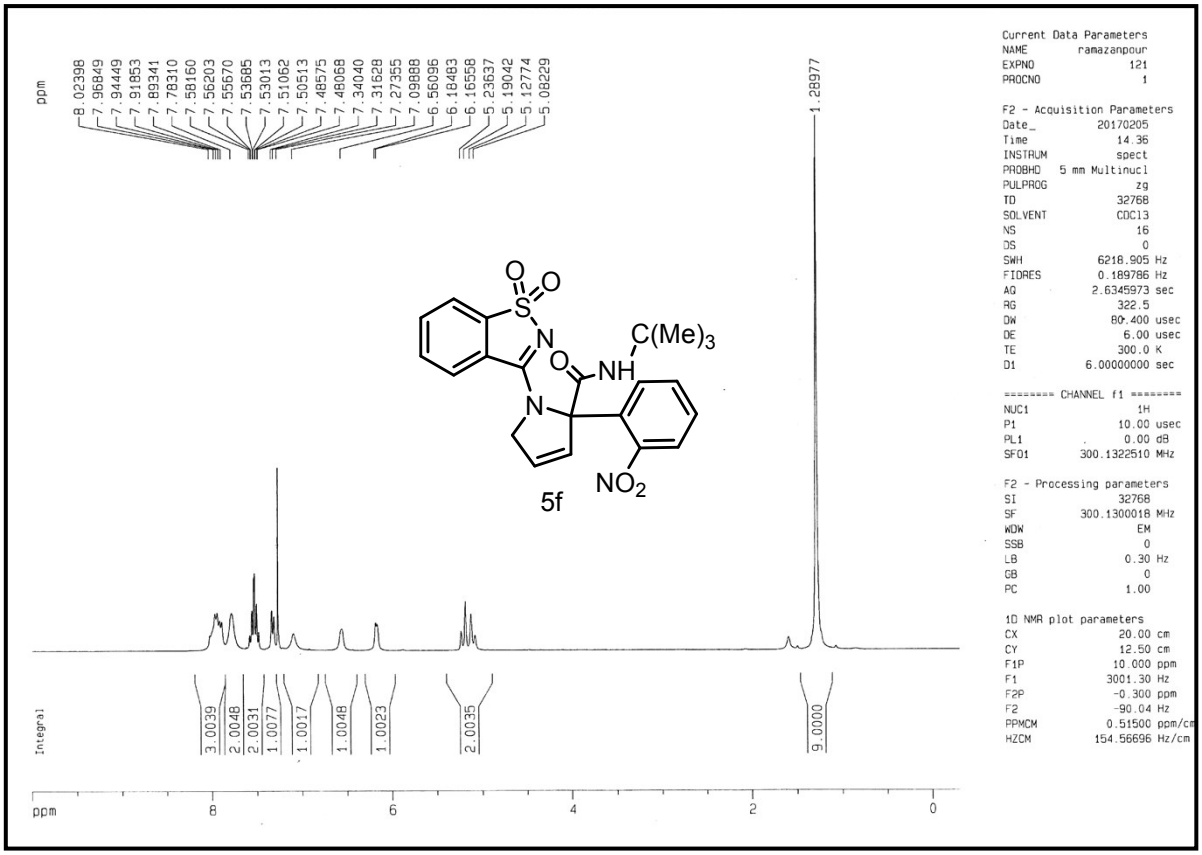
^{1}H NMR spectrum of compound **5e** (300 MHz, $CDCl_3$).



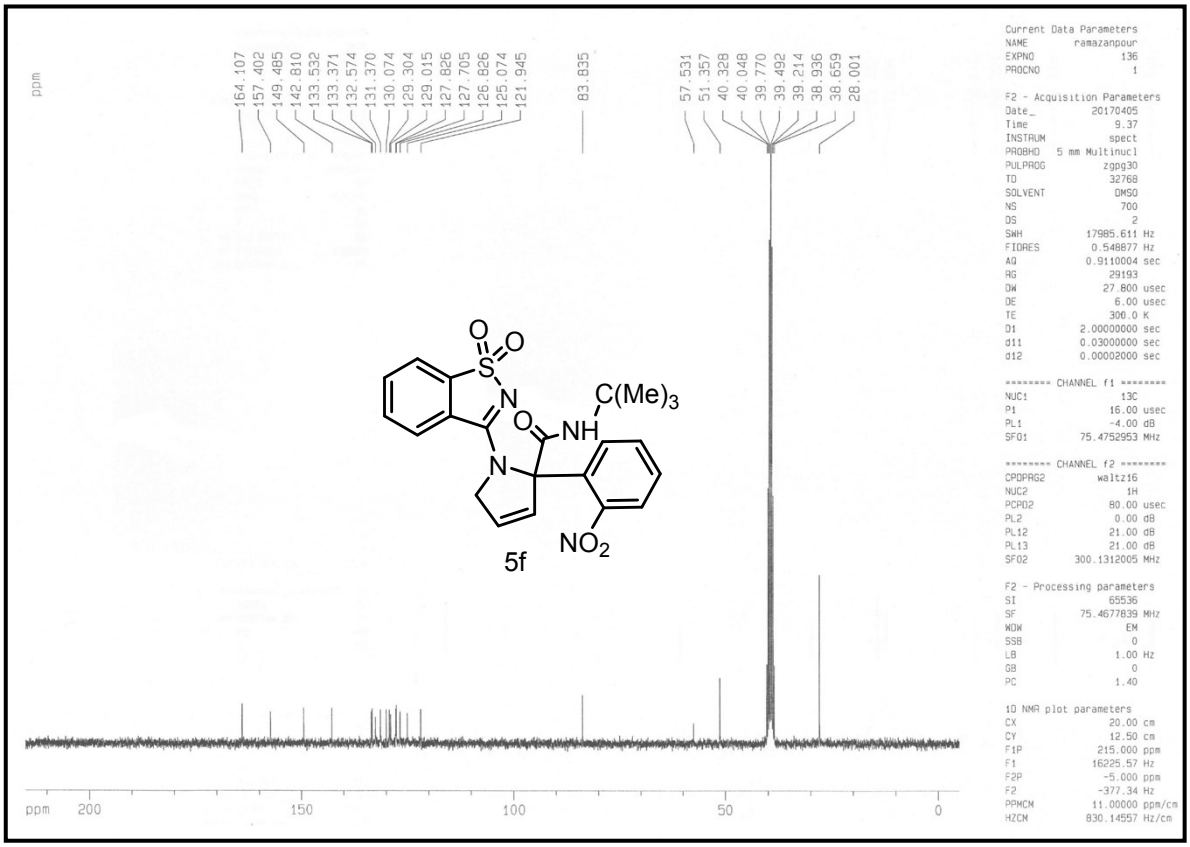
^{13}C NMR spectrum of compound **5e** (75 MHz, $CDCl_3$).



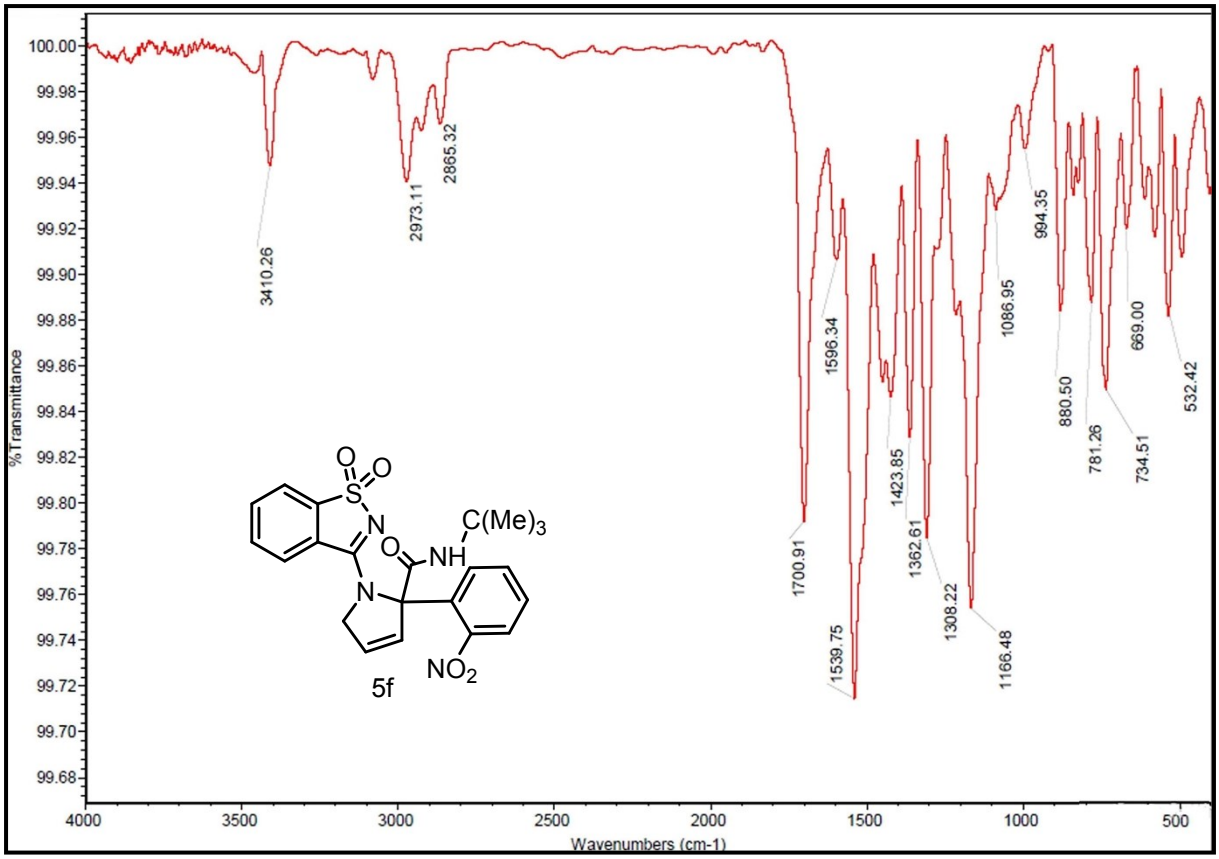
IR spectrum of compound **5e**.



