Electronic Supplementary Material (ESI) for *Organic & Biomolecular Chemistry*
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Supplementary Information for

Direct construction of 2-alkylbenzo-1,3-azoles via C-H activation of alkanes for C-C and C-X (X = O, S) bond formation

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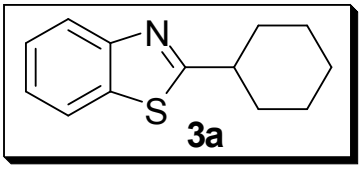
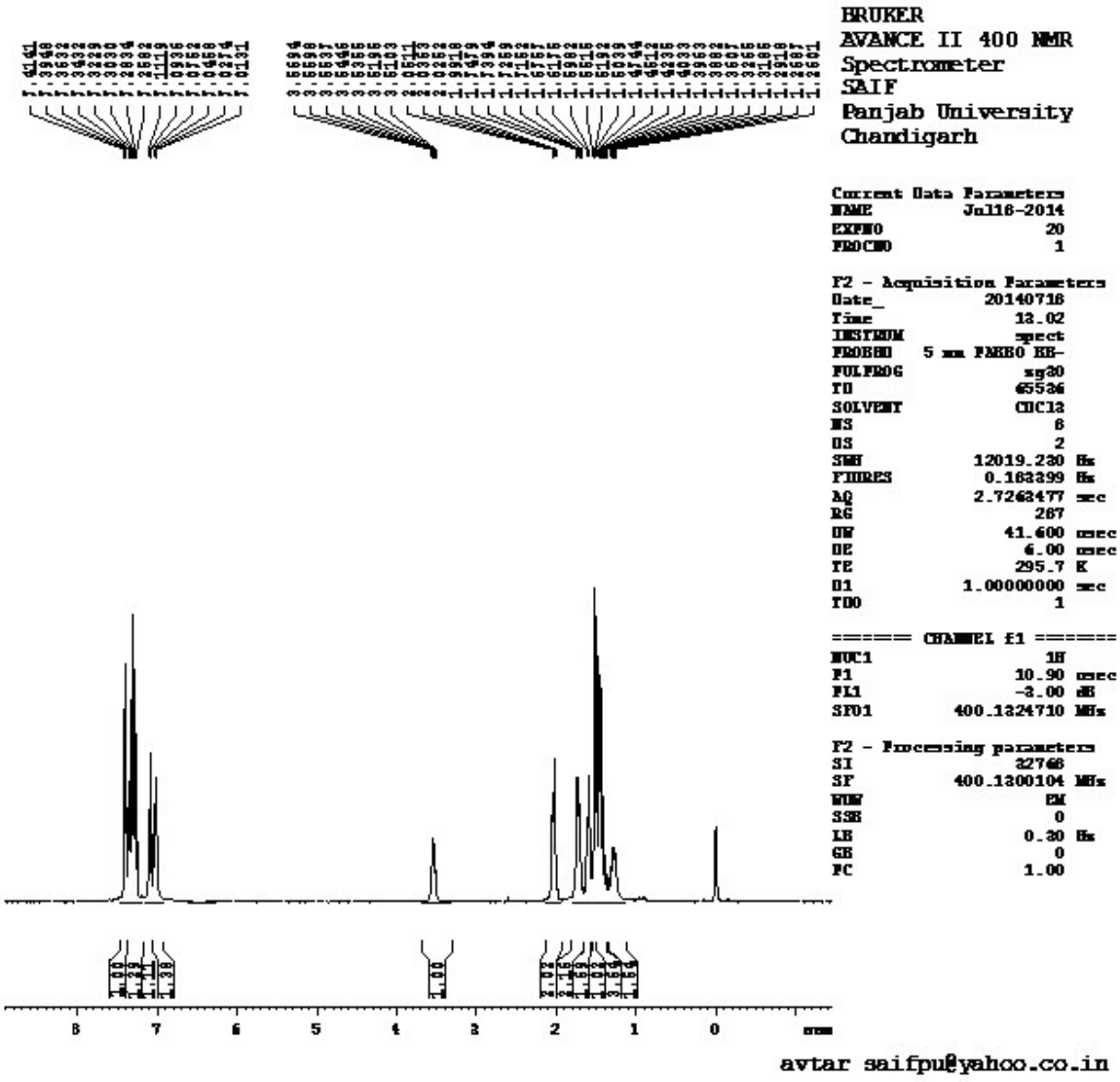
I. General Information: All commercially available reagents were used without further purification unless otherwise specified by a reference. Solvent was purified by the usual methods and stored over molecular sieves. All reactions were performed using oven-dried glassware under a nitrogen atmosphere. Organic solutions were concentrated using a Buchi rotary evaporator. Column chromatography was carried out over silica gel (Merck 100–200 mesh) and TLC was performed using silica gel GF254 (Merck) plates. Melting point (m.p.) was determined by open glass tube. ^{1}H (400 MHz), ^{13}C (100 MHz) NMR spectra were recorded on a Bruker AVII spectrometer in $CDCl_3$ using TMS as internal reference. All chemical shifts are reported in δ/ppm and coupling constants (*J*) in Hertz (Hz). MS (EI) spectra were recorded double focusing mass spectrometer.

II. General procedure for the synthesis of 2-alkylbenzo-1,3-azoles: Charged a round bottom flask with aryl isothiocyanate/isocyanate **1** (1.0 mmol), alkane **2** (1.0 mL), DTBP (4.0 equiv.), $Cu(OAc)_2$ (10 mol%) and CH_3CN (3 mL). The contents were stirred at 120 ^{o}C under a nitrogen atmosphere for 6-12 h. After the completion of reaction (as indicated by TLC), it was quenched with water (10 mL) and extracted with ethyl acetate (3 × 10 mL). The organic phase was dried over anhydrous magnesium sulfate and concentrated under reduced pressure to yield the crude product, which was purified by silica gel column chromatography using a mixture of EtOAc-Hexane (1:9) to give the pure product **3** in high yields (Table 2). The structure of the products was confirmed by comparison of ^{1}H, ^{13}C NMR and MS spectral data with those reported in the literature.[1]

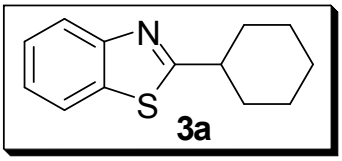
III. Copies of ^{1}H and ^{13}C NMR spectra.

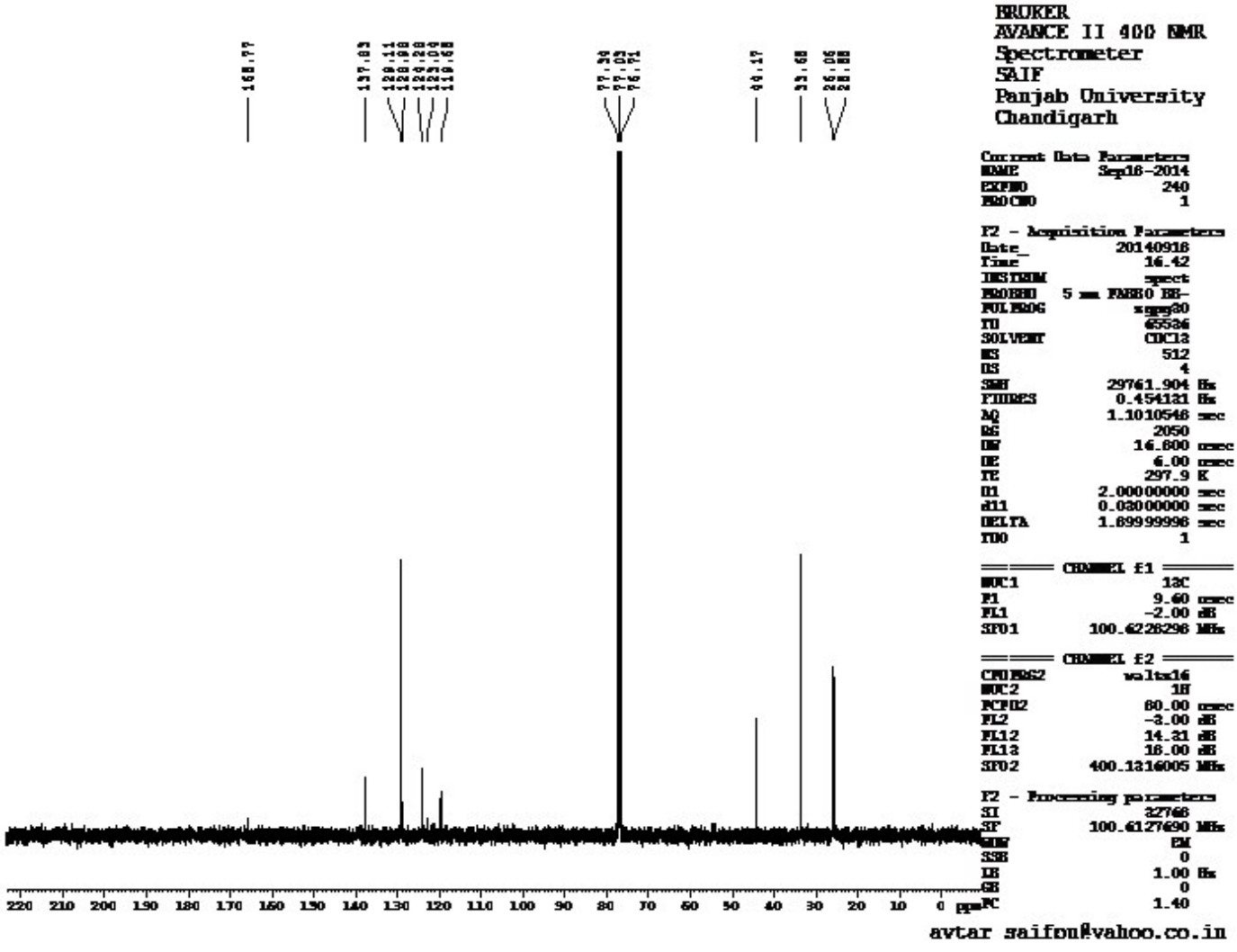
Compound 3a. ^{1}H NMR Spectrum ($CDCl_3$).



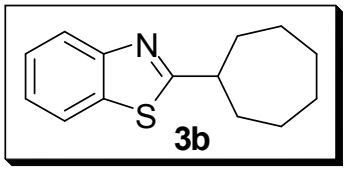


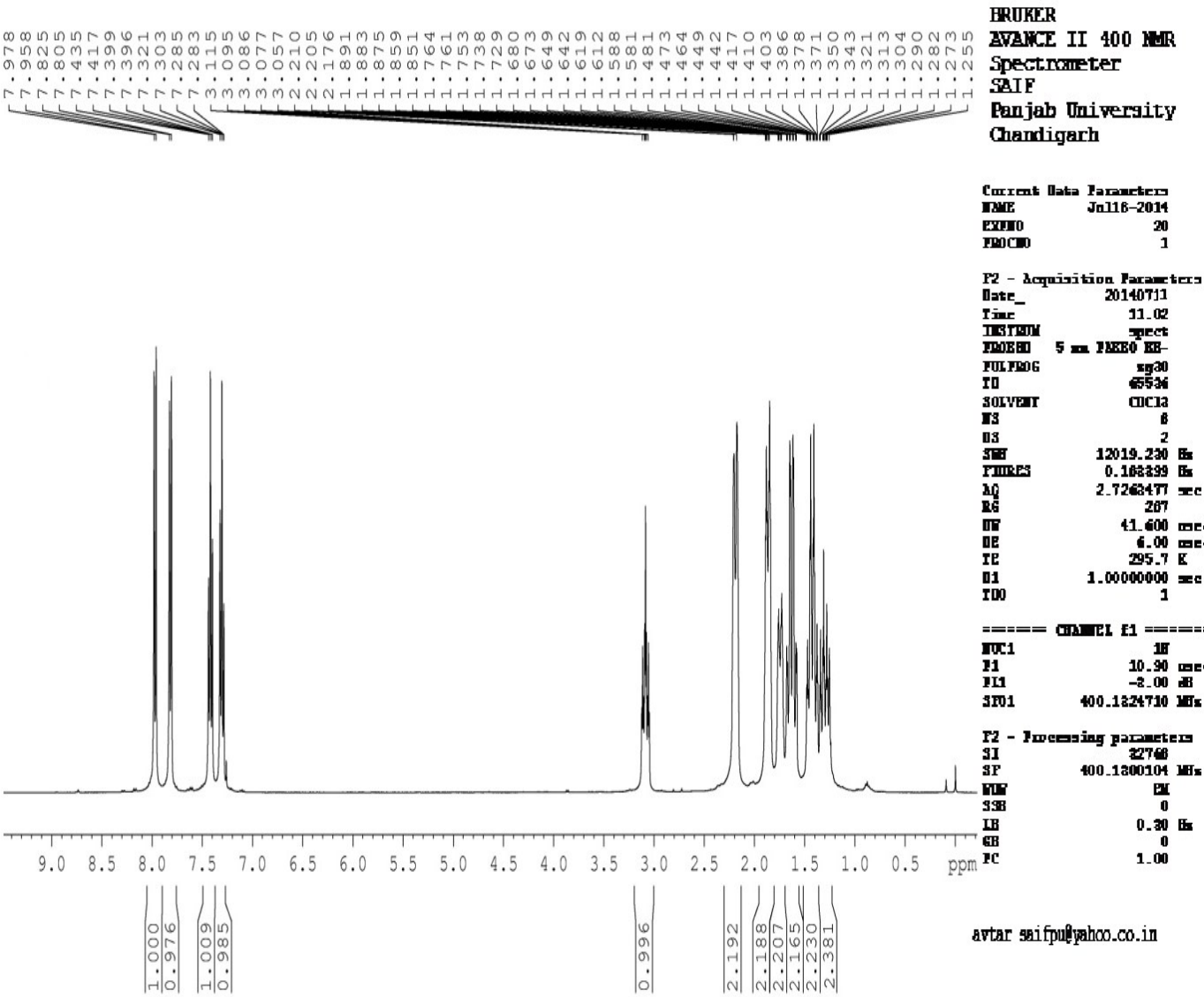
Compound 3a. ^{13}C NMR Spectrum ($CDCl_3$).



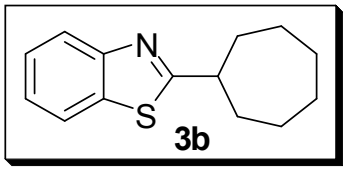


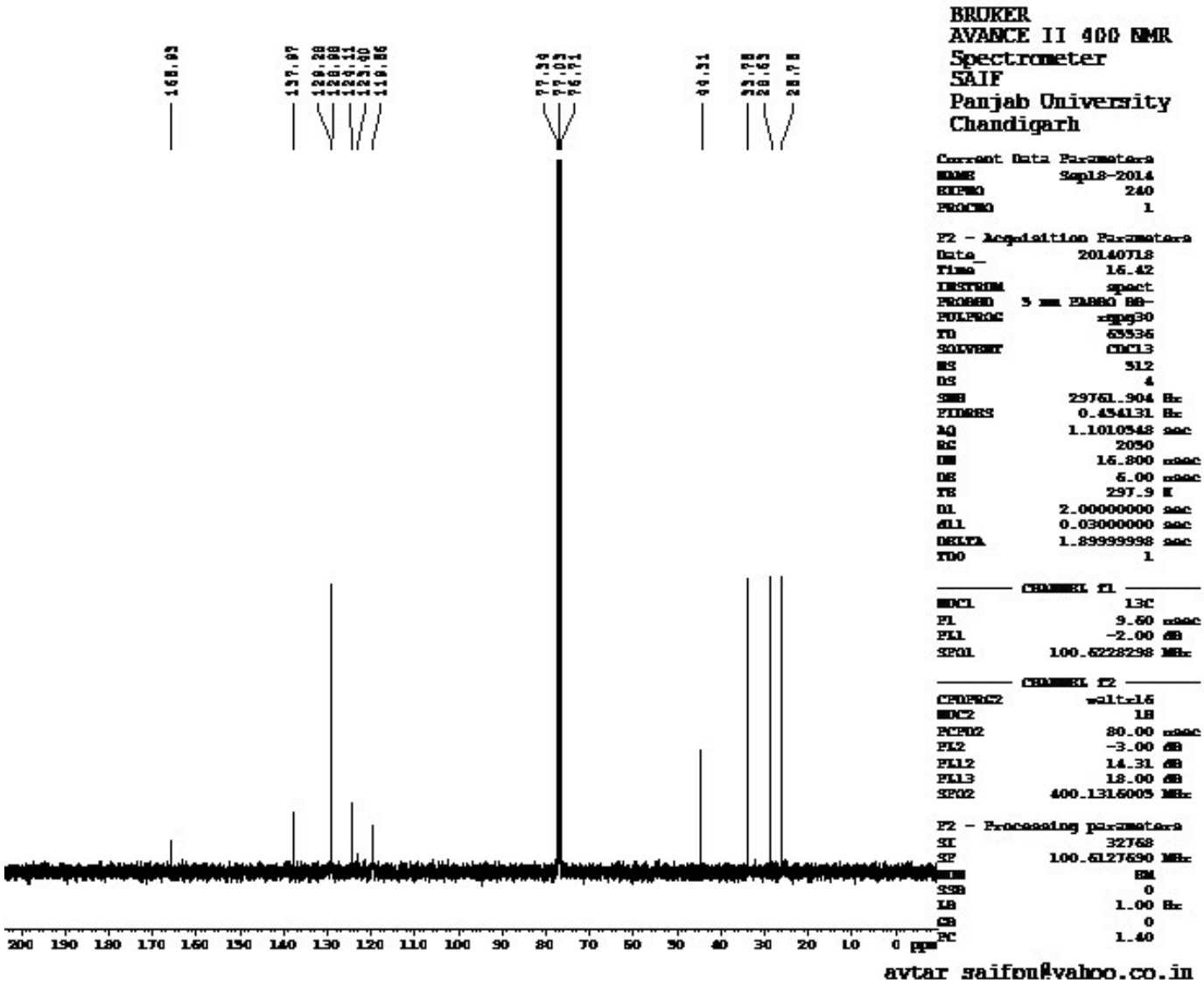
Compound 3b. ^{1}H NMR Spectrum ($CDCl_3$).