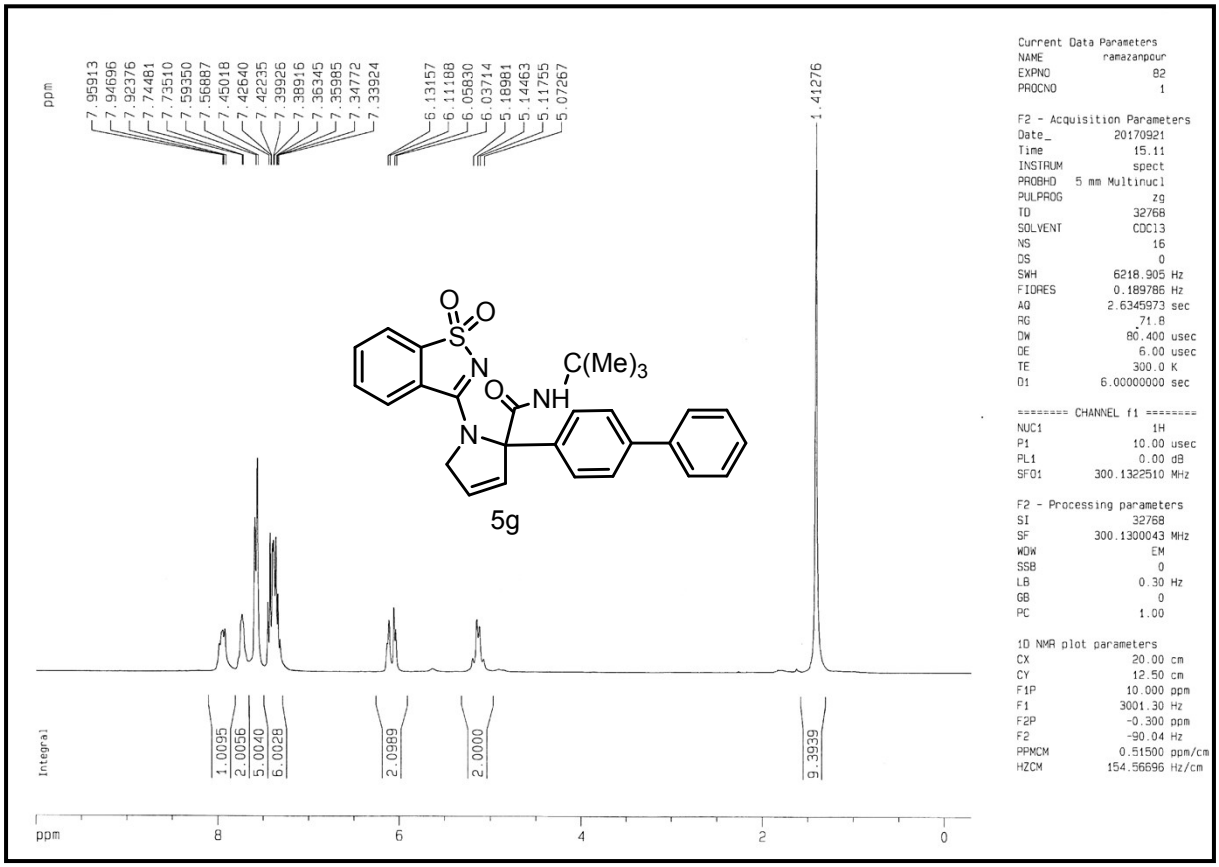
^{1}H NMR spectrum of compound **5f** (300 MHz, $CDCl_3$).



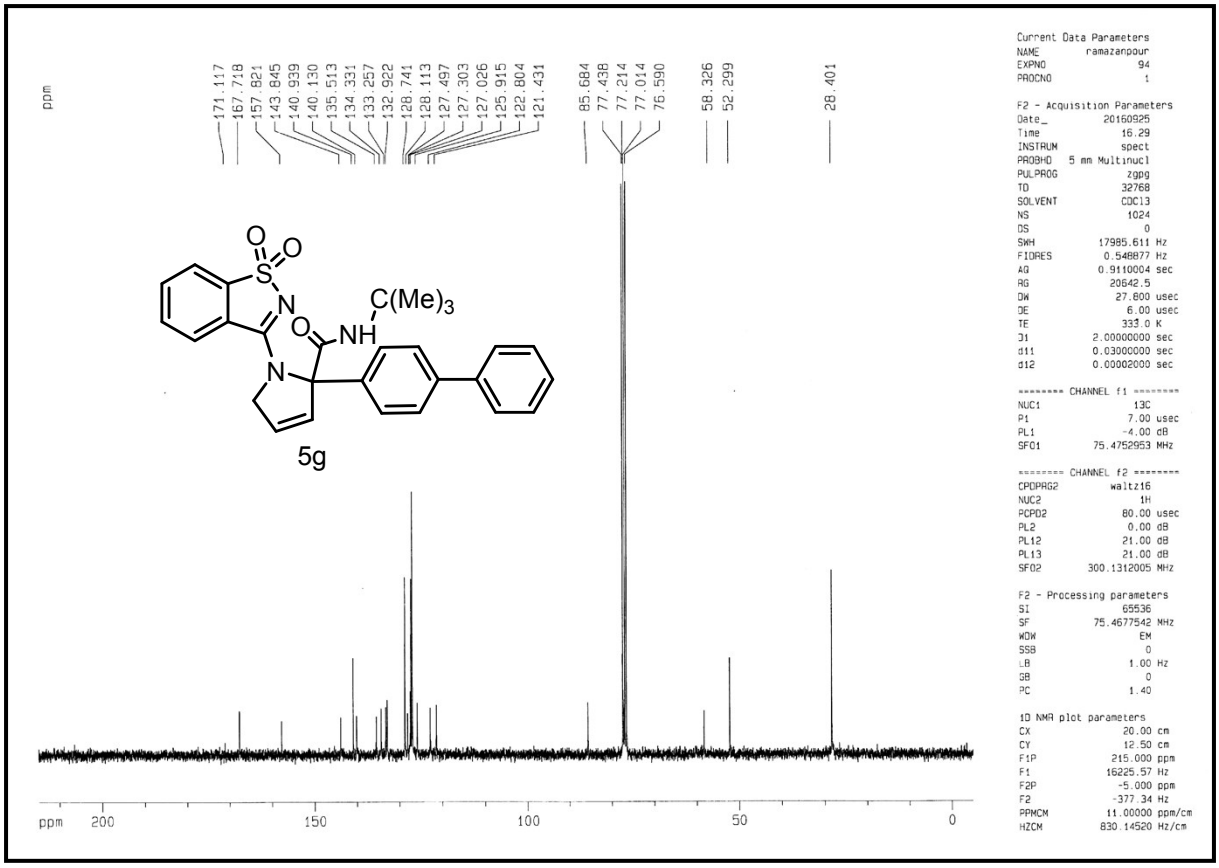
^{13}C NMR spectrum of compound **5f** (75 MHz, $CDCl_3$).



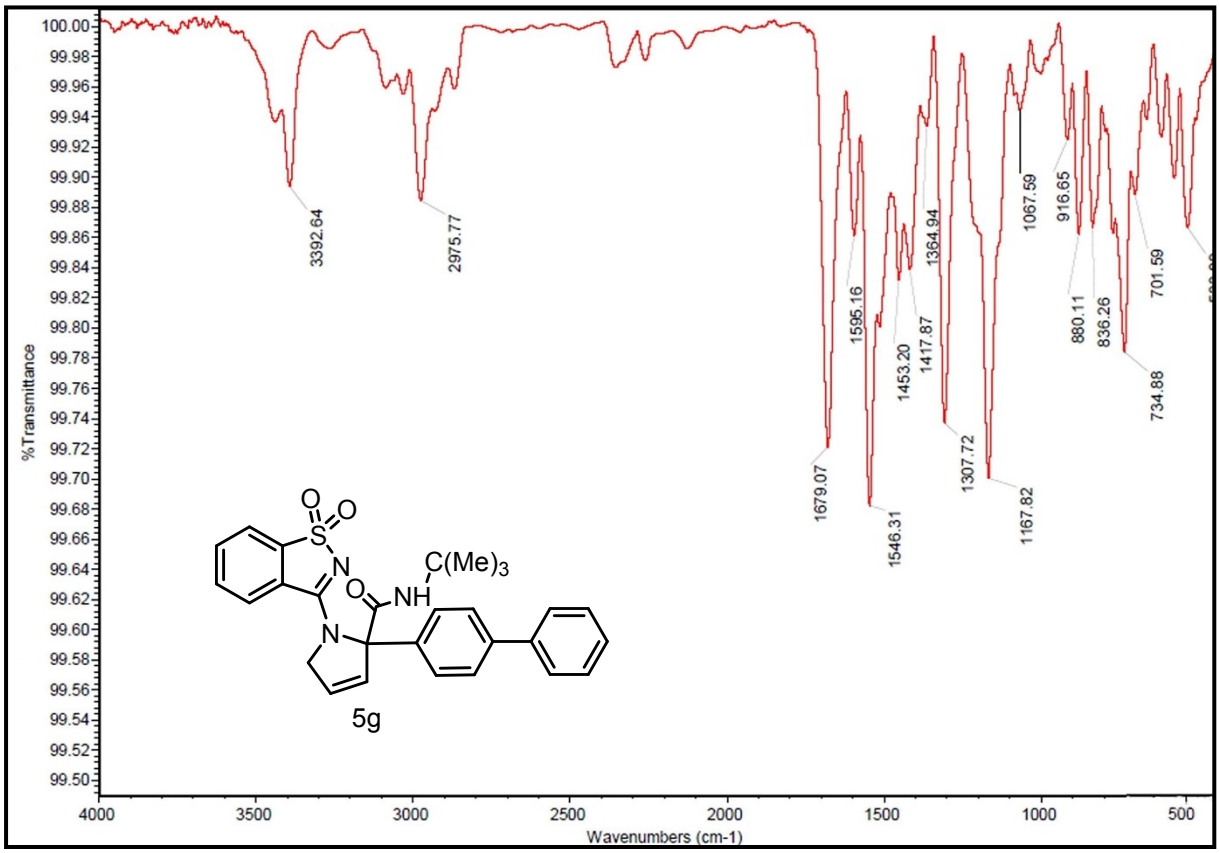
IR spectrum of compound **5f**.



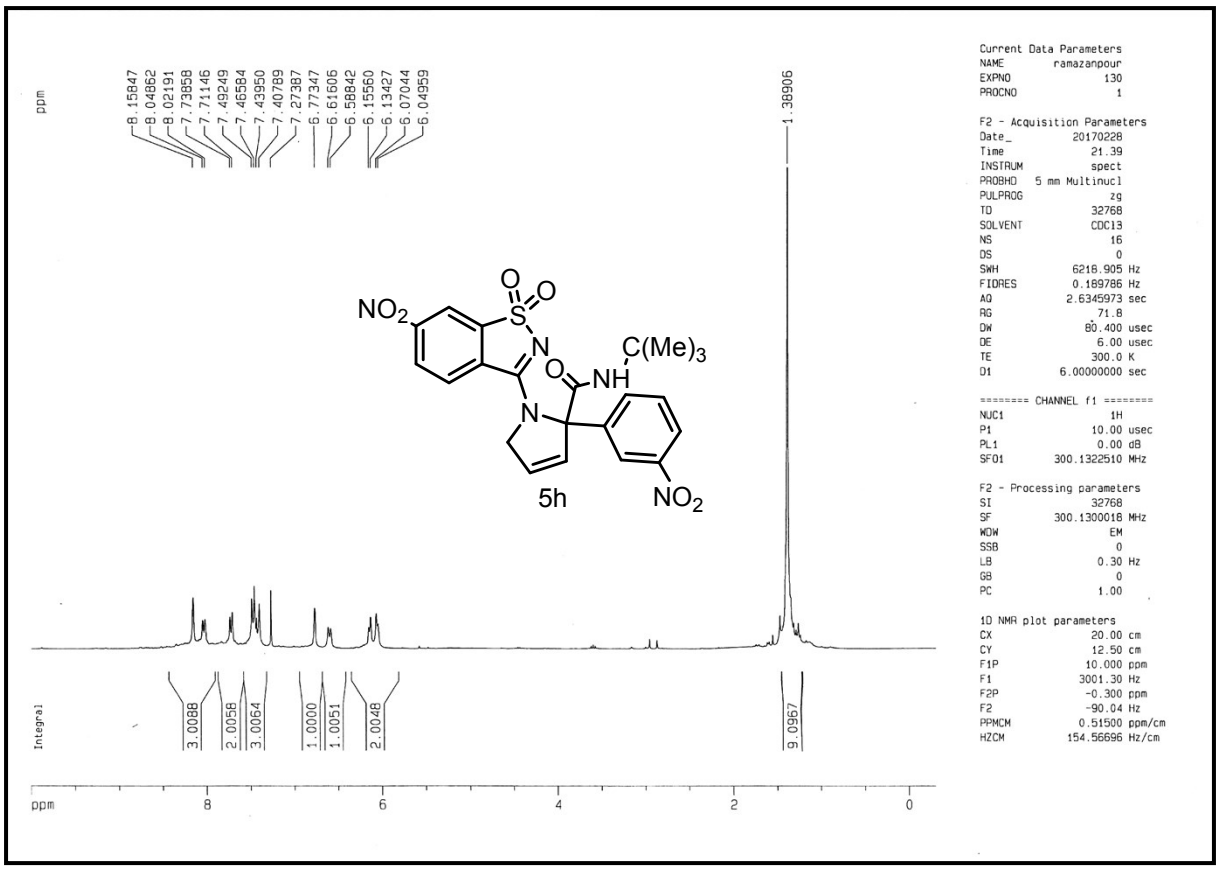
^{1}H NMR spectrum of compound **5g** (300 MHz, $CDCl_3$).



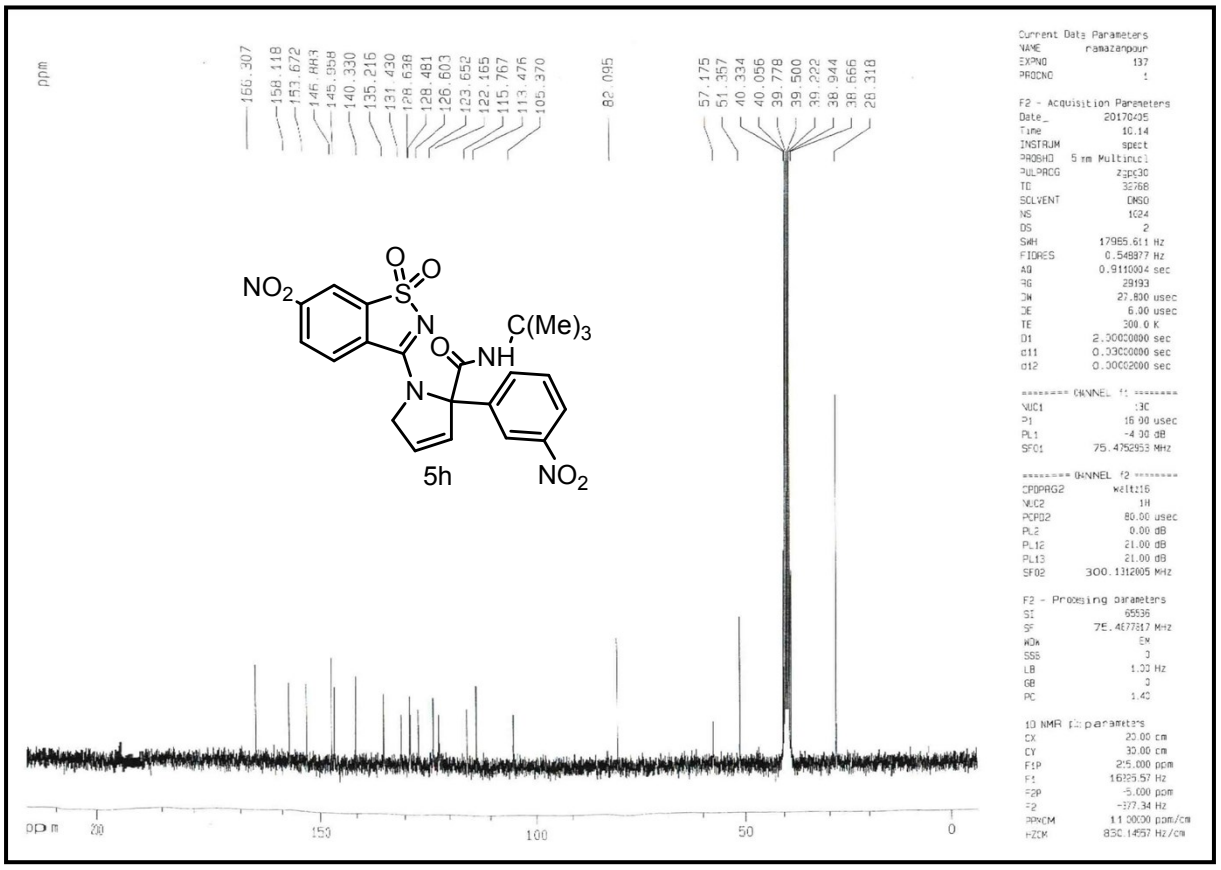
^{13}C NMR spectrum of compound **5g** (75 MHz, $CDCl_3$).



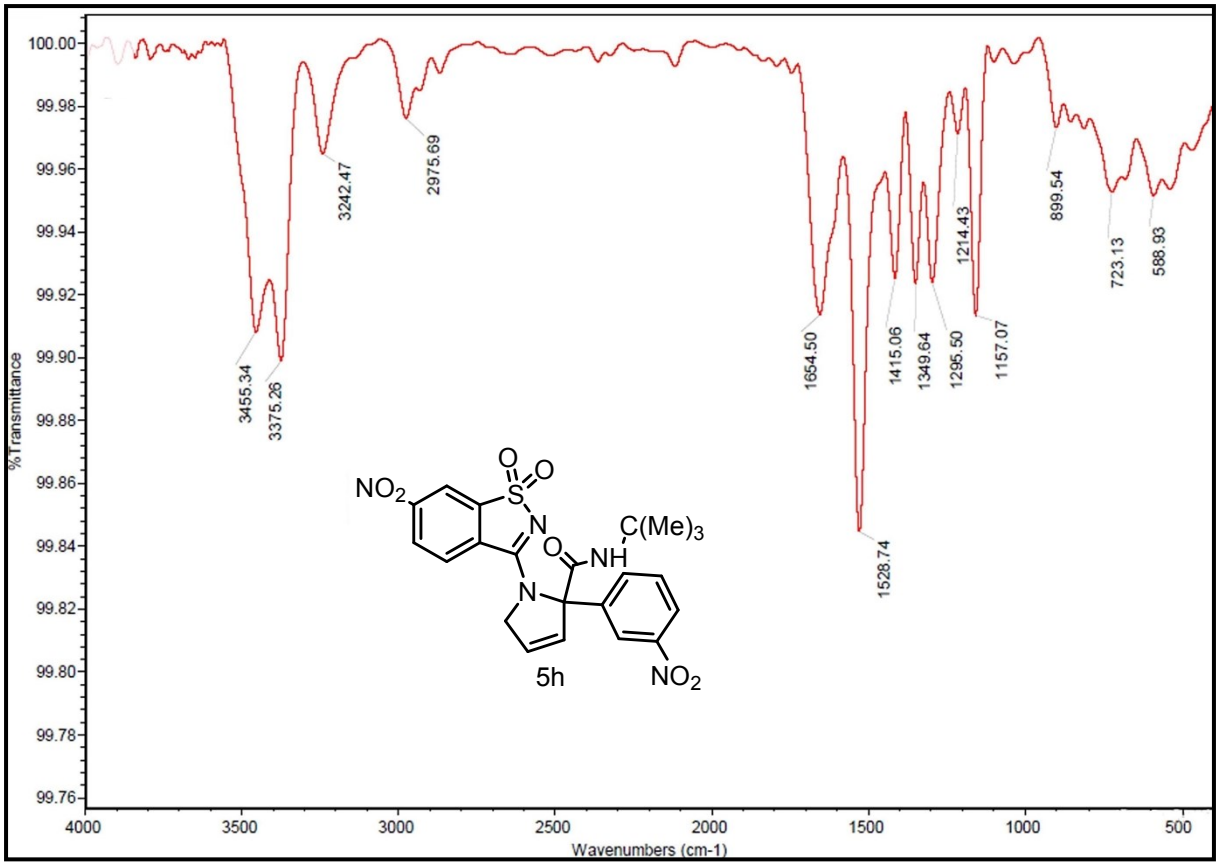
IR spectrum of compound **5g**.