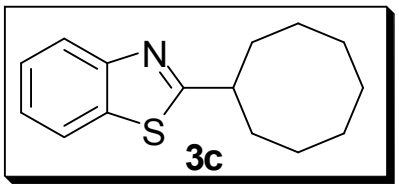


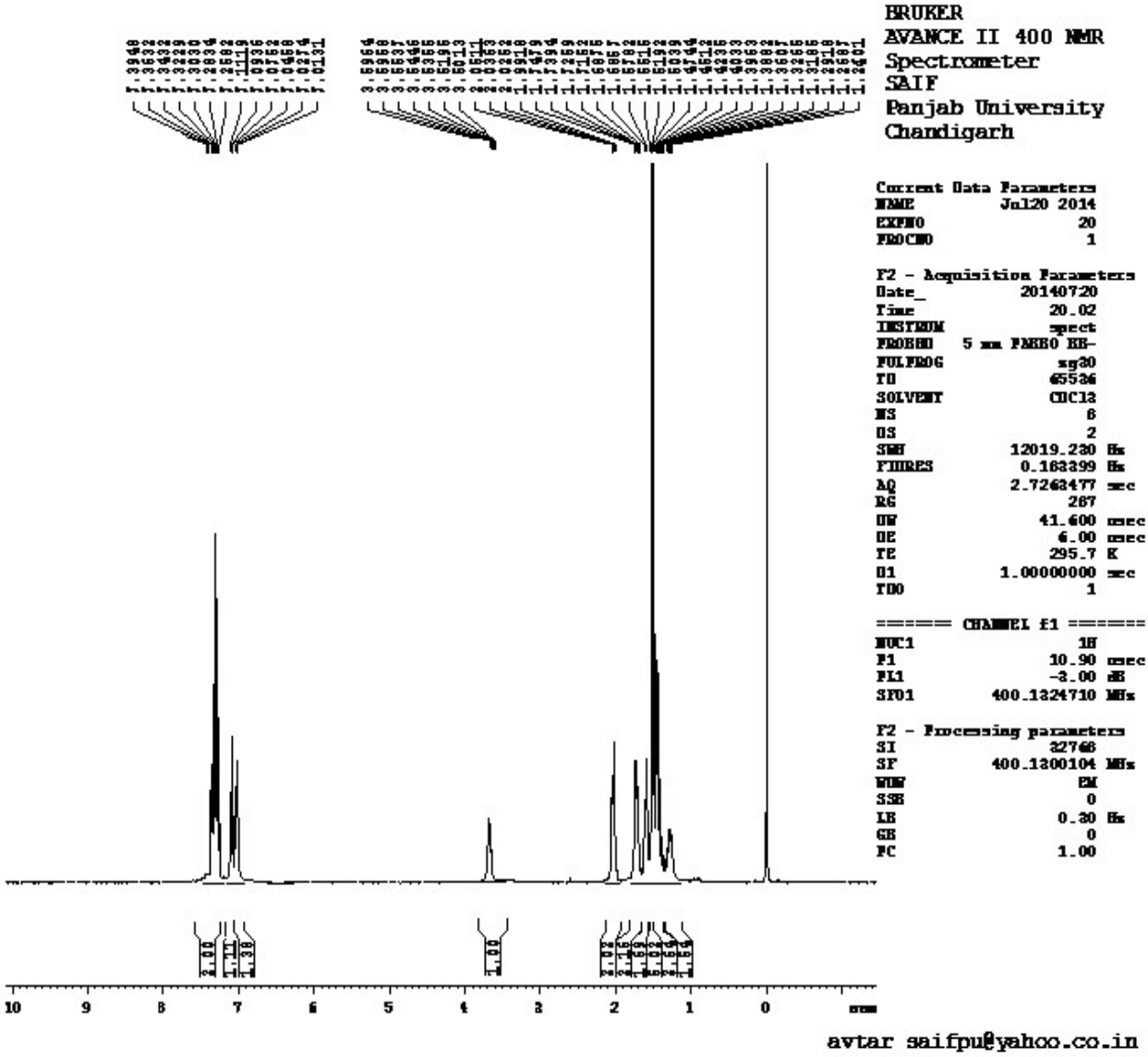
Compound 3b. ^{13}C NMR Spectrum ($CDCl_3$).



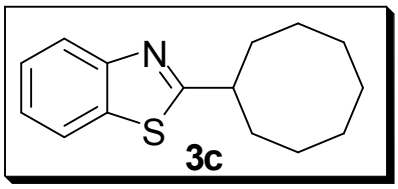


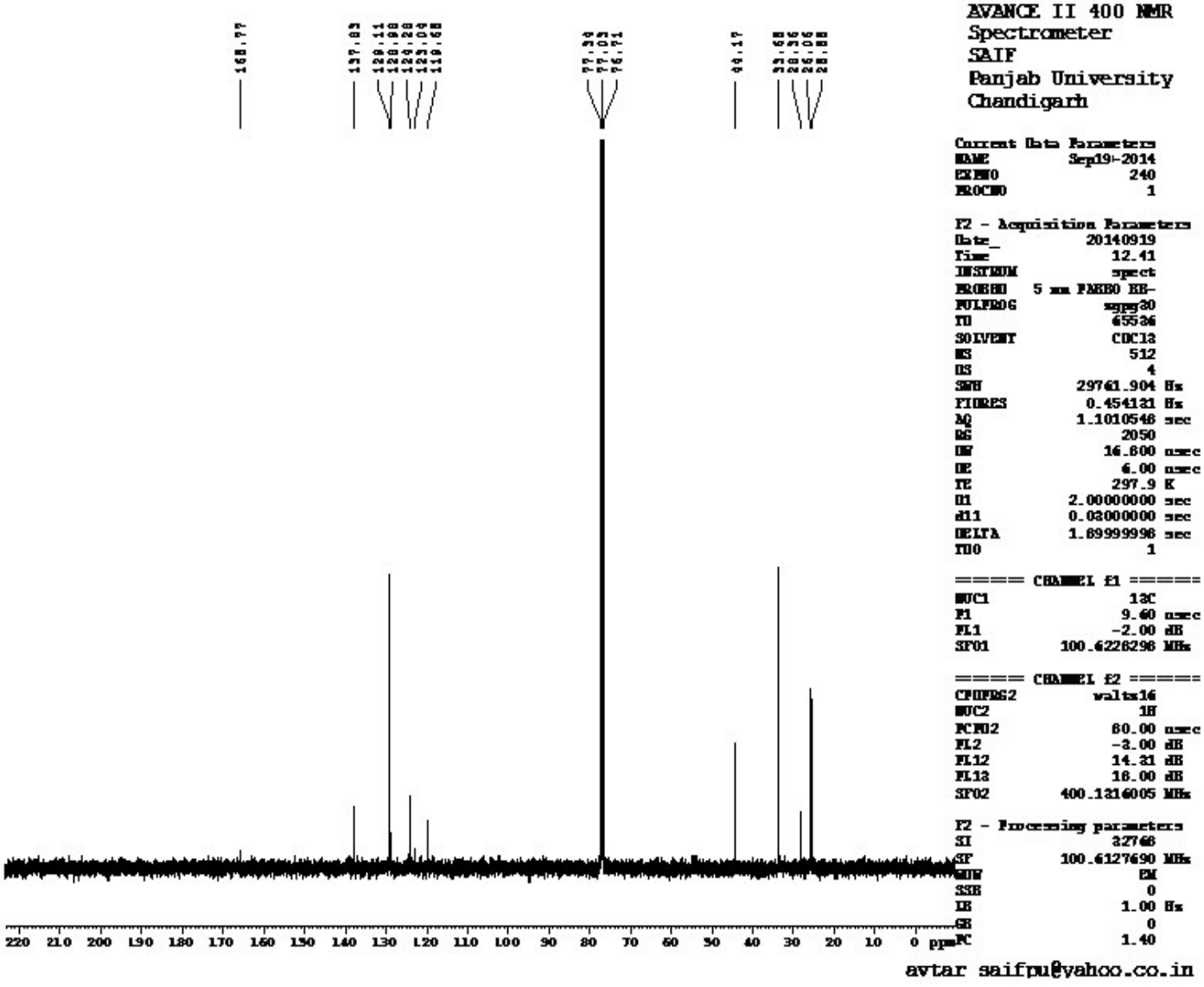
Compound 3c. ^{1}H NMR Spectrum ($CDCl_3$).



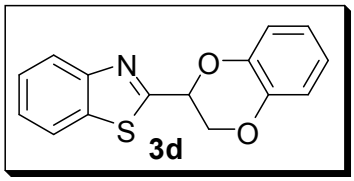


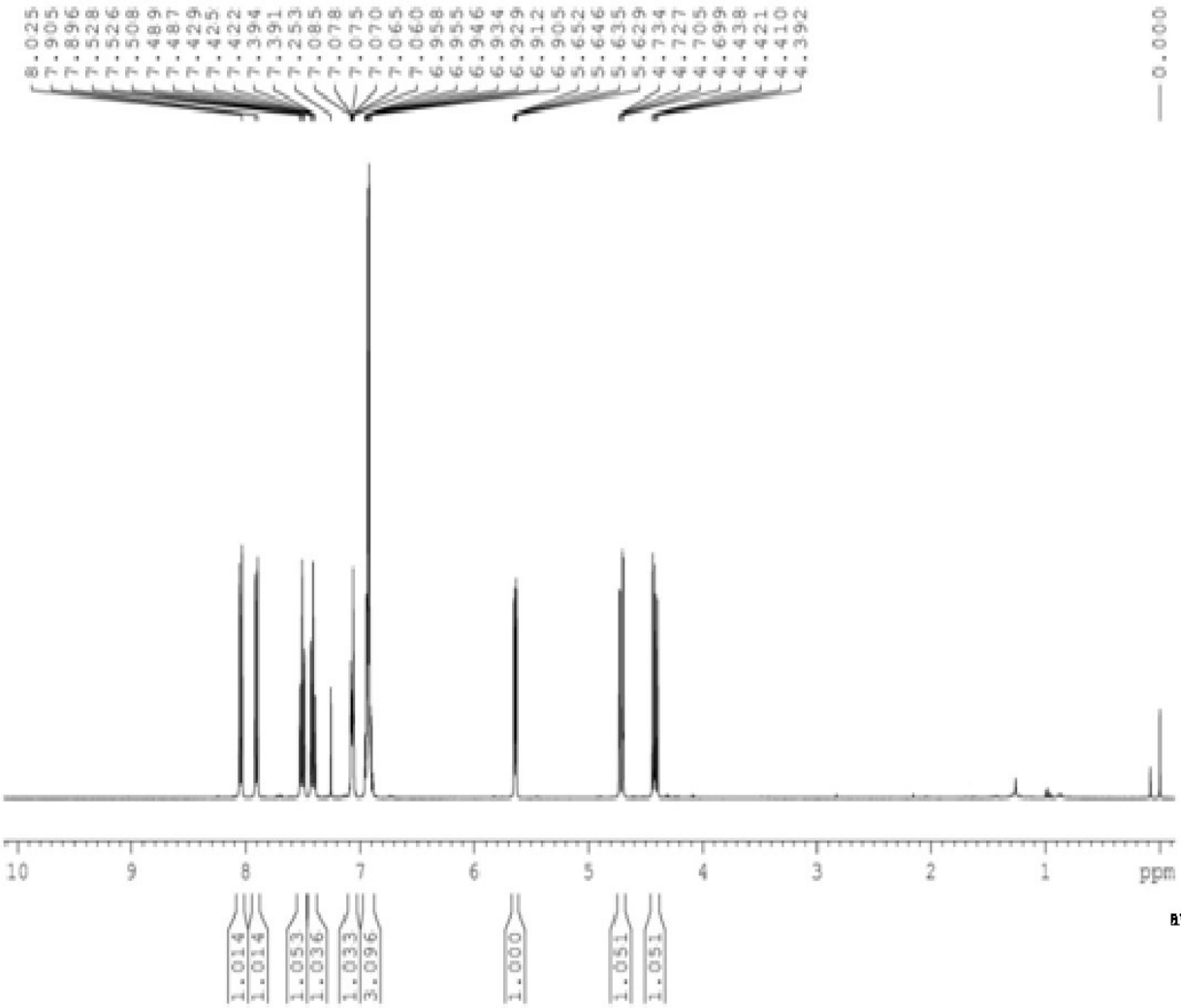
Compound 3c. ^{13}C NMR Spectrum ($CDCl_3$).



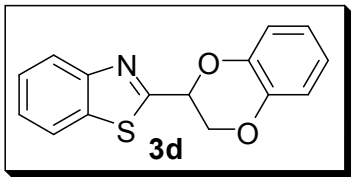


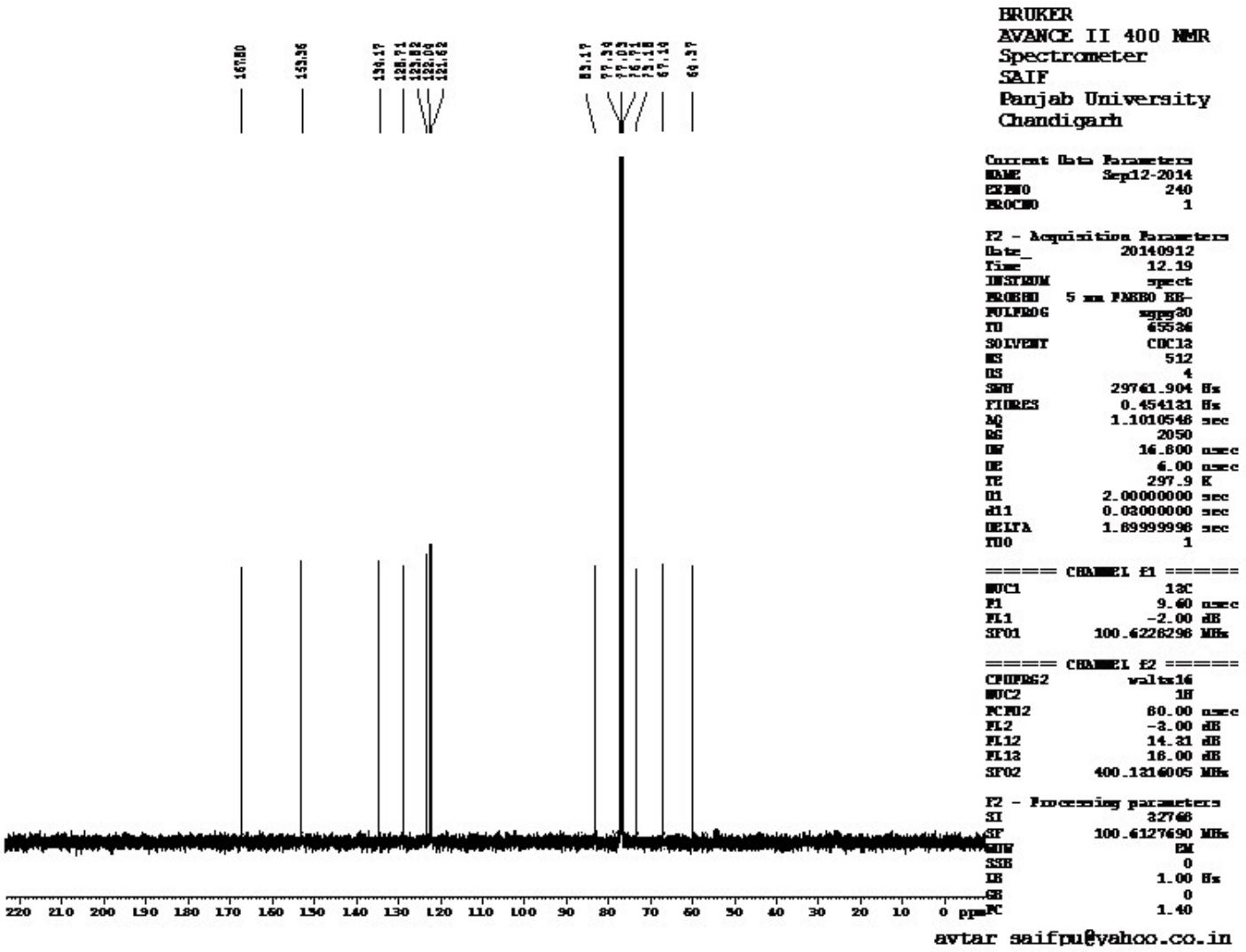
Compound 3d. ^{1}H NMR Spectrum ($CDCl_3$).