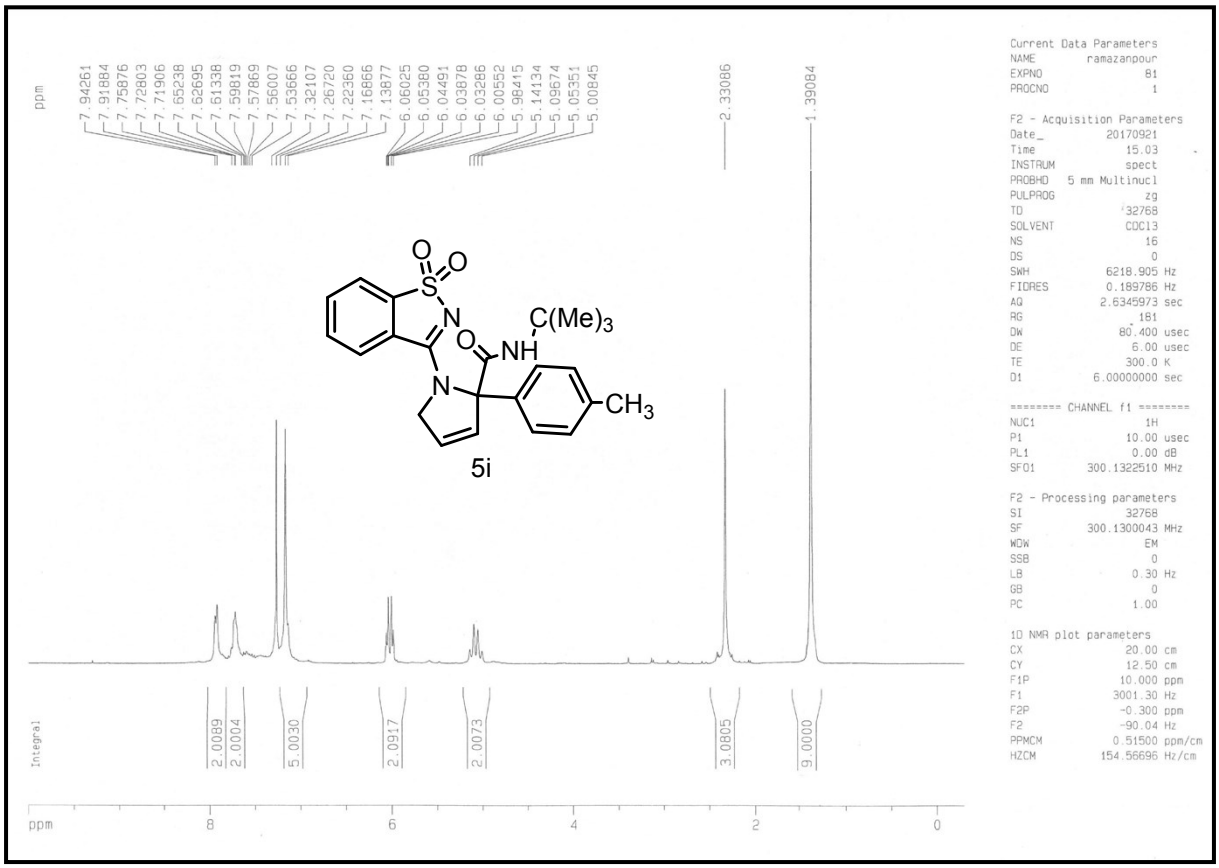
^{1}H NMR spectrum of compound **5h** (300 MHz, $CDCl_3$).



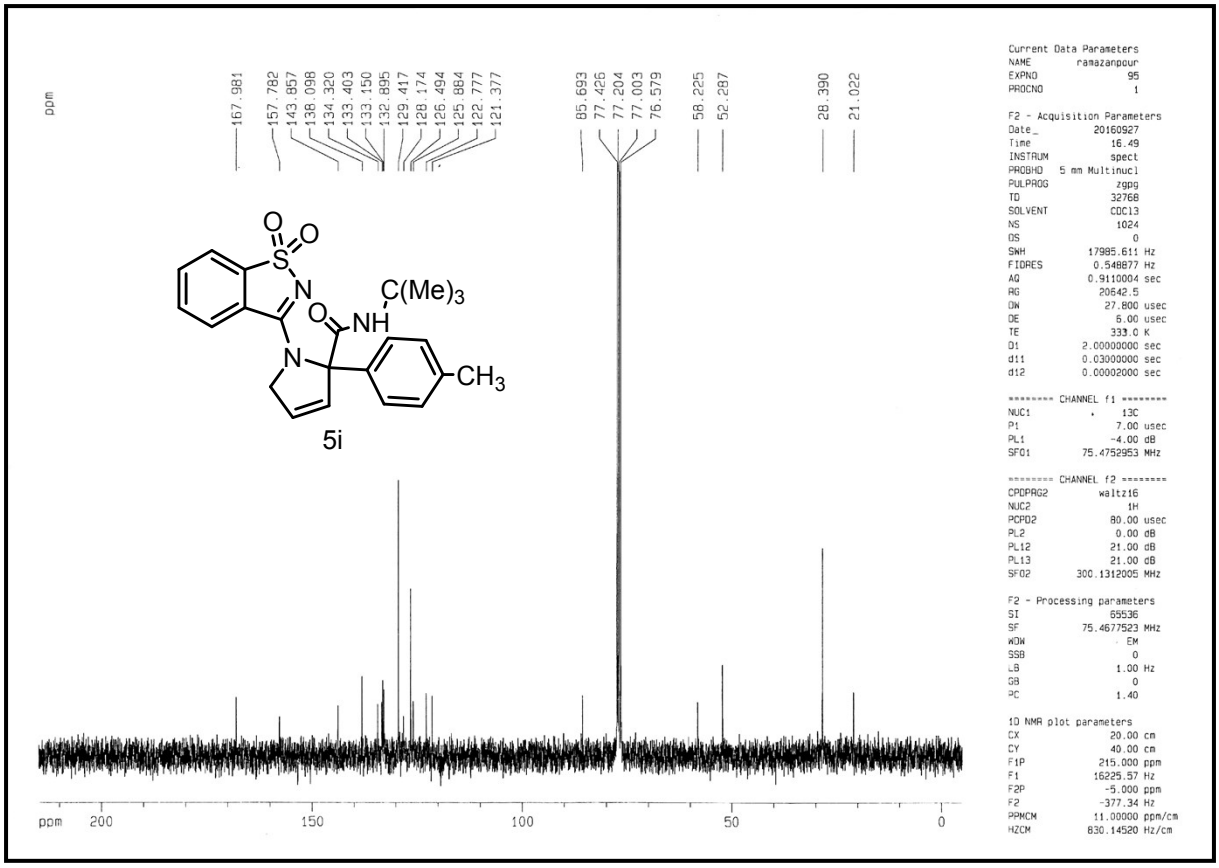
^{13}C NMR spectrum of compound **5h** (75 MHz, DMSO).