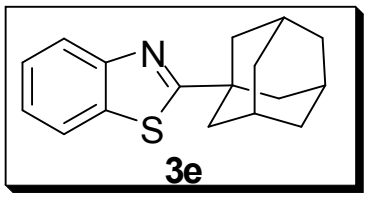


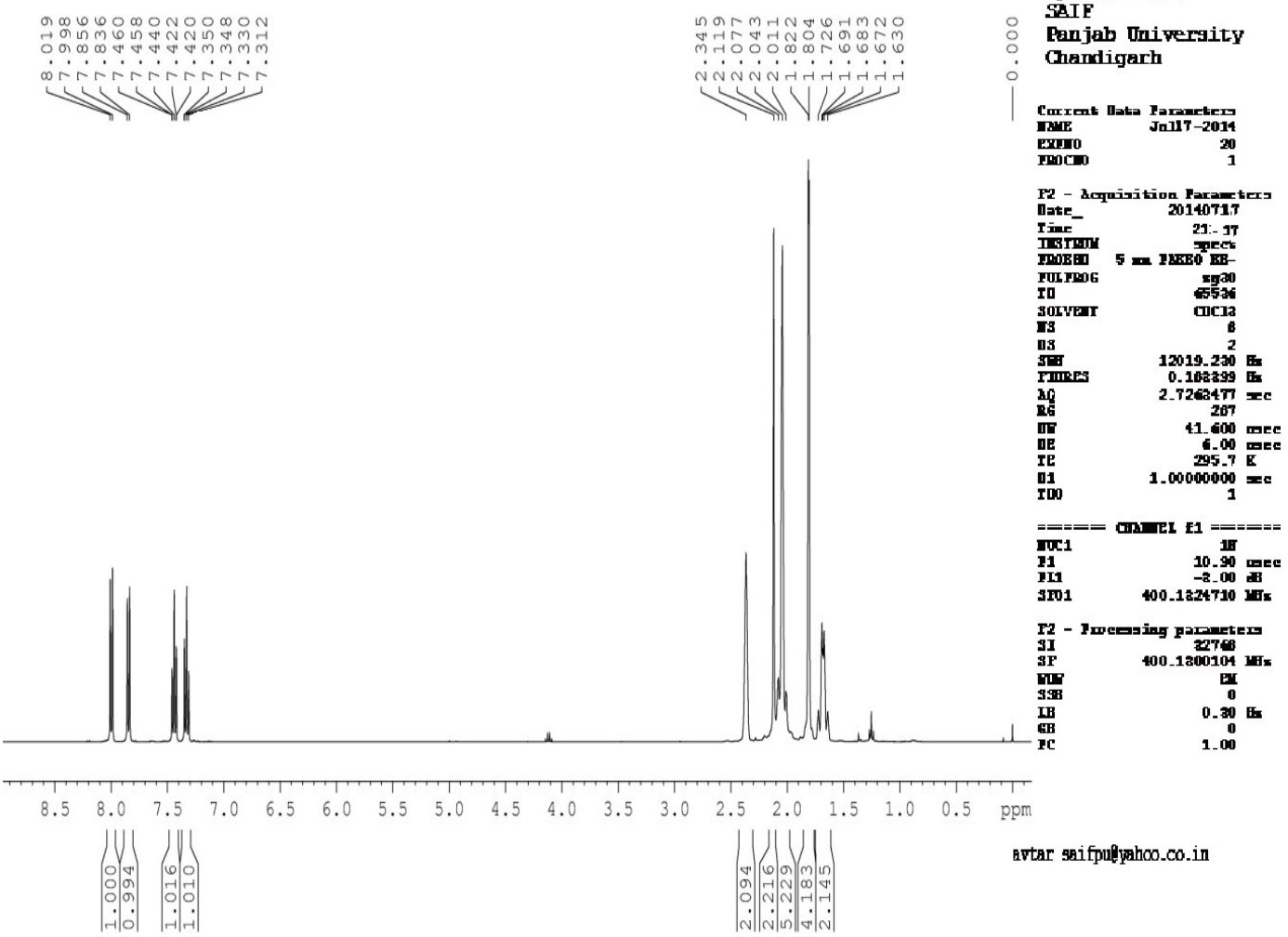
Compound 3d. ^{13}C NMR Spectrum ($CDCl_3$).



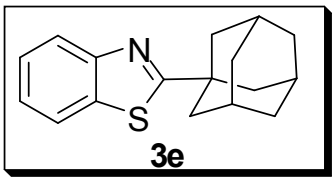


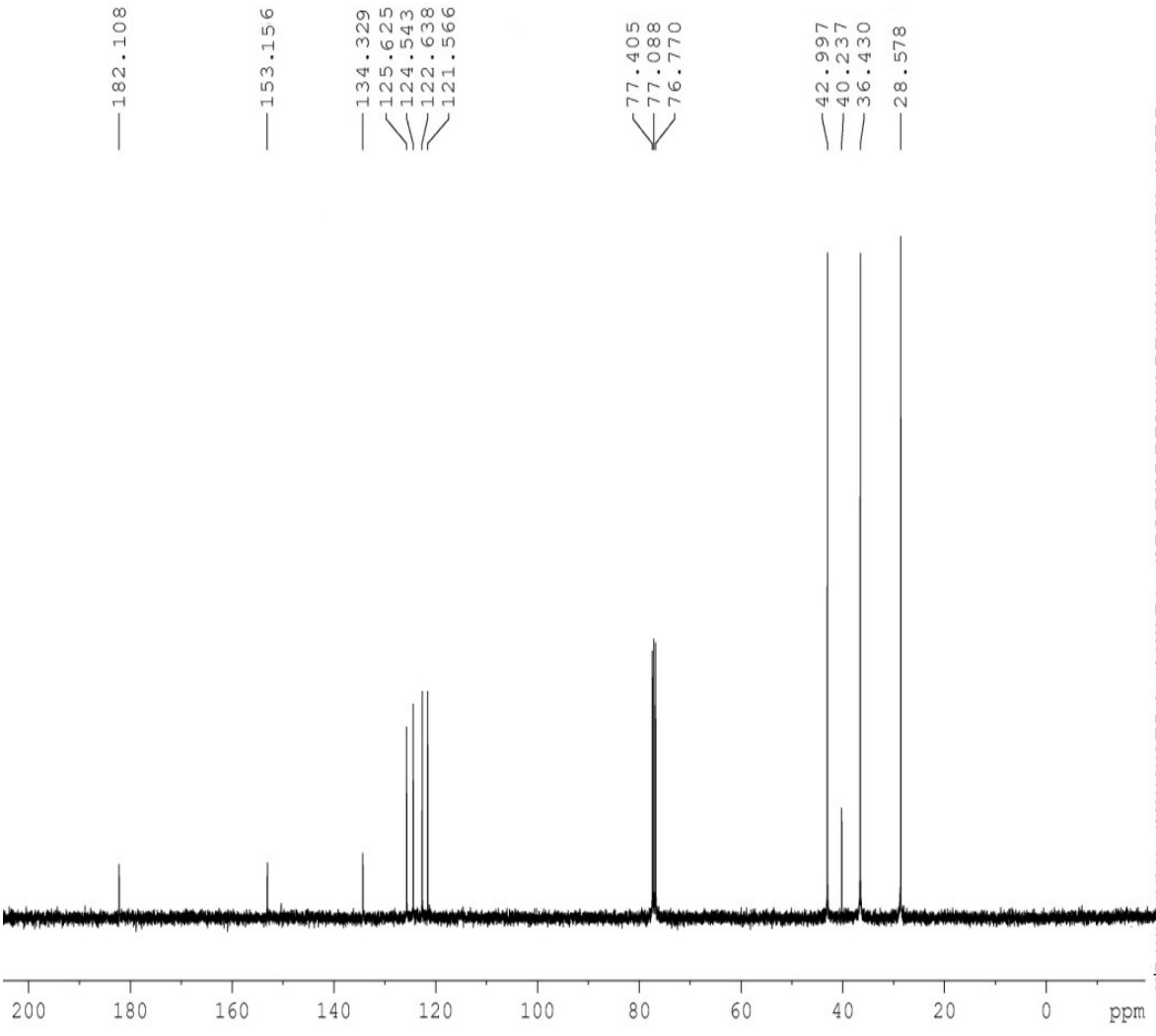
Compound 3e. ^{1}H NMR Spectrum ($CDCl_3$).



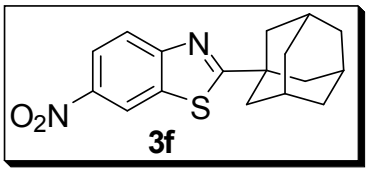


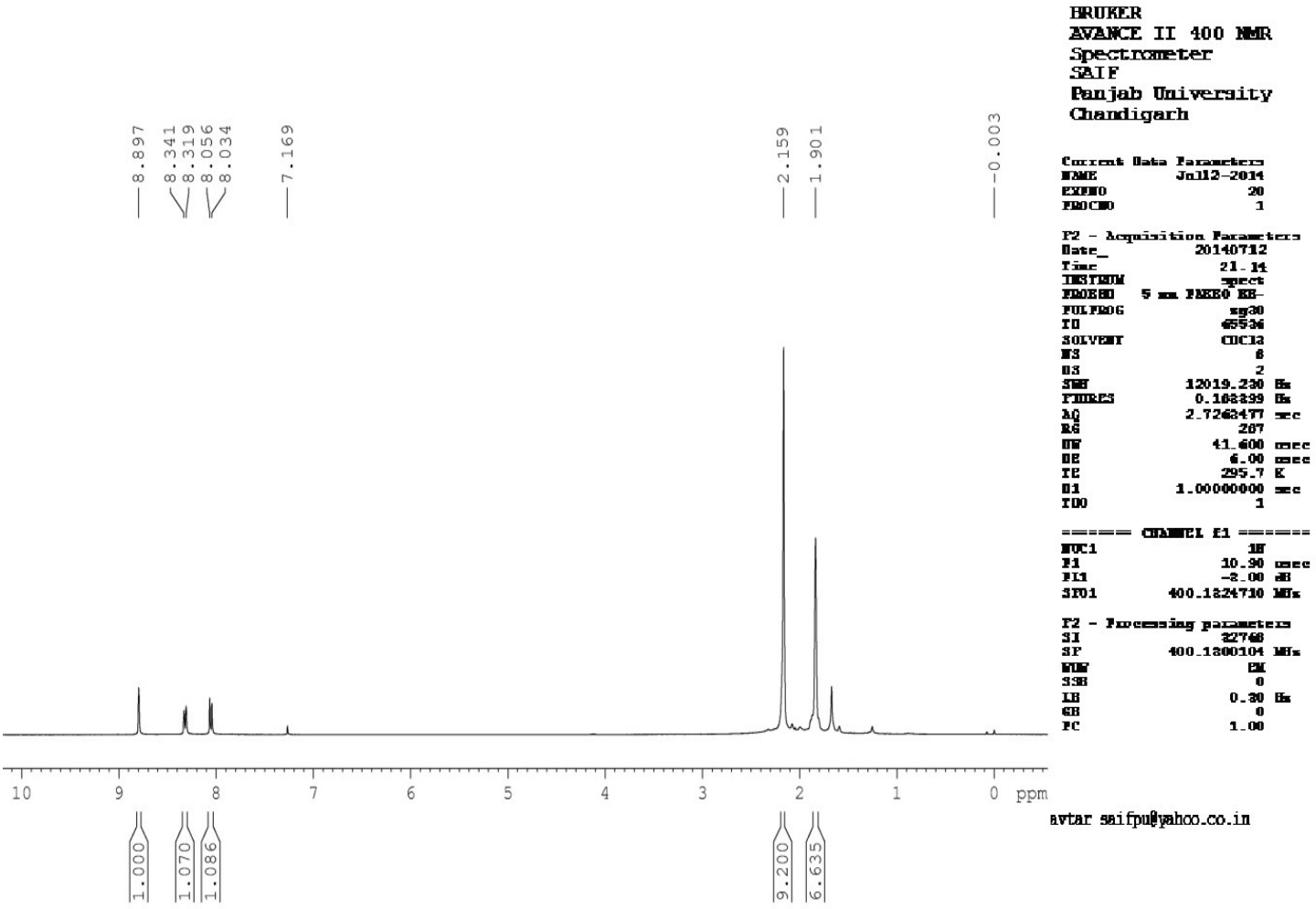
Compound 3e. ^{13}C NMR Spectrum ($CDCl_3$).



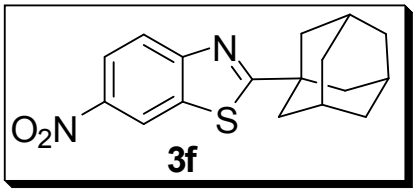


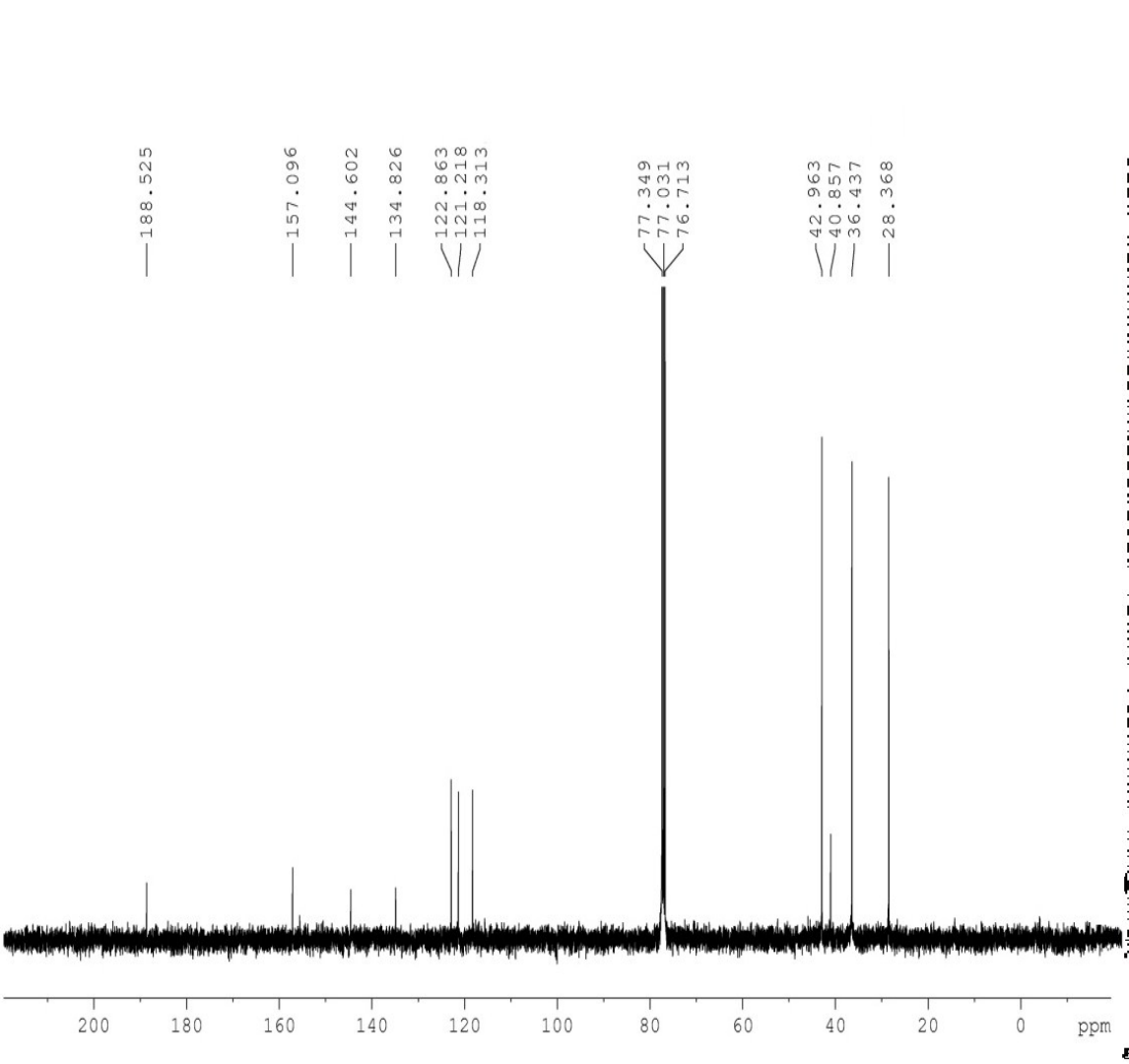
Compound 3f. ^{1}H NMR Spectrum ($CDCl_3$).



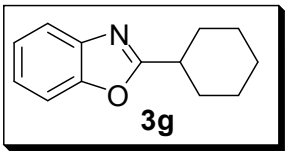


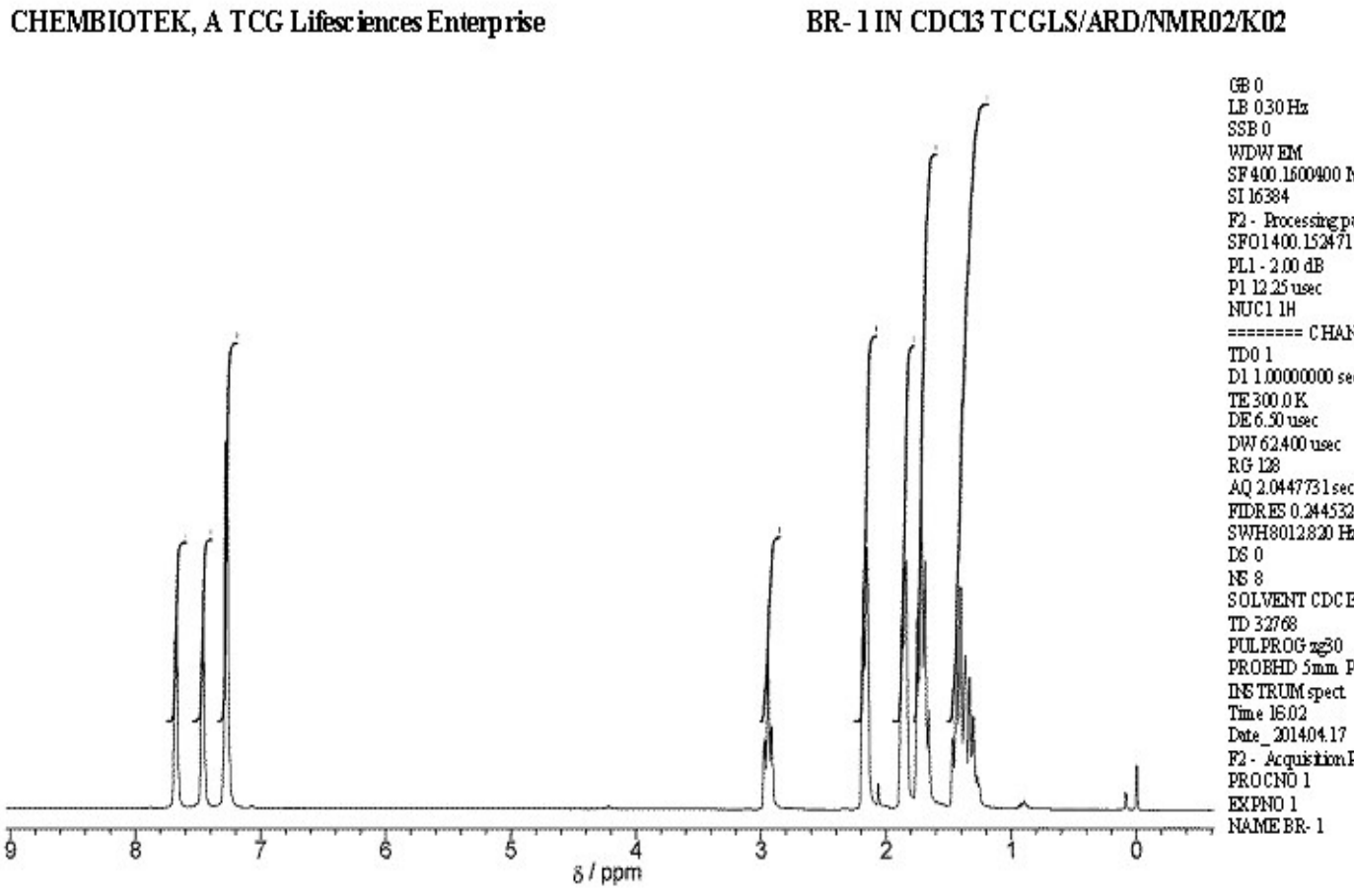
Compound 3f. ^{13}C NMR Spectrum ($CDCl_3$).



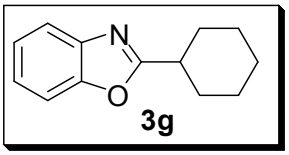


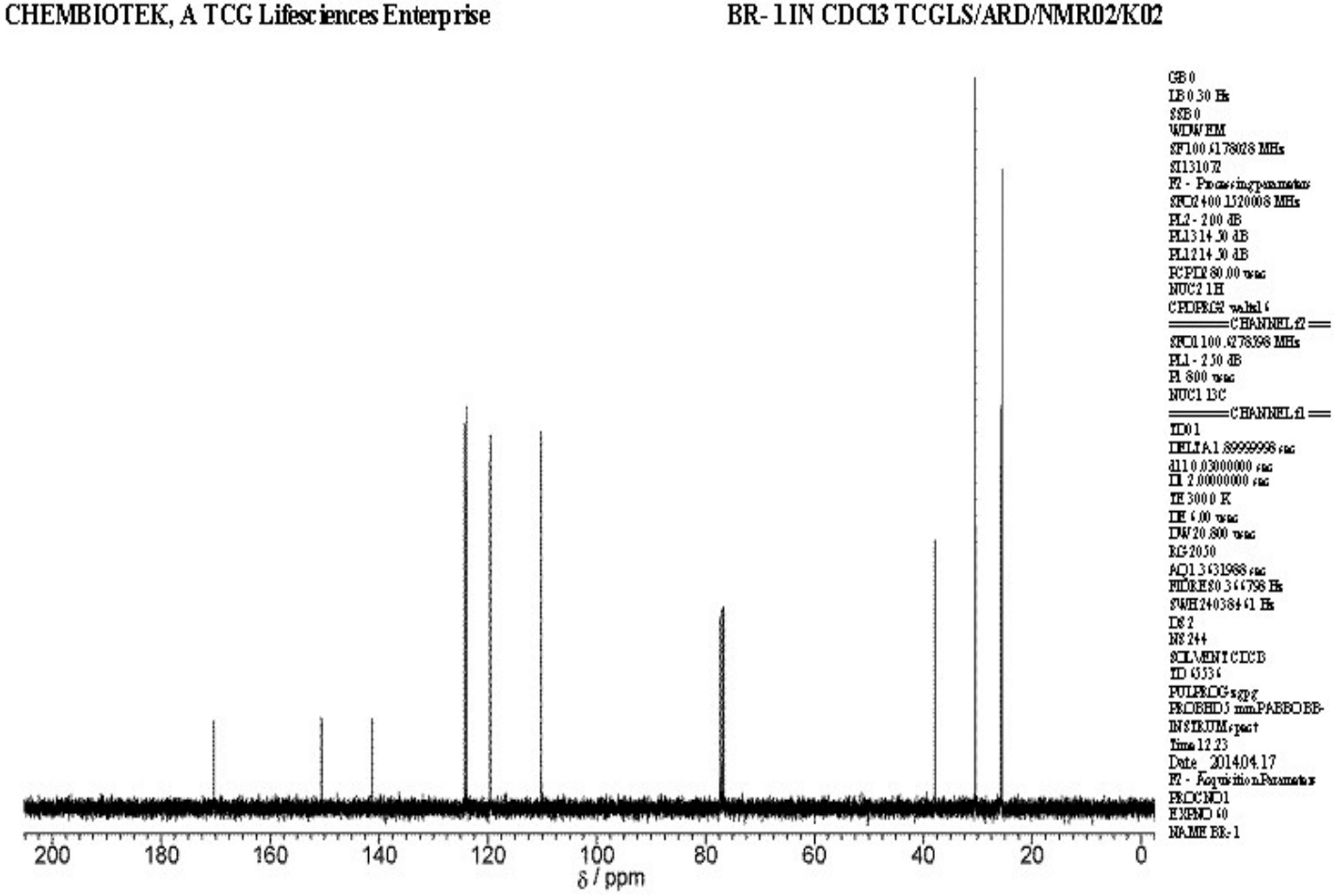
Compound 3g. ^{1}H NMR Spectrum ($CDCl_3$).



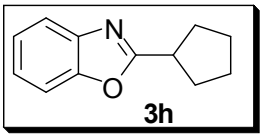


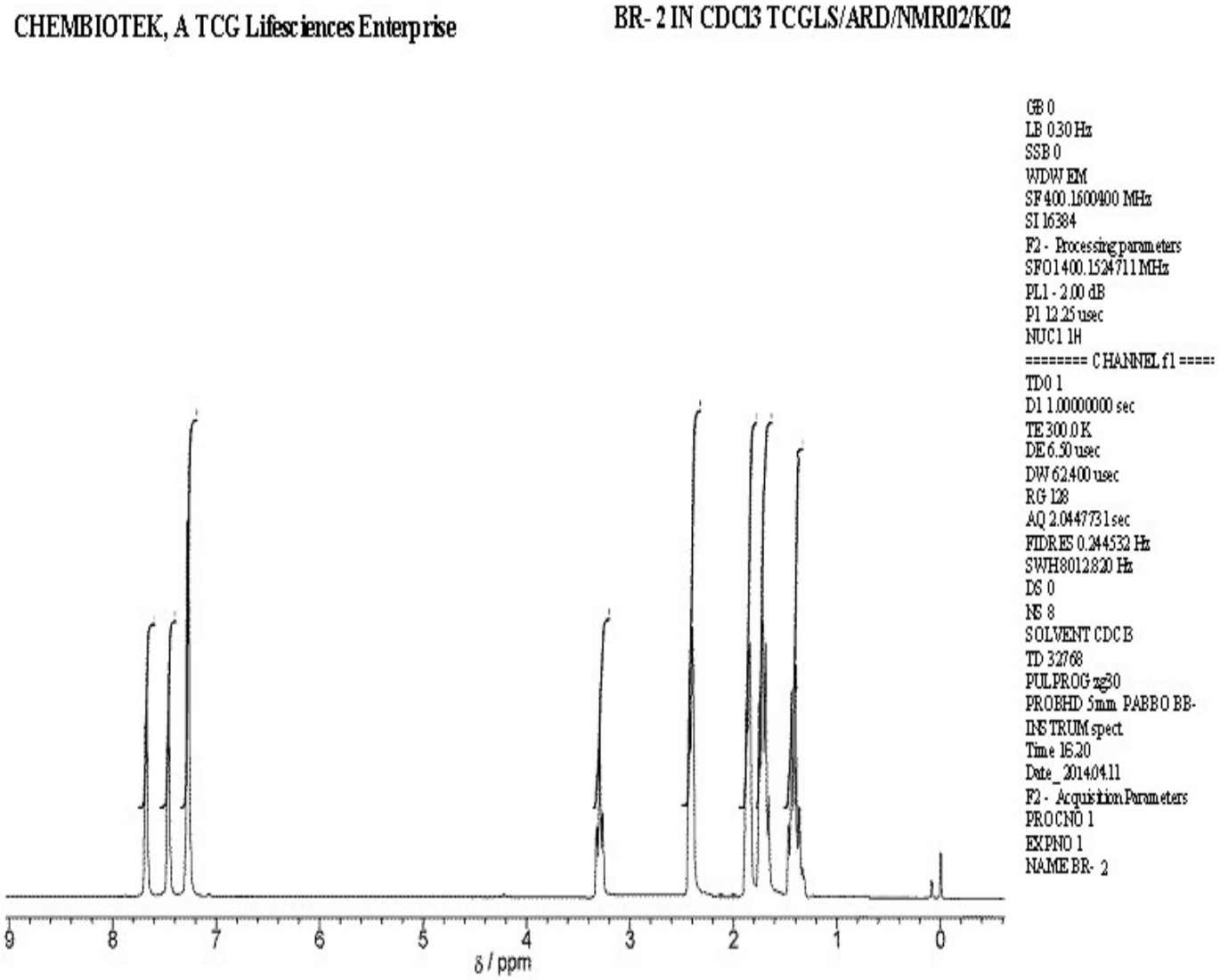
Compound 3g. ^{13}C NMR Spectrum ($CDCl_3$).



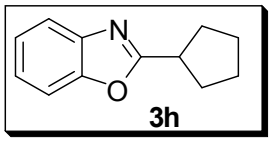


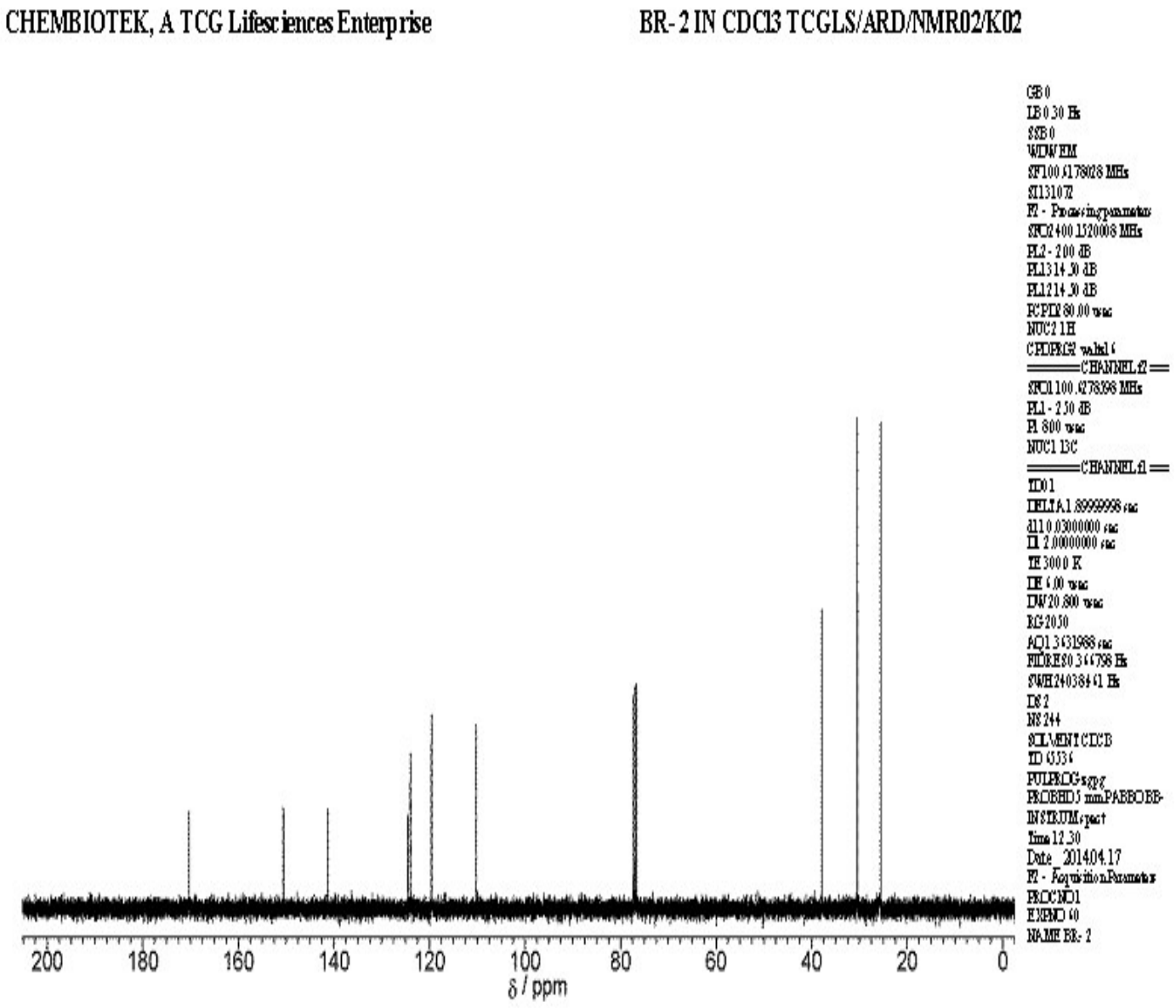
Compound 3h. ^{1}H NMR Spectrum ($CDCl_3$).