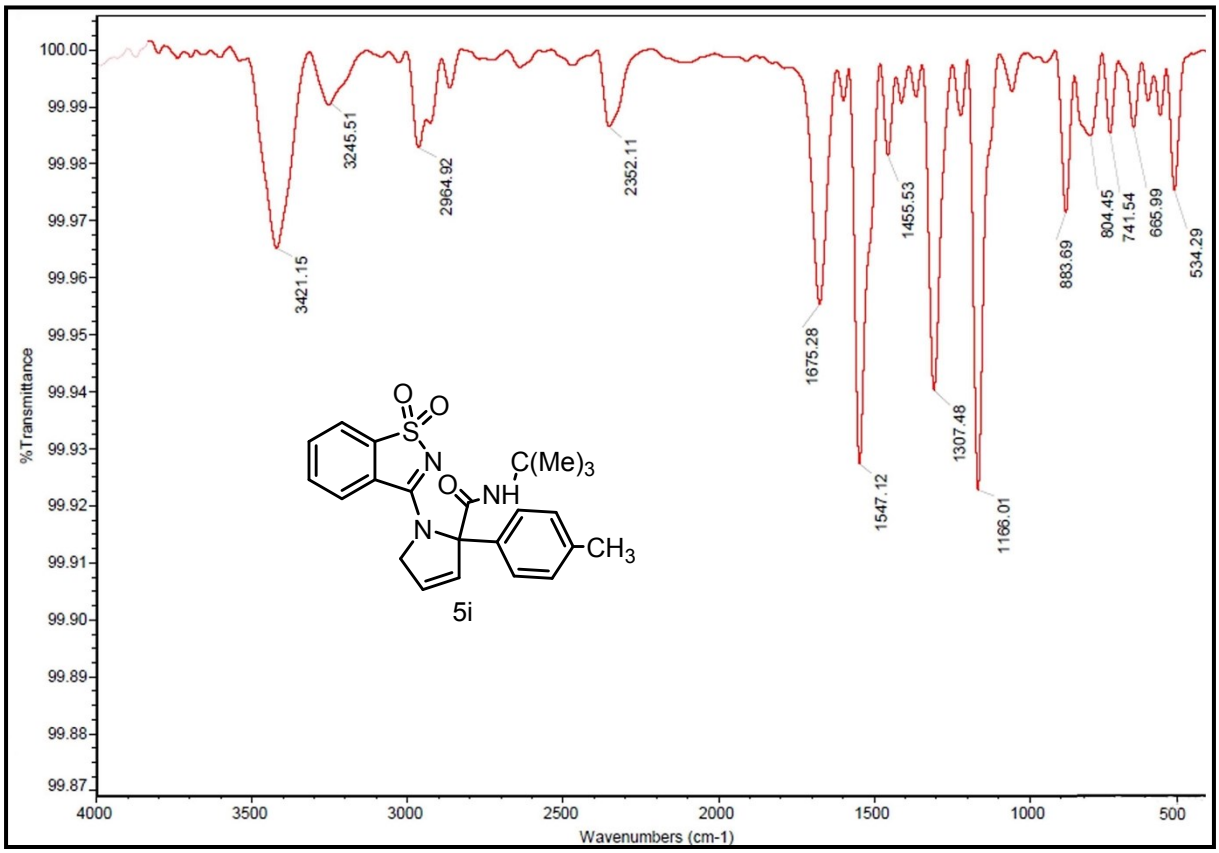
IR spectrum of compound **5h**.



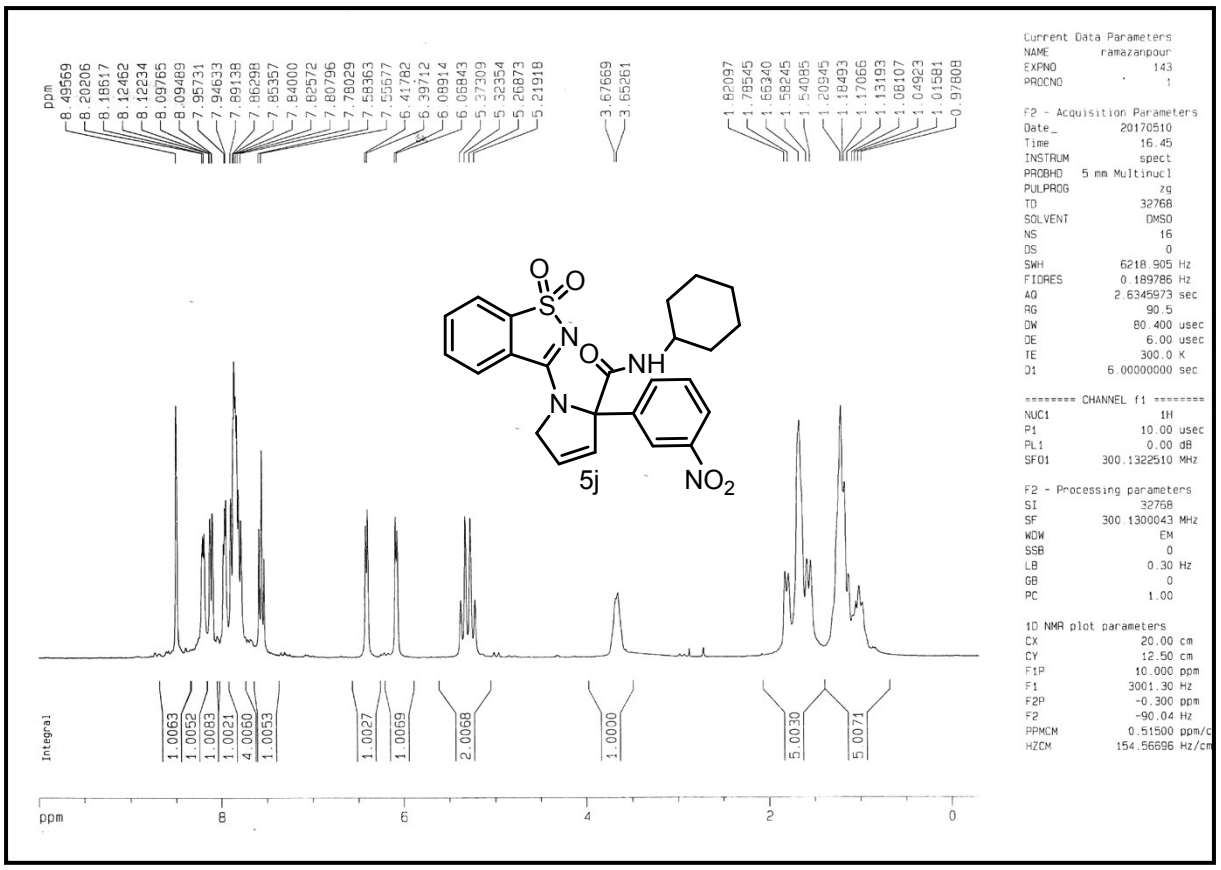
^{1}H NMR spectrum of compound **5i** (300 MHz, $CDCl_3$).



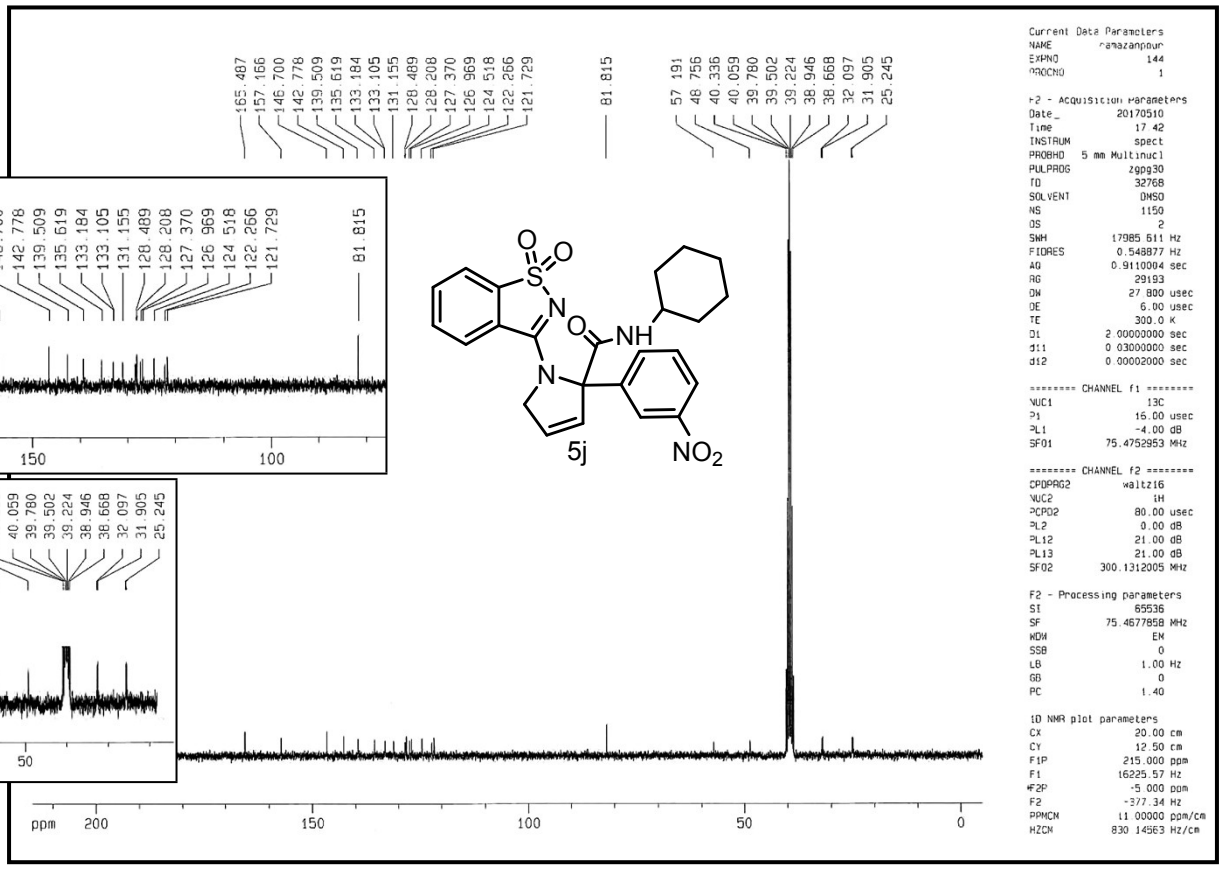
^{13}C NMR spectrum of compound **5i** (75 MHz, $CDCl_3$).