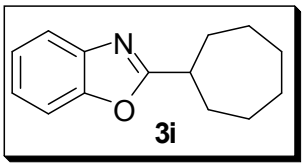


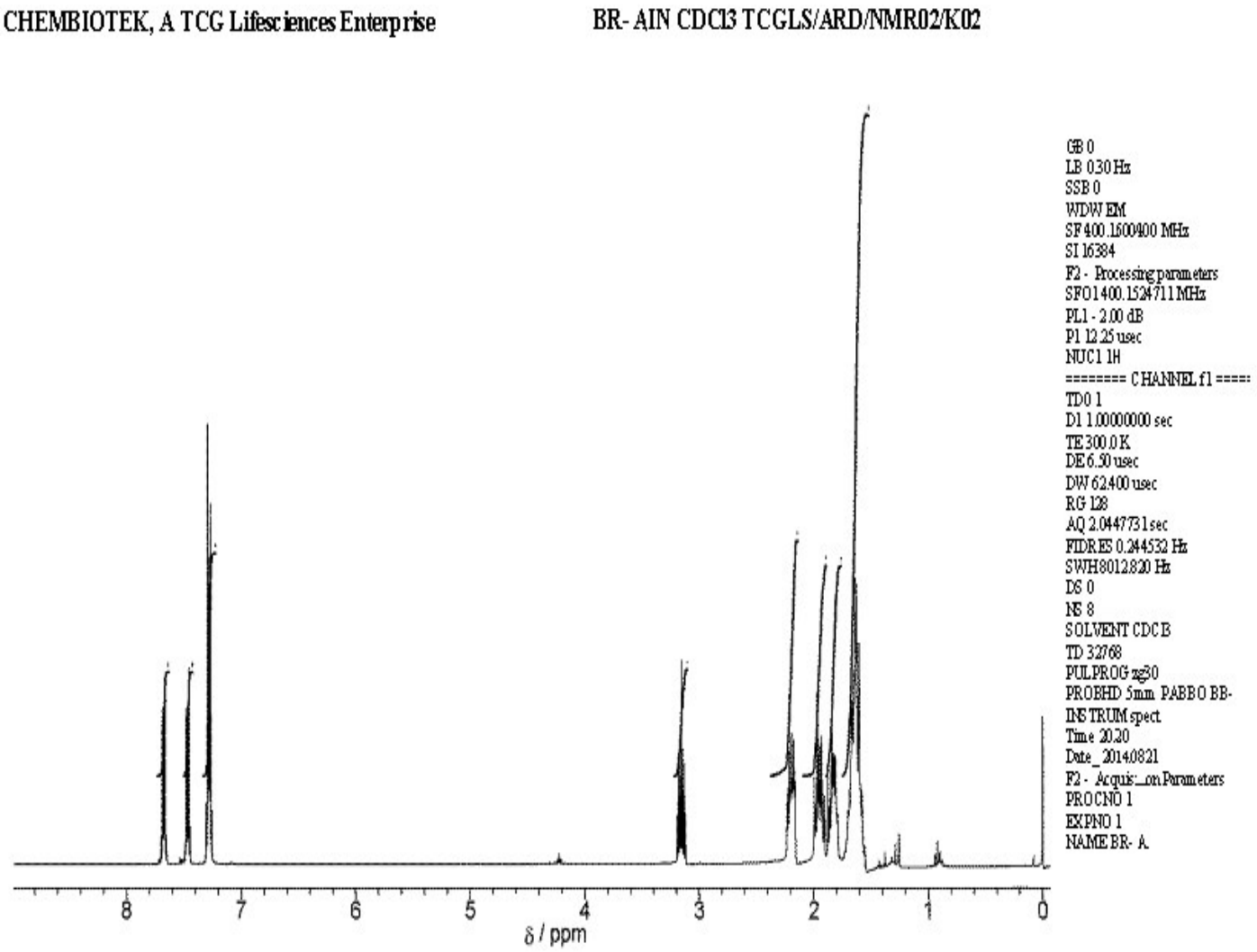
Compound 3h. ^{13}C NMR Spectrum ($CDCl_3$).



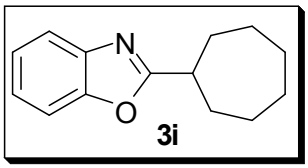


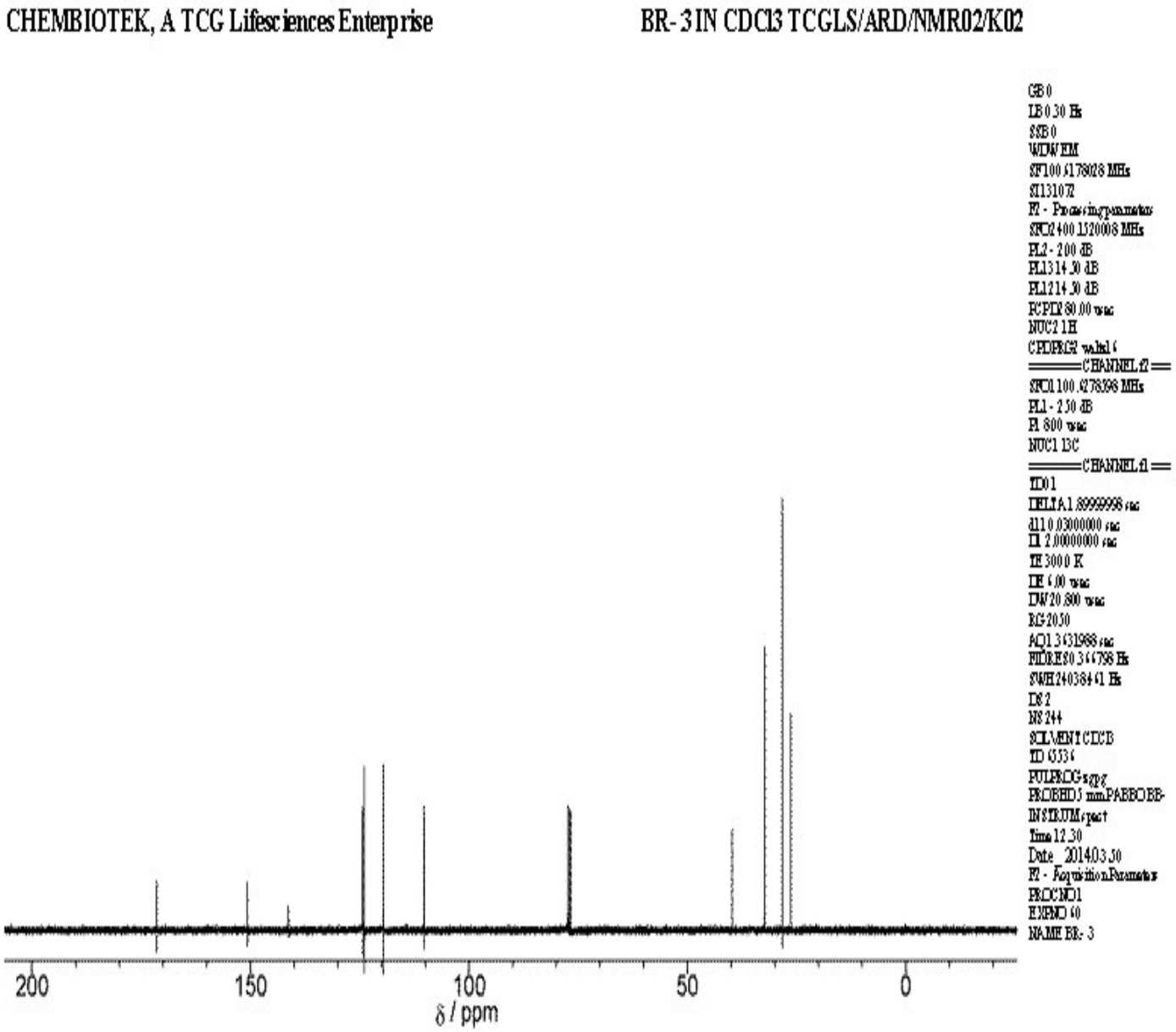
Compound 3i. ^{1}H NMR Spectrum ($CDCl_3$).



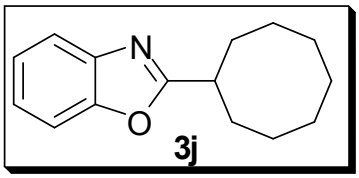


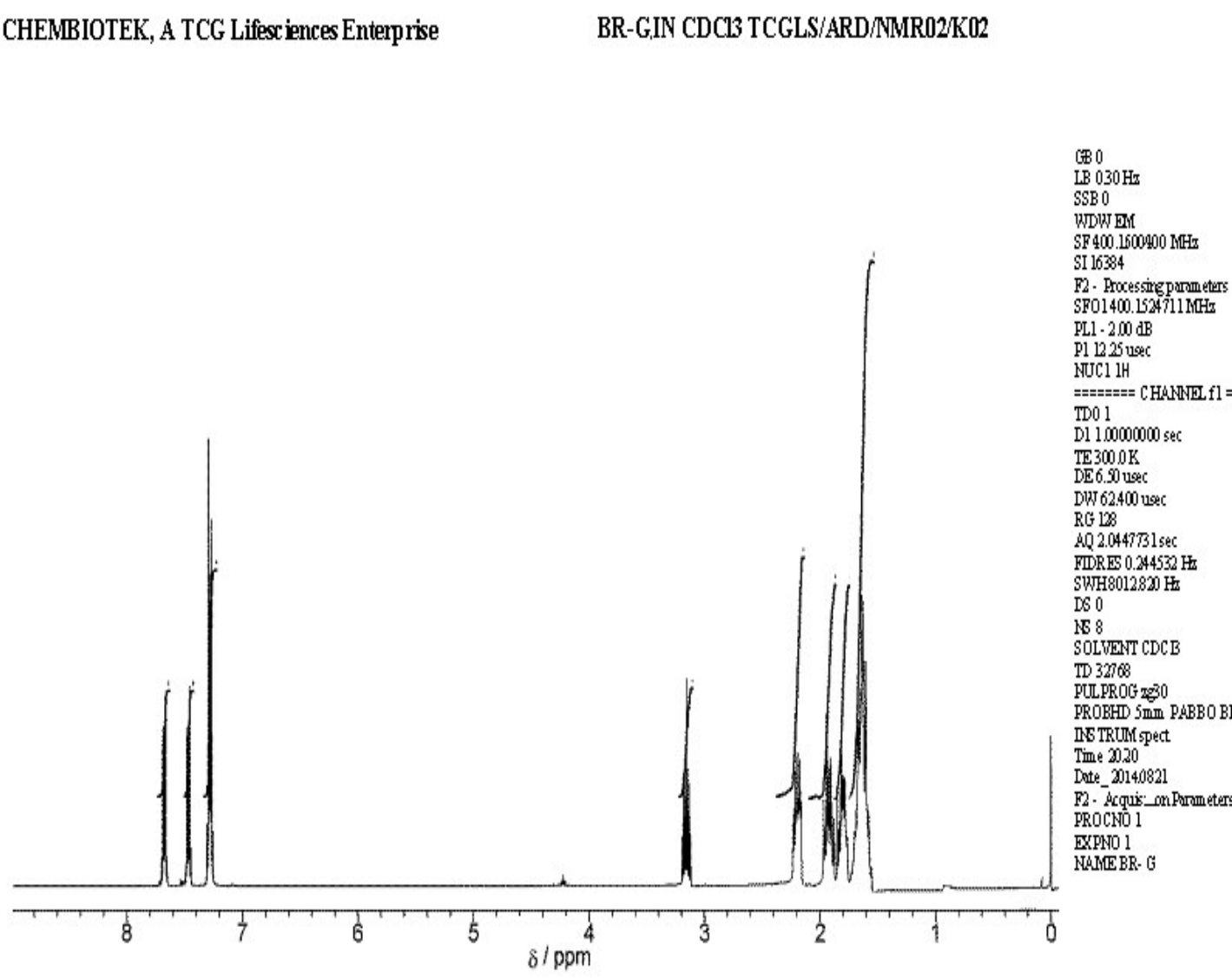
Compound 3i. ^{13}C NMR Spectrum ($CDCl_3$).



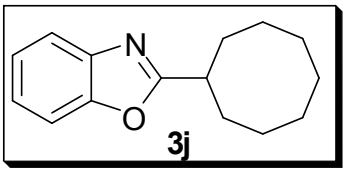


Compound 3j. ^{1}H NMR Spectrum ($CDCl_3$).

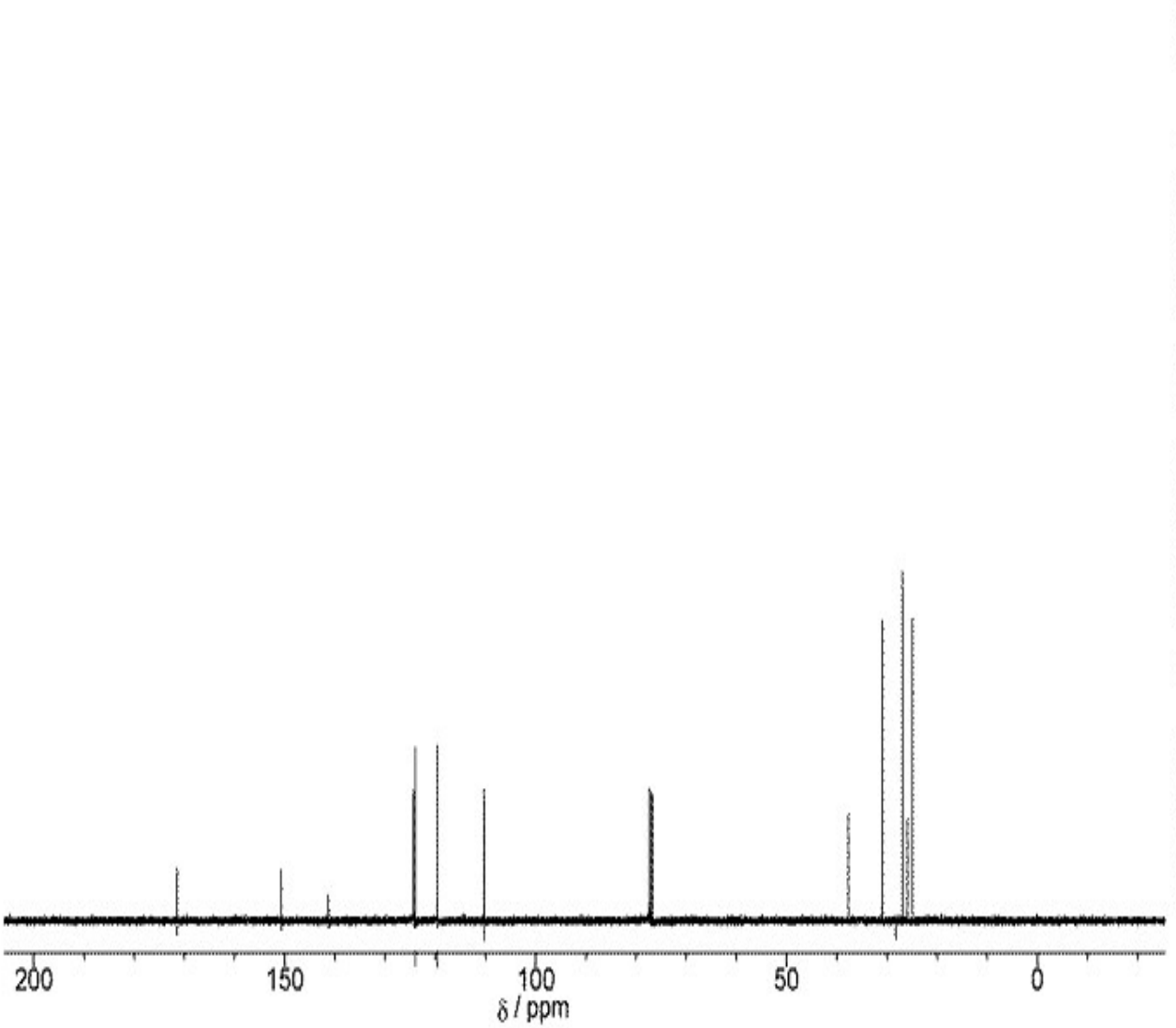




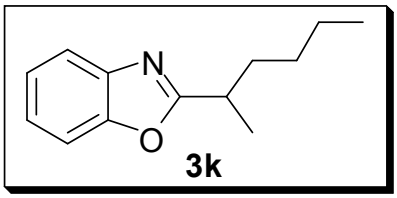
Compound 3j. ^{13}C NMR Spectrum ($CDCl_3$).