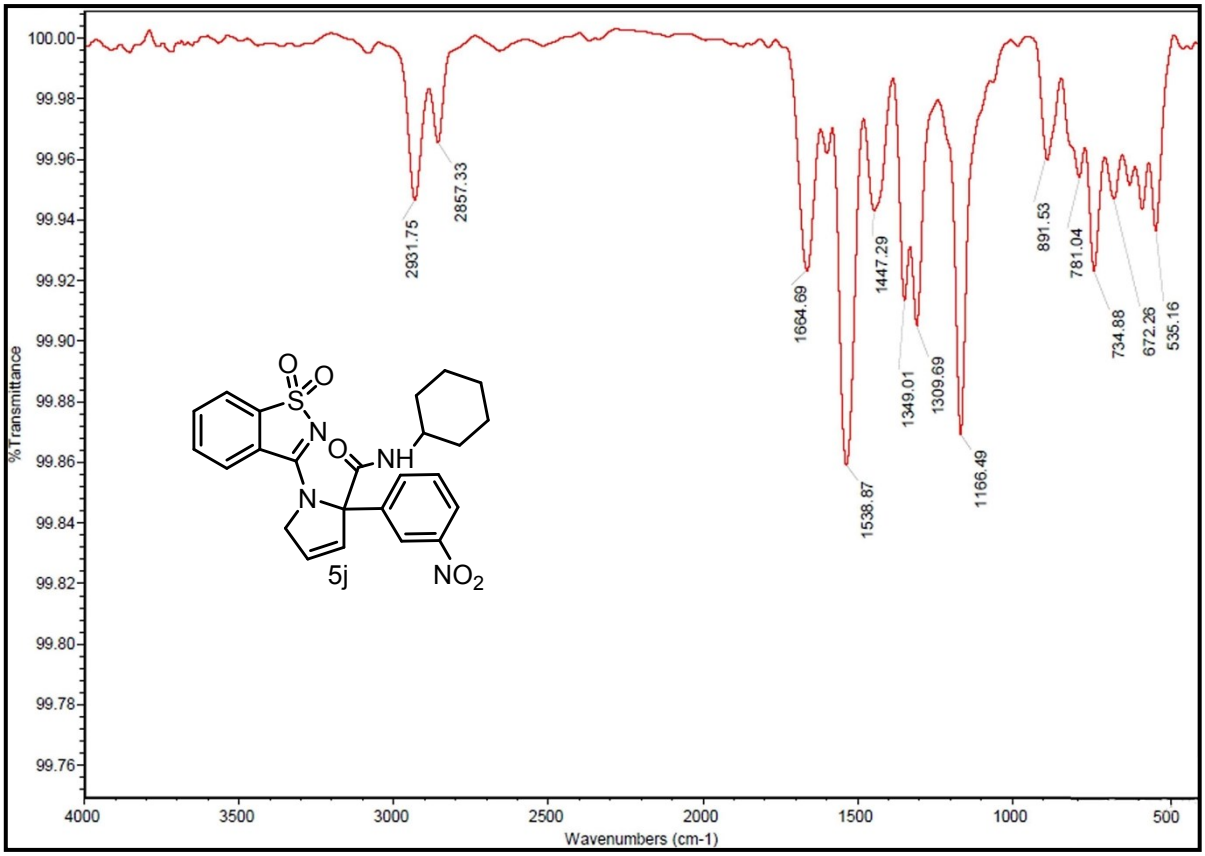
IR spectrum of compound **5i**.



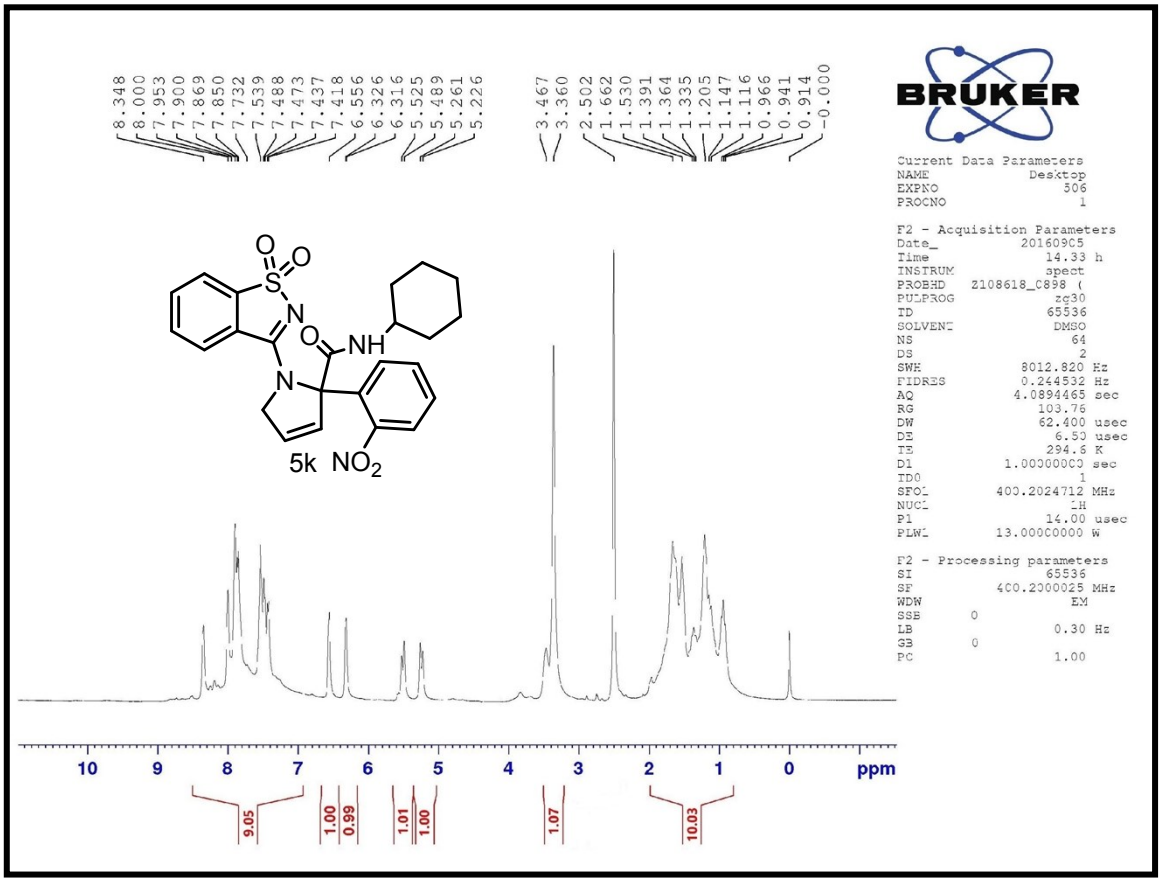
^{1}H NMR spectrum of compound **5j** (300 MHz, DMSO).



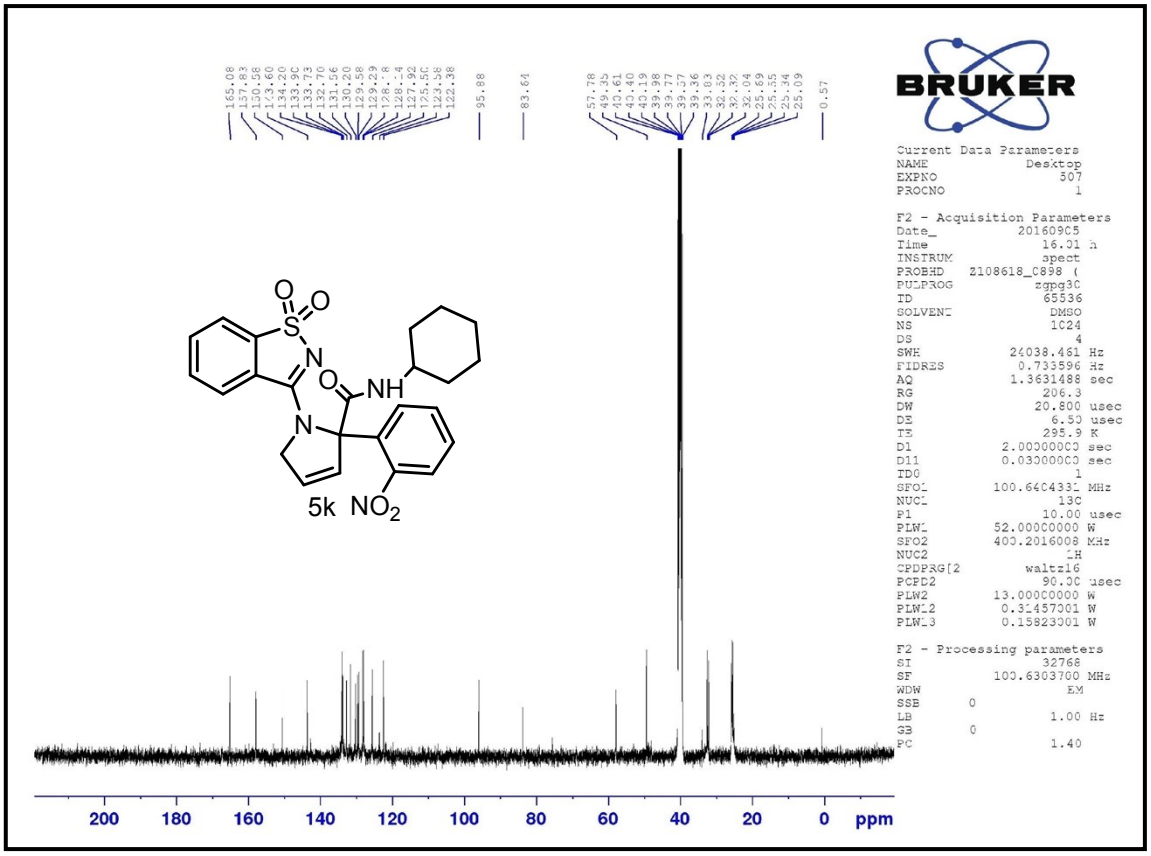
^{13}C NMR spectrum of compound **5j** (75 MHz, DMSO).



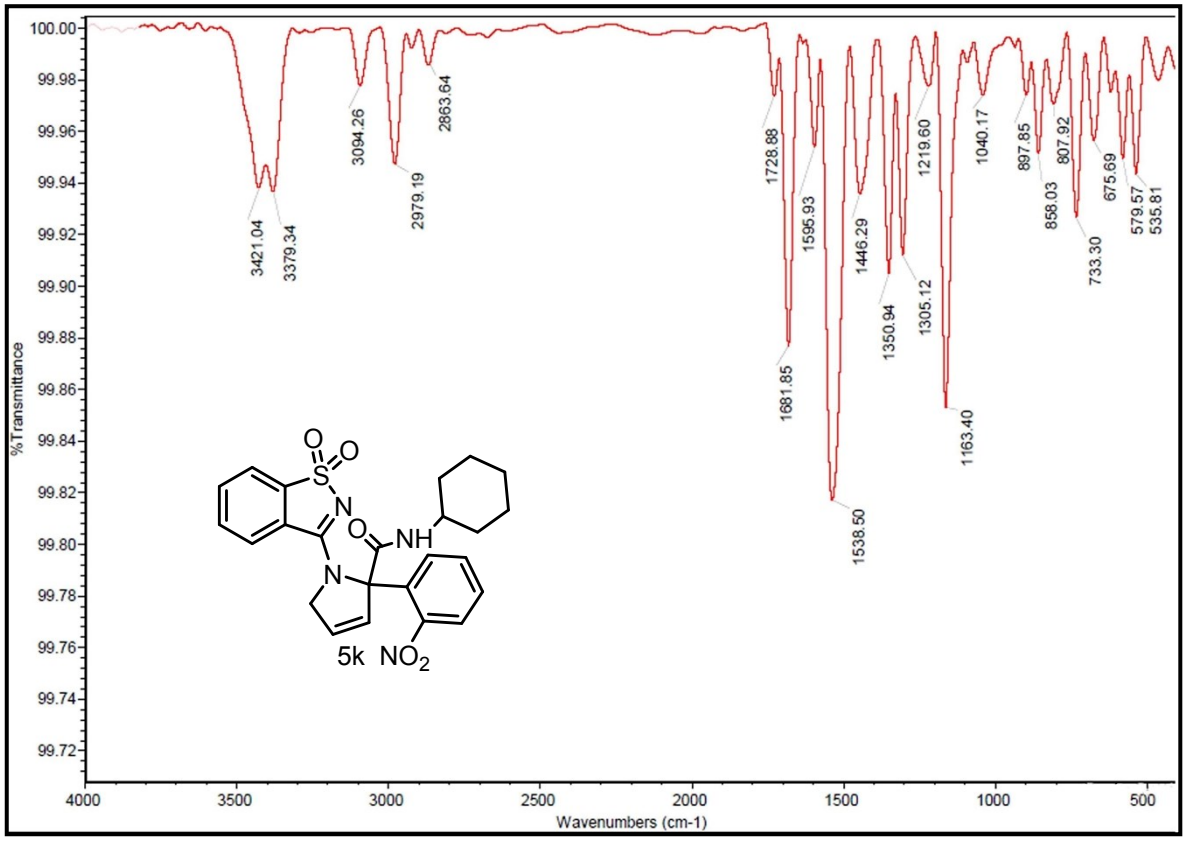
IR spectrum of compound **5j**.



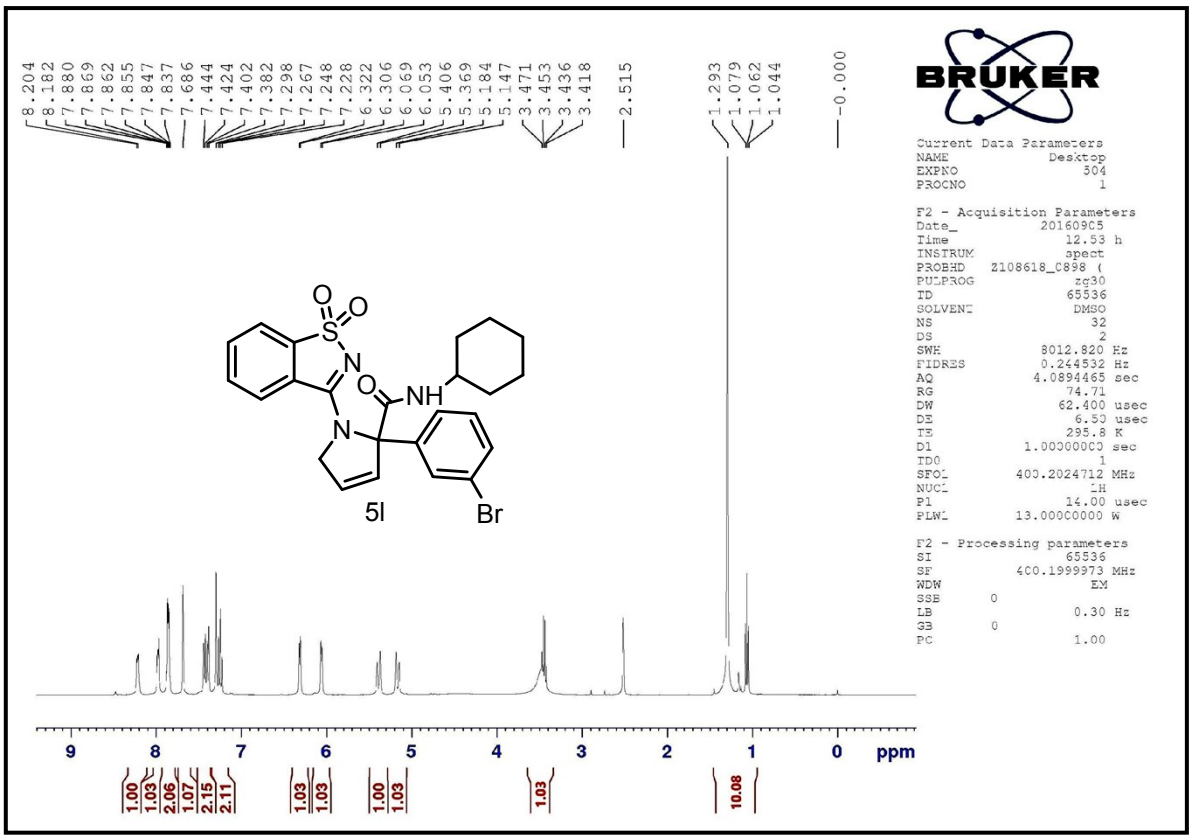
^{1}H NMR spectrum of compound **5k** (400 MHz, DMSO).



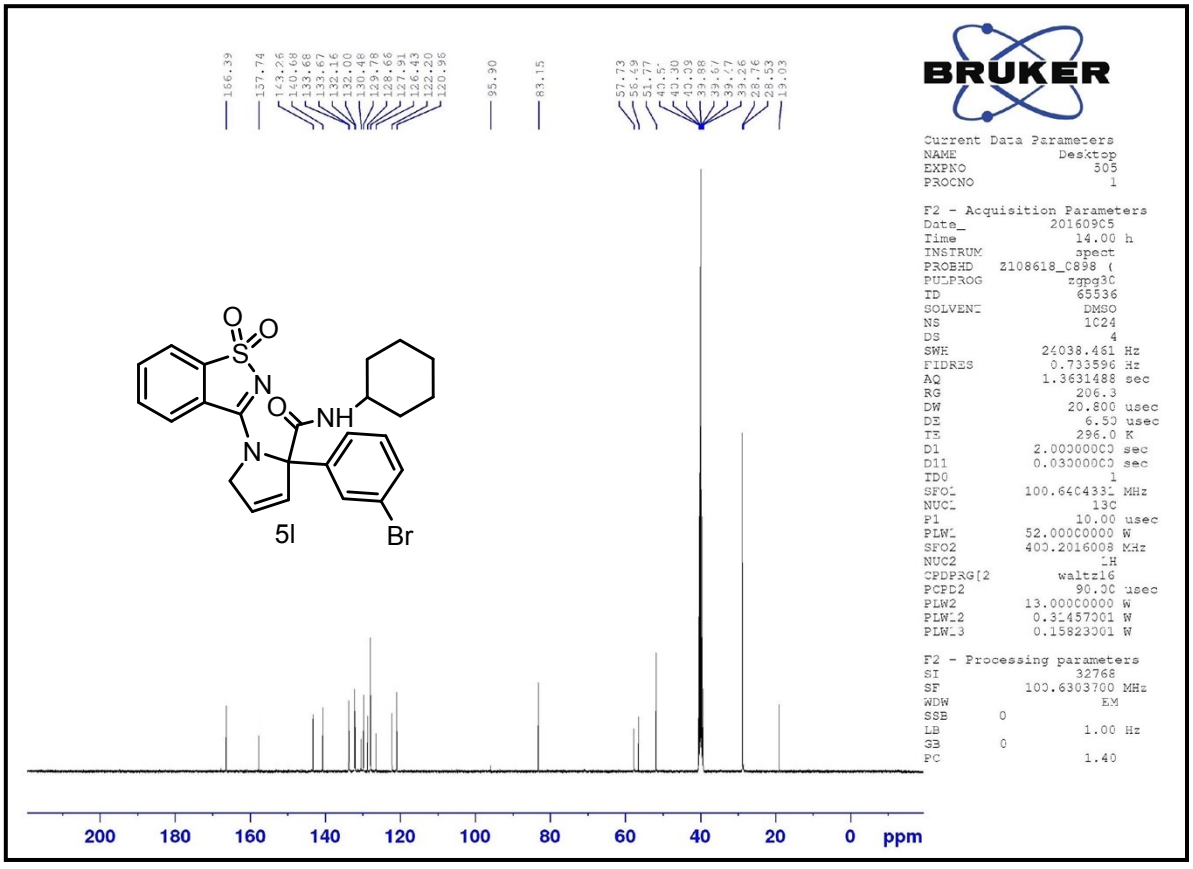
^{13}C NMR spectrum of compound **5k** (100 MHz, DMSO).