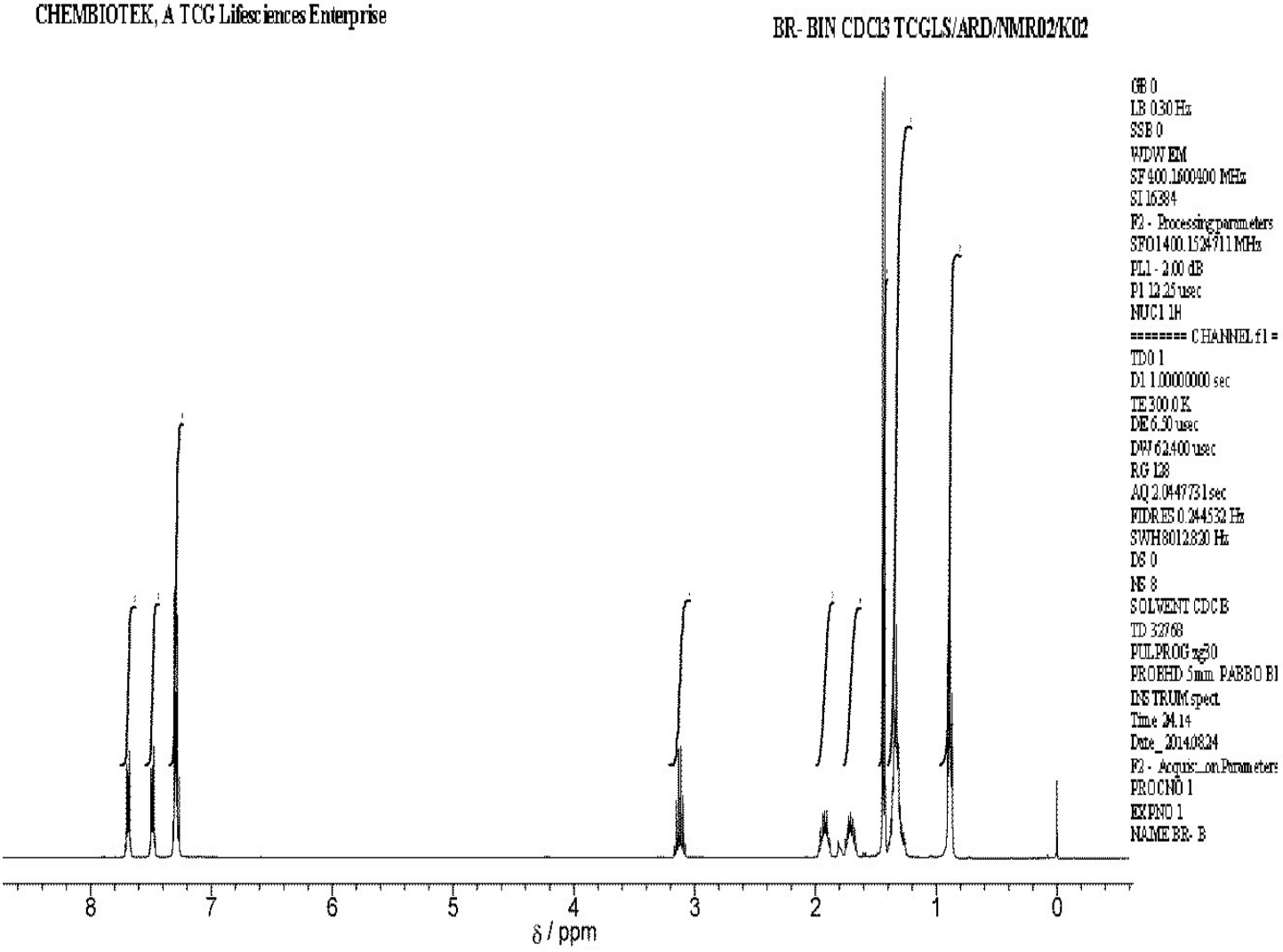


CHEMBIOTEK, A TCG Lifesciences Enterprise BR-GIN CDCl3 TCGLS/ARD/NMR02/K02

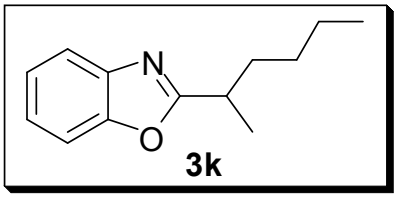


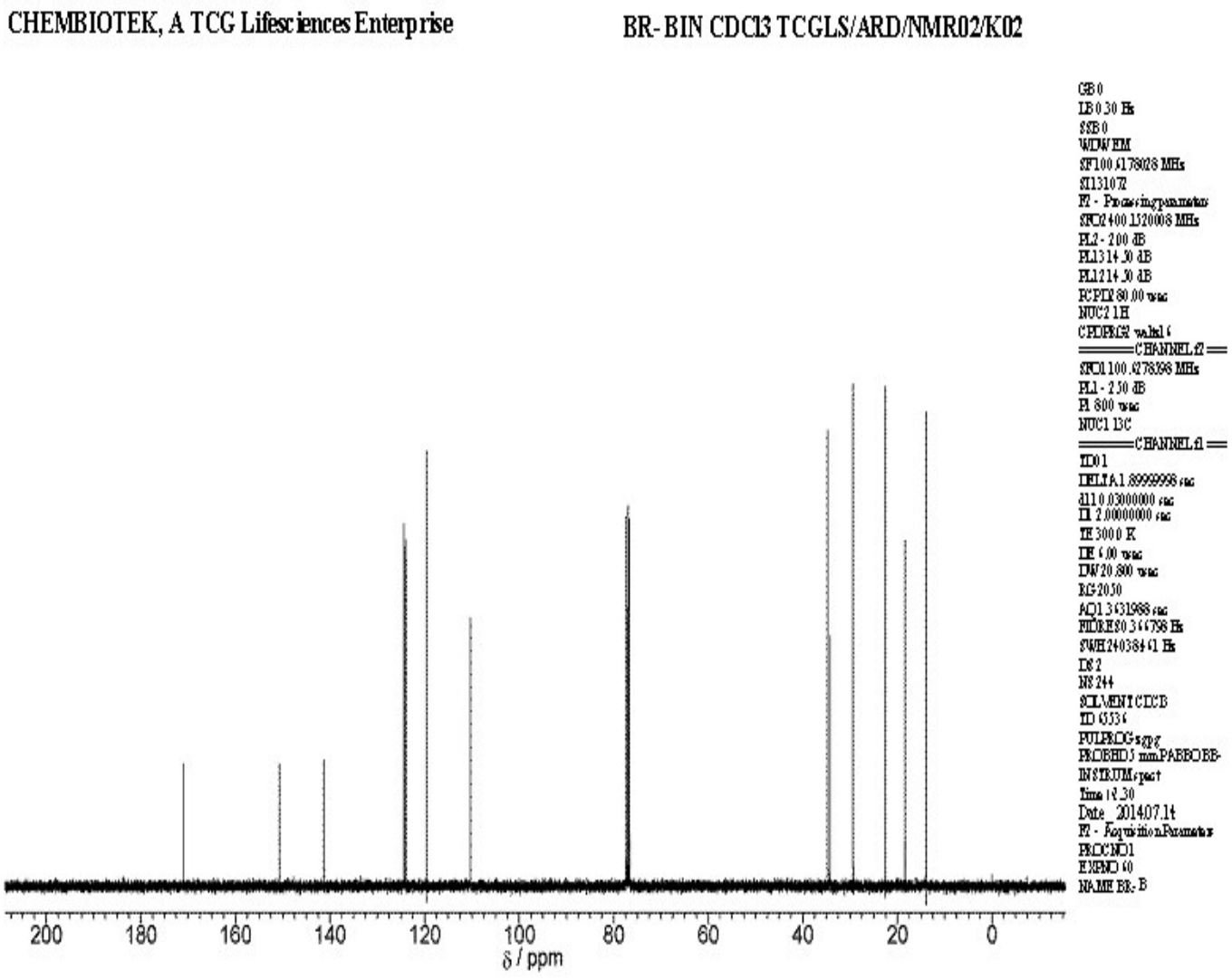
Compound 3k. ^{1}H NMR Spectrum ($CDCl_3$).



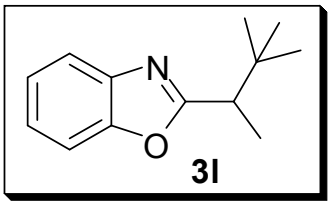


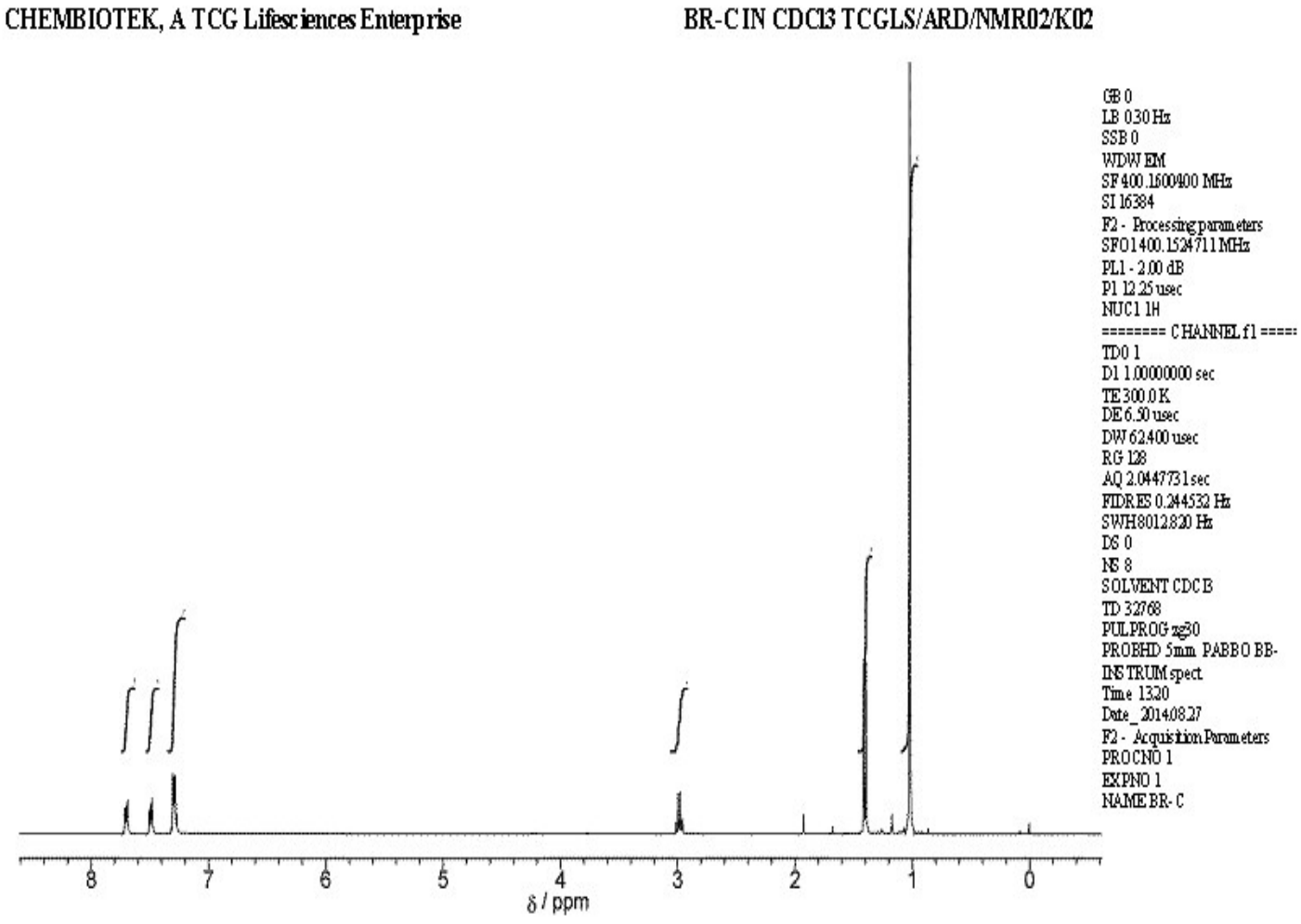
Compound 3k. ^{13}C NMR Spectrum ($CDCl_3$).



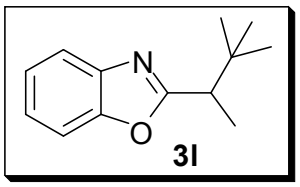


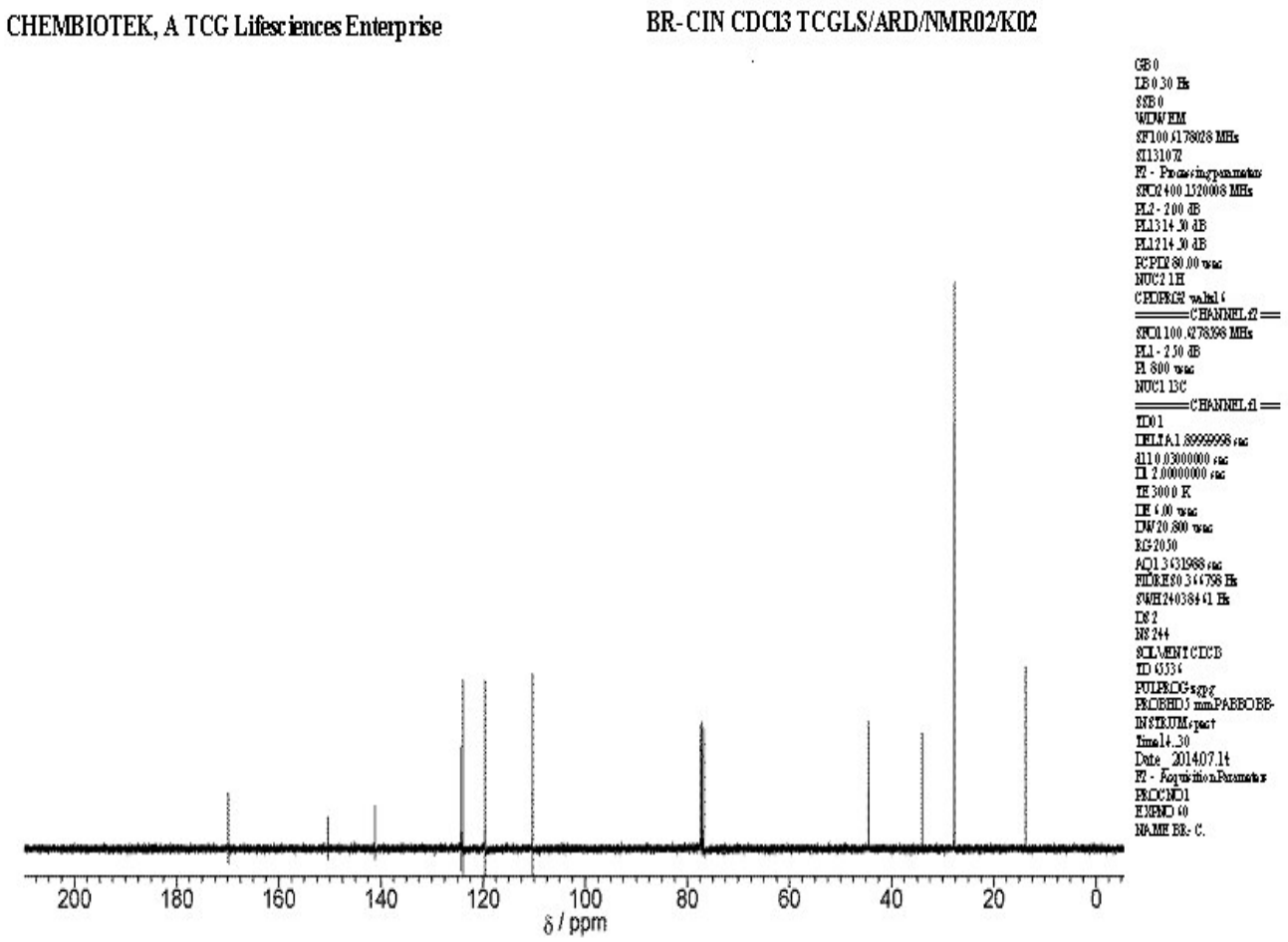
Compound 3l. ^{1}H NMR Spectrum ($CDCl_3$).



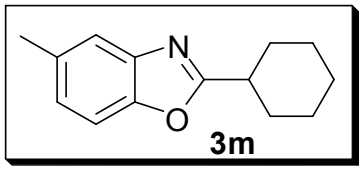


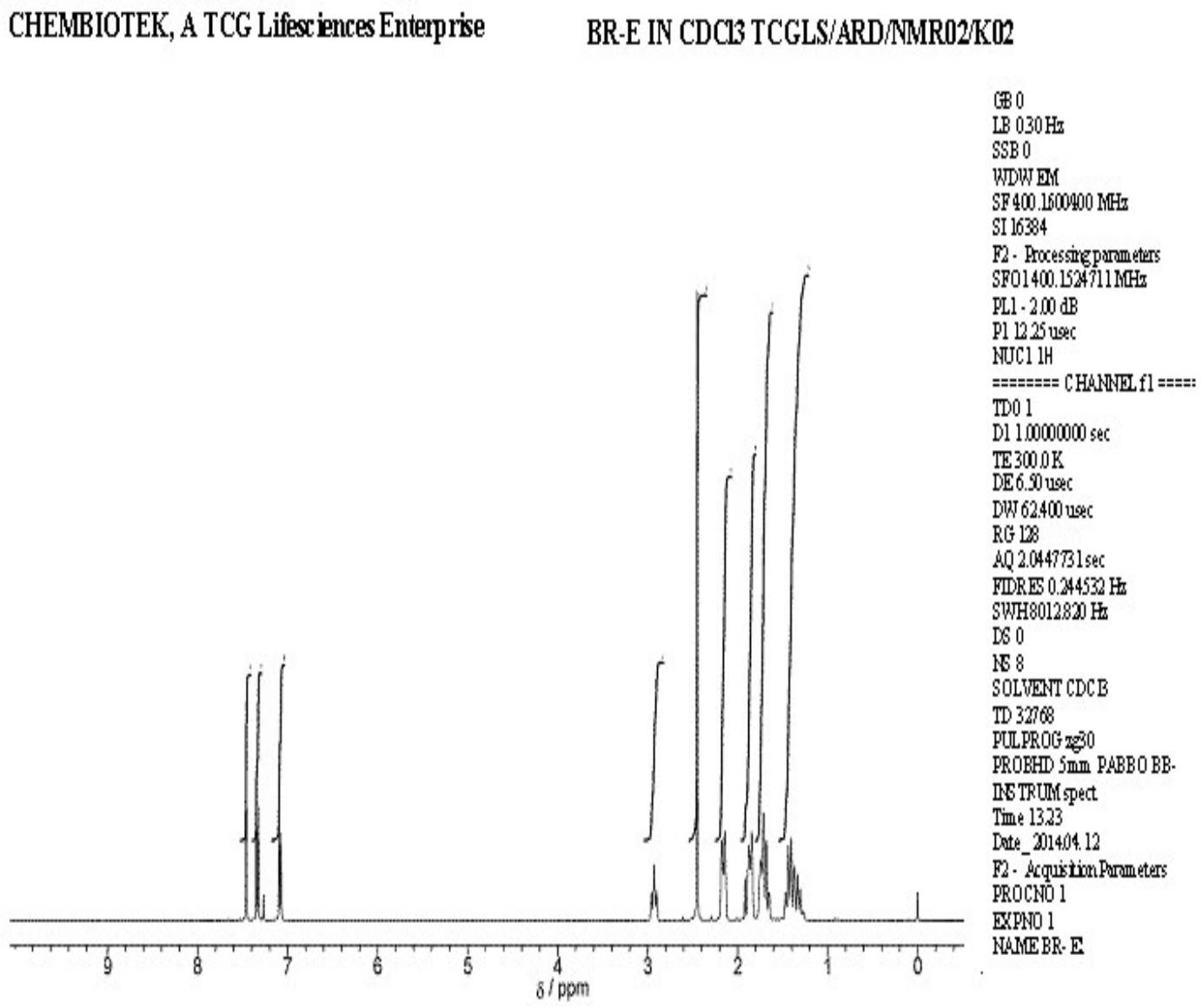
Compound 3l. ^{13}C NMR Spectrum ($CDCl_3$).



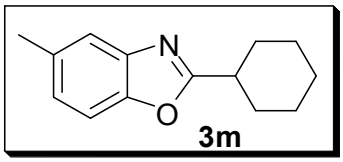


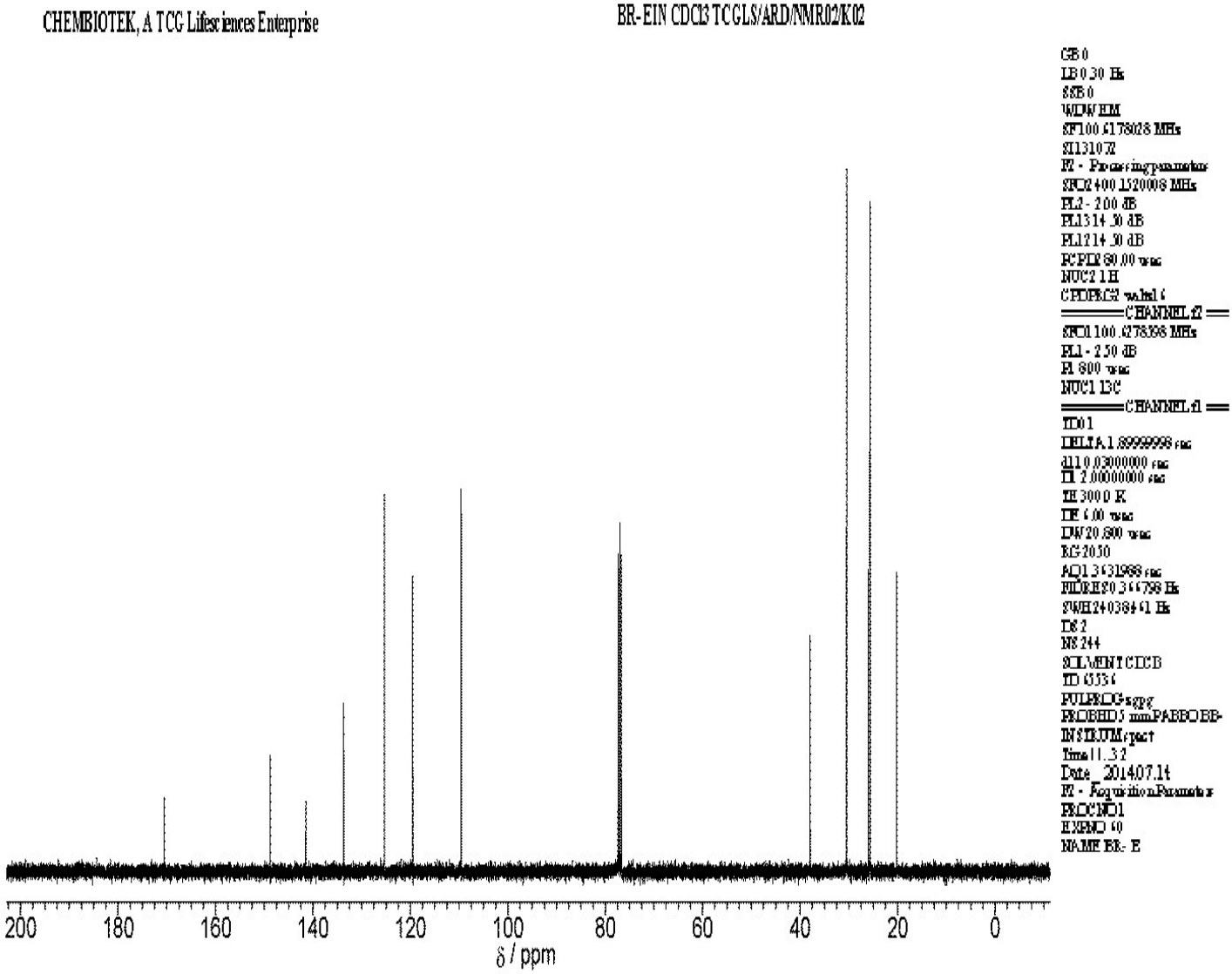
Compound 3m. ^{1}H NMR Spectrum ($CDCl_3$).



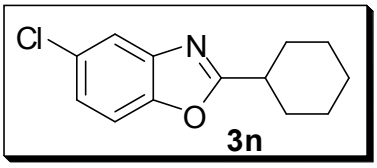


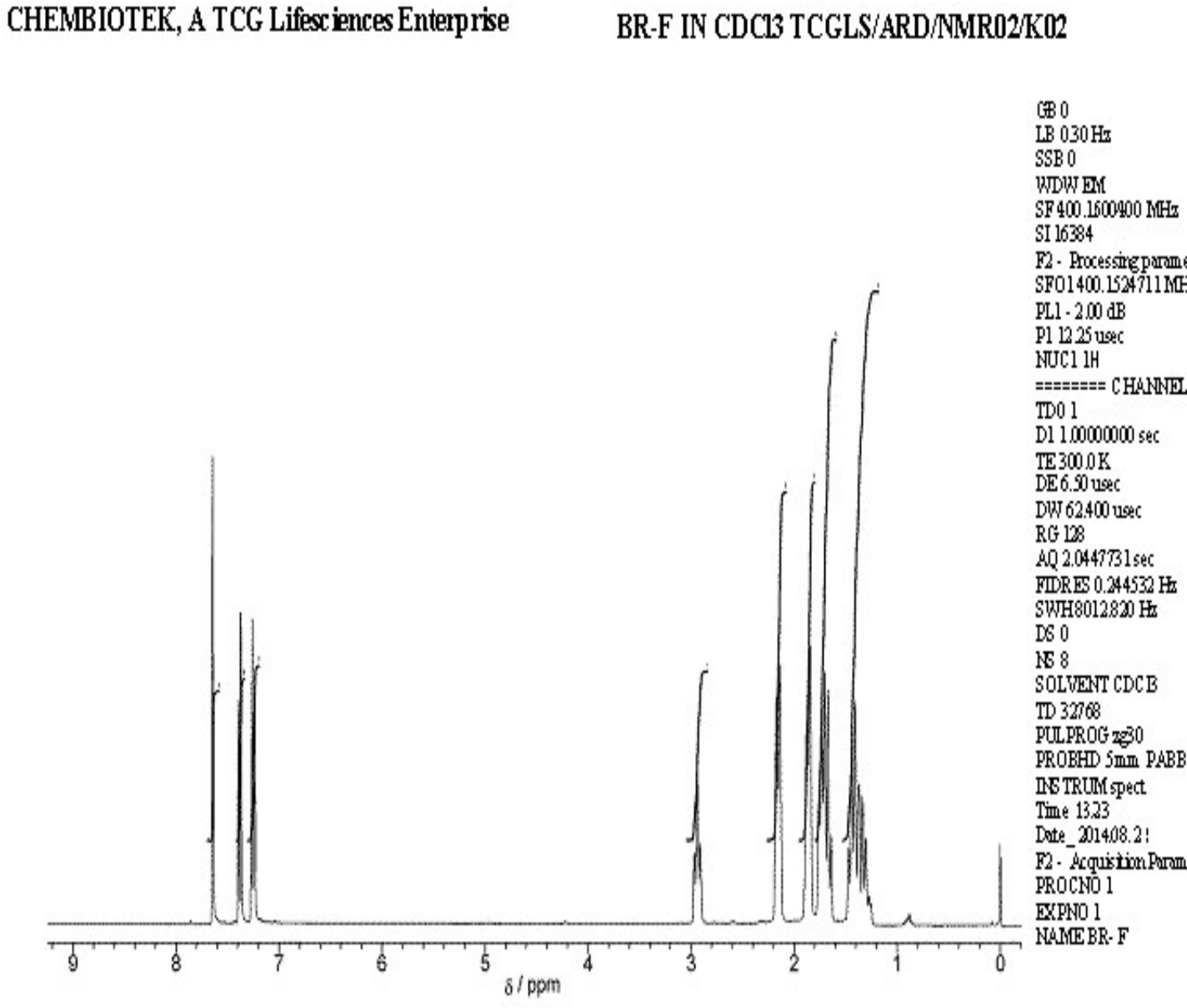
Compound 3m. ^{13}C NMR Spectrum ($CDCl_3$).



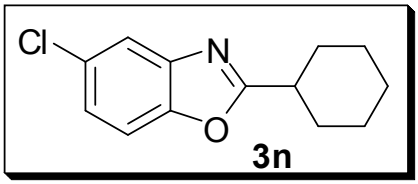


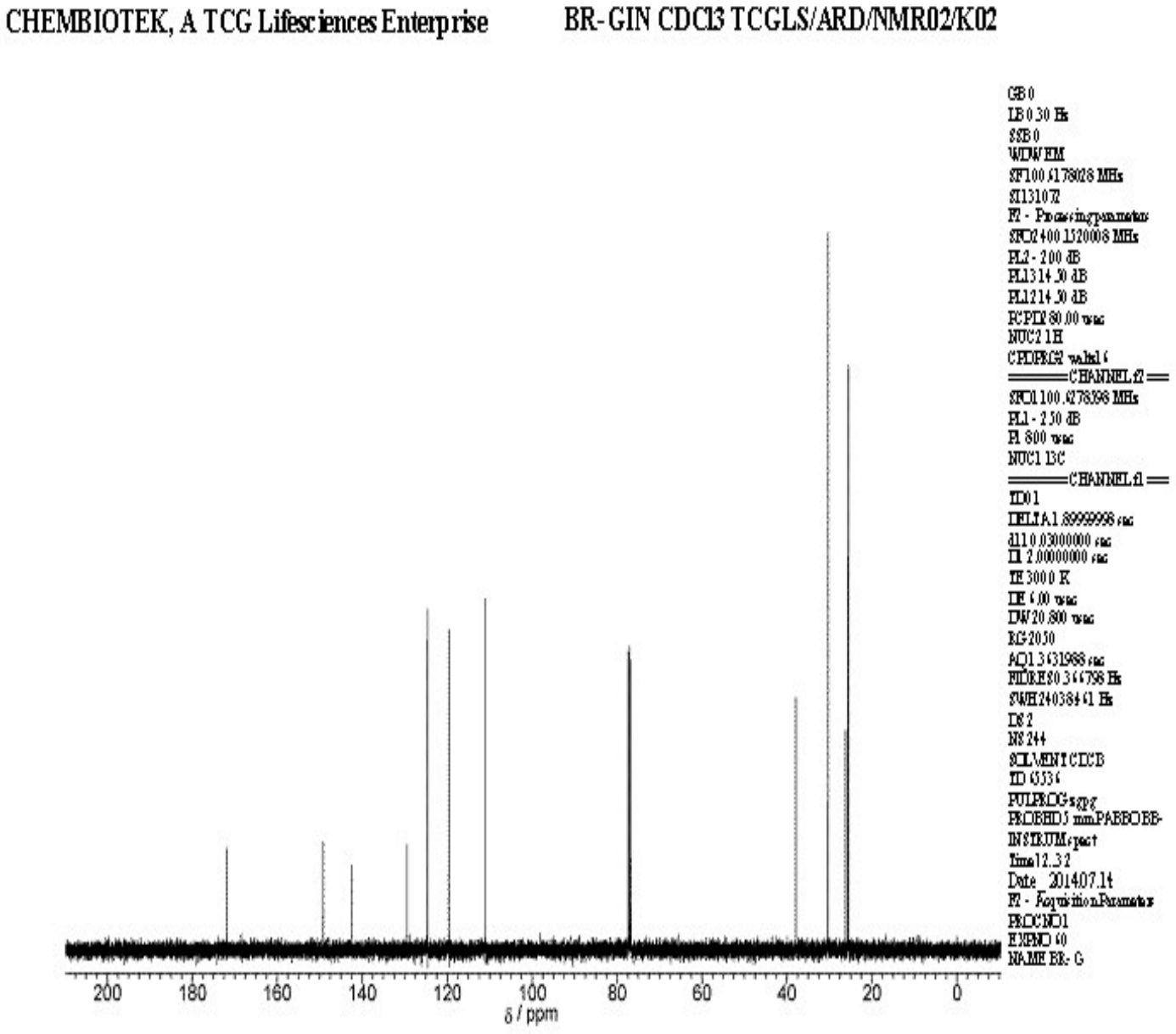
Compound 3n. ^{1}H NMR Spectrum ($CDCl_3$).