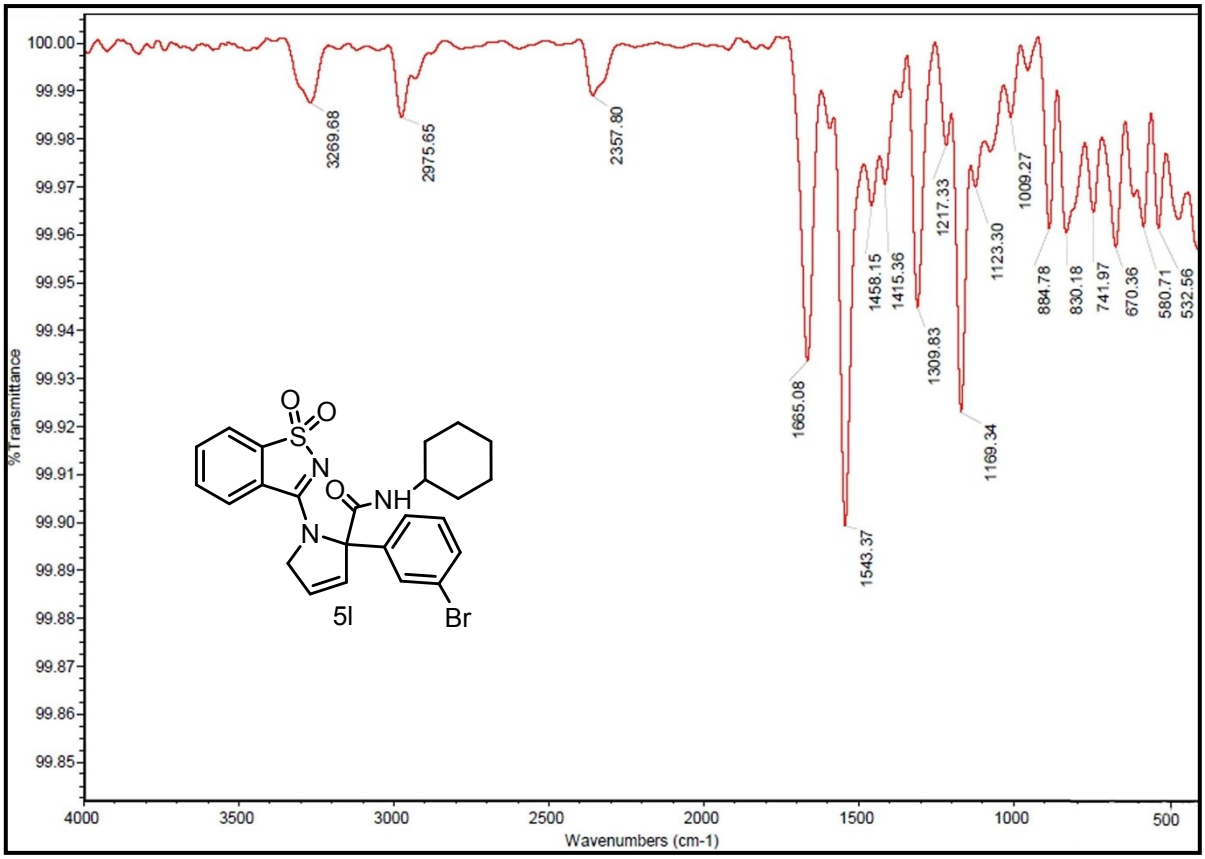
IR spectrum of compound **5k**.



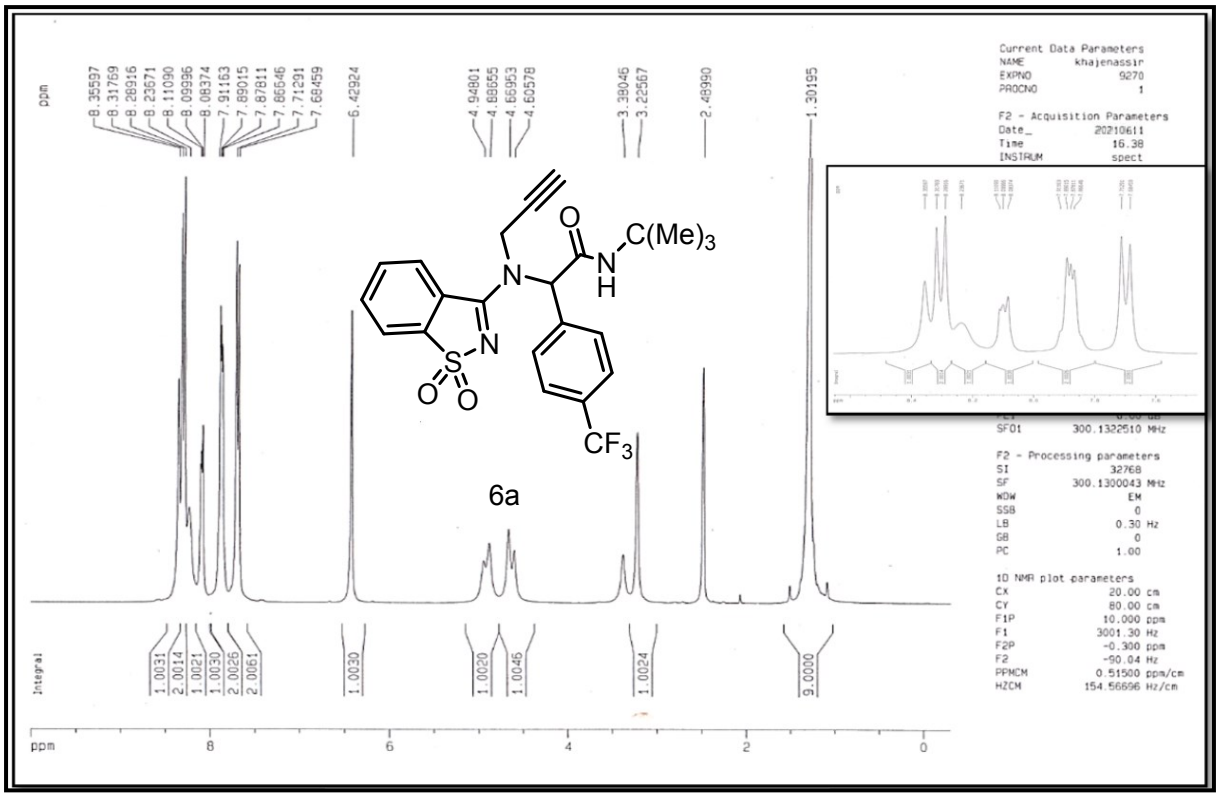
^{1}H NMR spectrum of compound **5l** (400 MHz, DMSO).



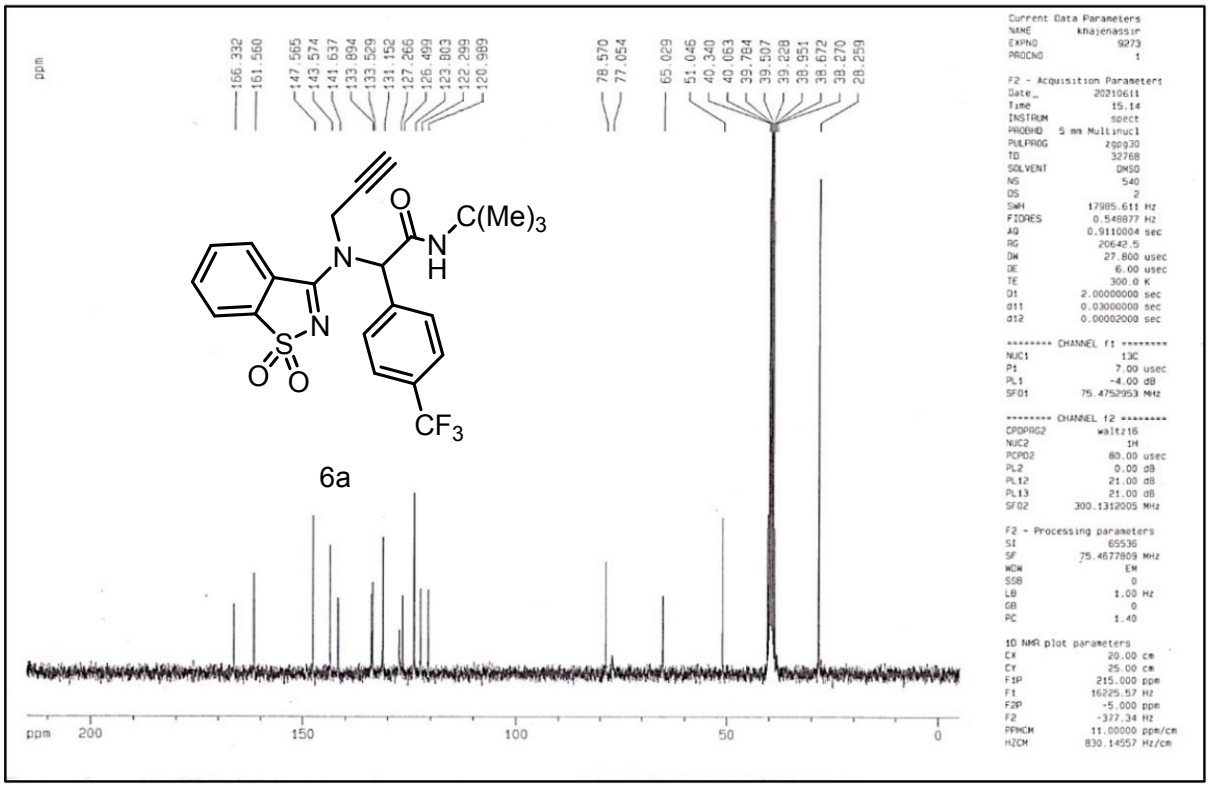
^{13}C NMR spectrum of compound **5l** (100 MHz, DMSO).



IR spectrum of compound **5l**.



^{1}H NMR spectrum of compound **6a** (300 MHz, DMSO).



^{13}C NMR spectrum of compound **6a** (75 MHz, DMSO).