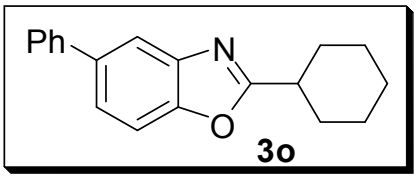


Compound 3n. ^{13}C NMR Spectrum ($CDCl_3$).

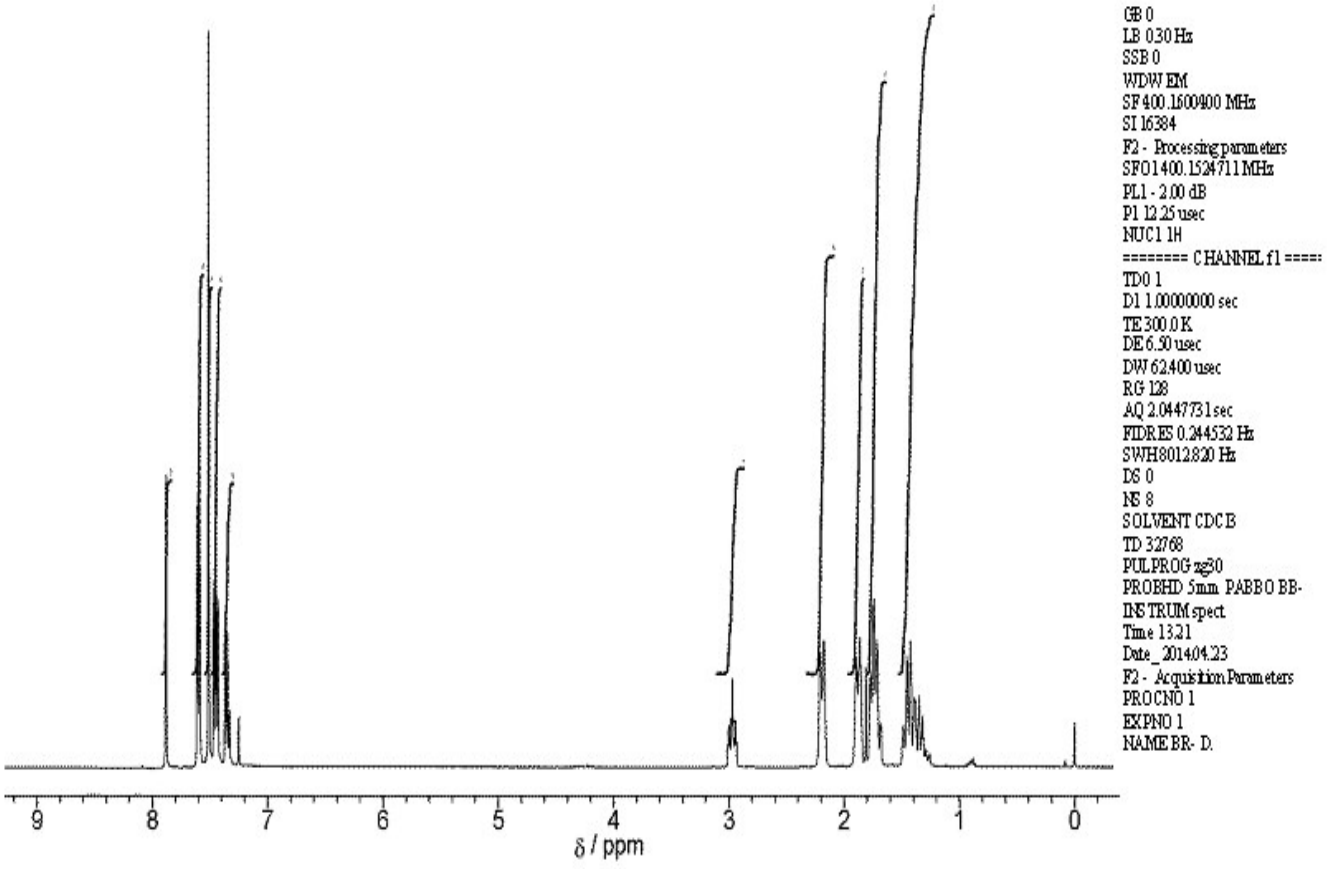




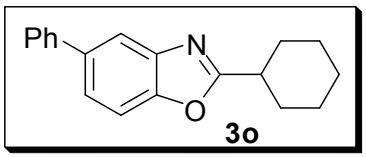
Compound 3o. ^{1}H NMR Spectrum ($CDCl_3$).

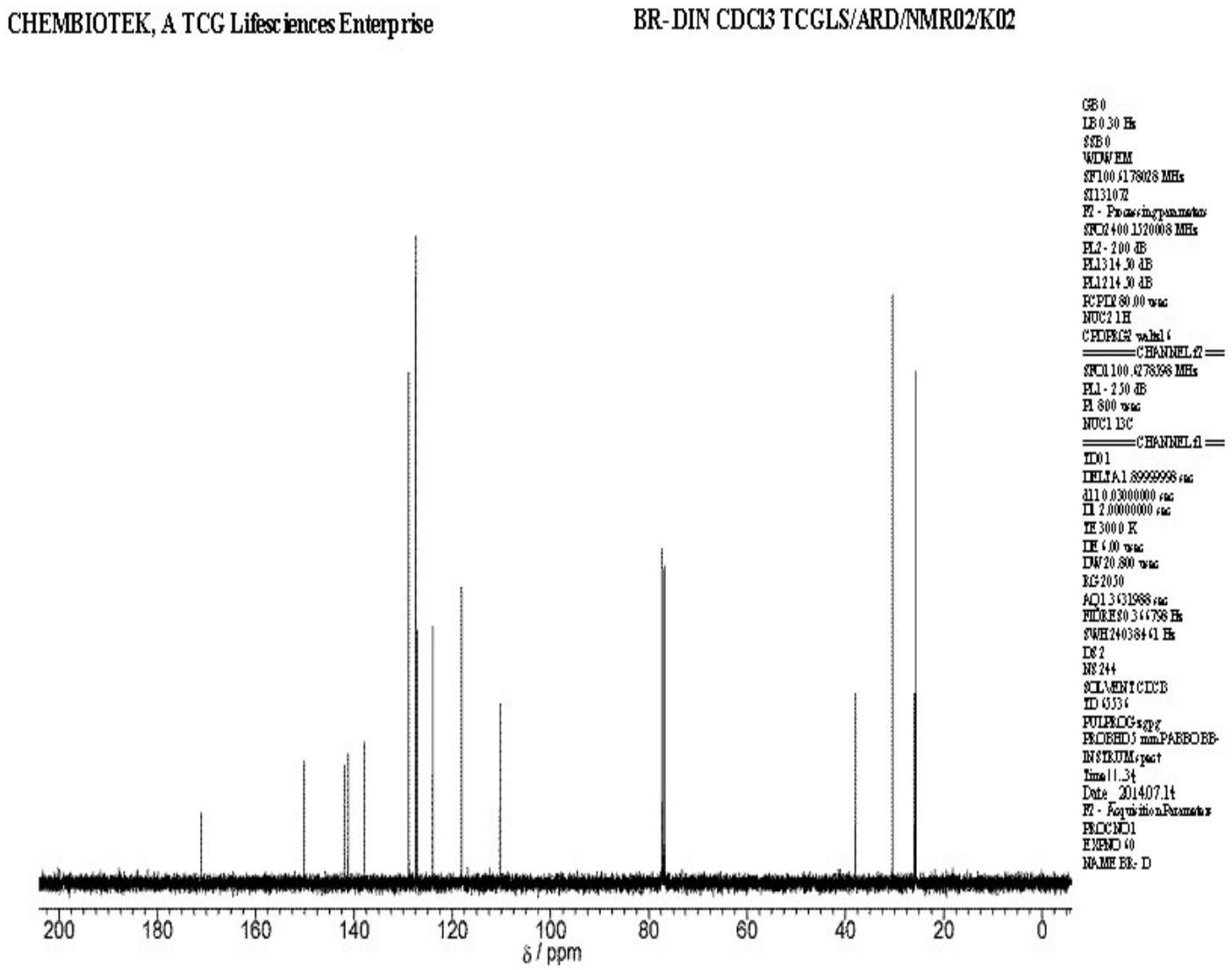


CHEMBIOTEK, A TCG Lifesciences Enterprise BR- DIN CDCl3 TCGLS/ARD/NMR02/K02

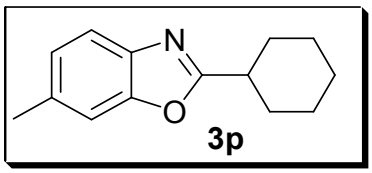


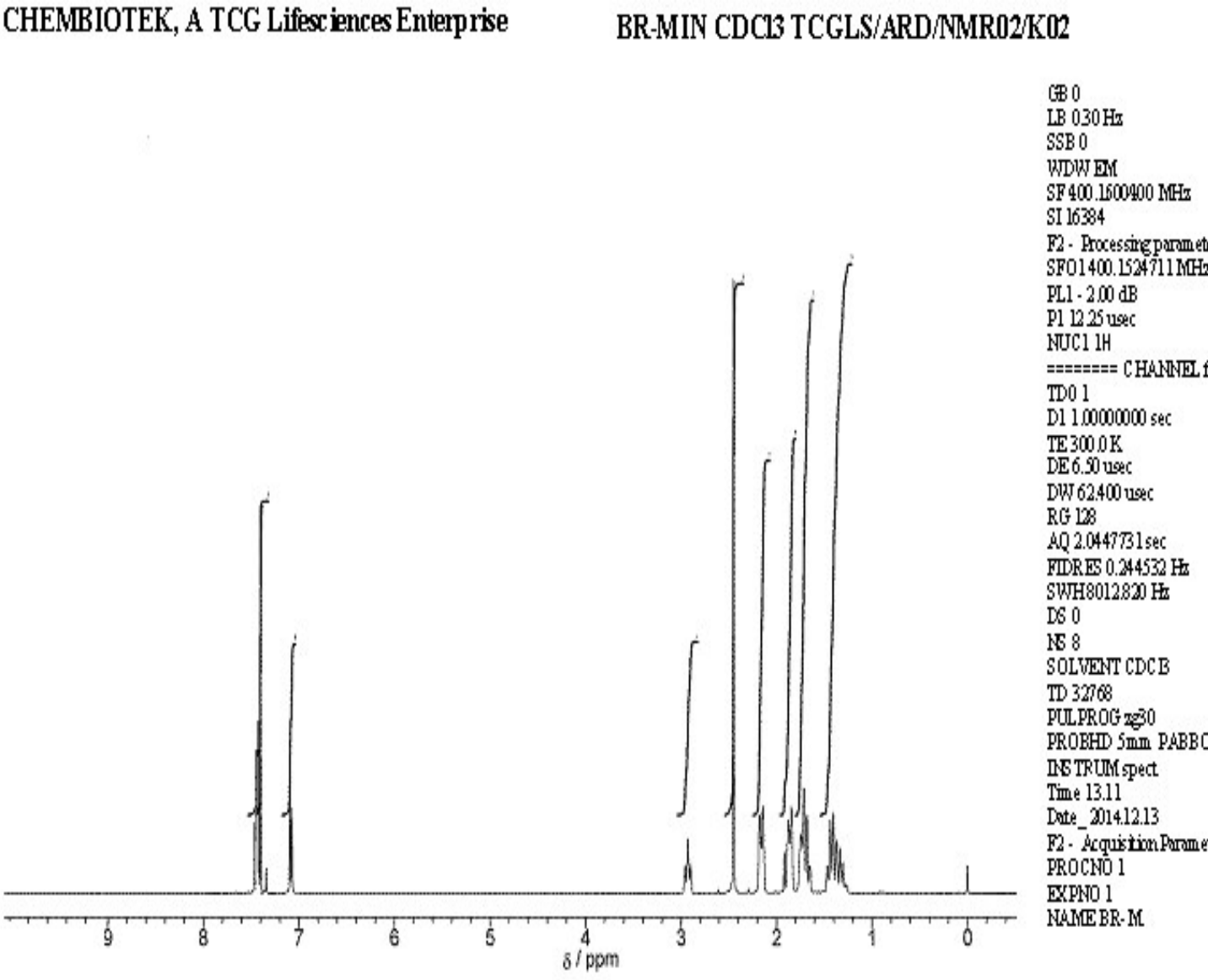
Compound 3o. ^{13}C NMR Spectrum ($CDCl_3$).



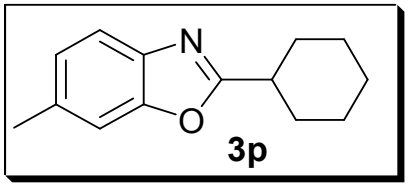


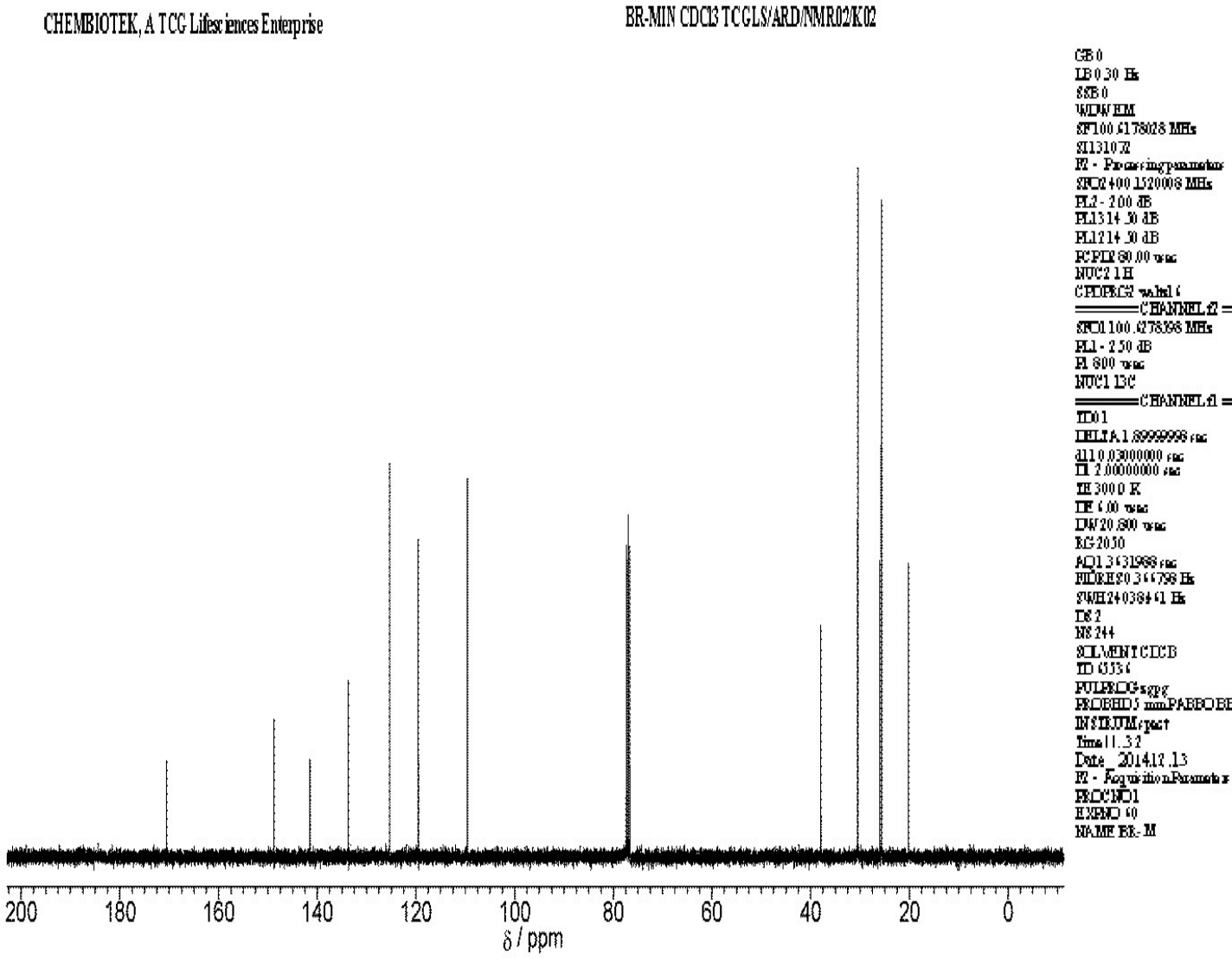
Compound 3p. ^{1}H NMR Spectrum ($CDCl_3$).



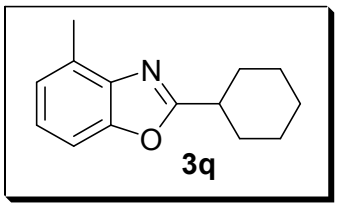


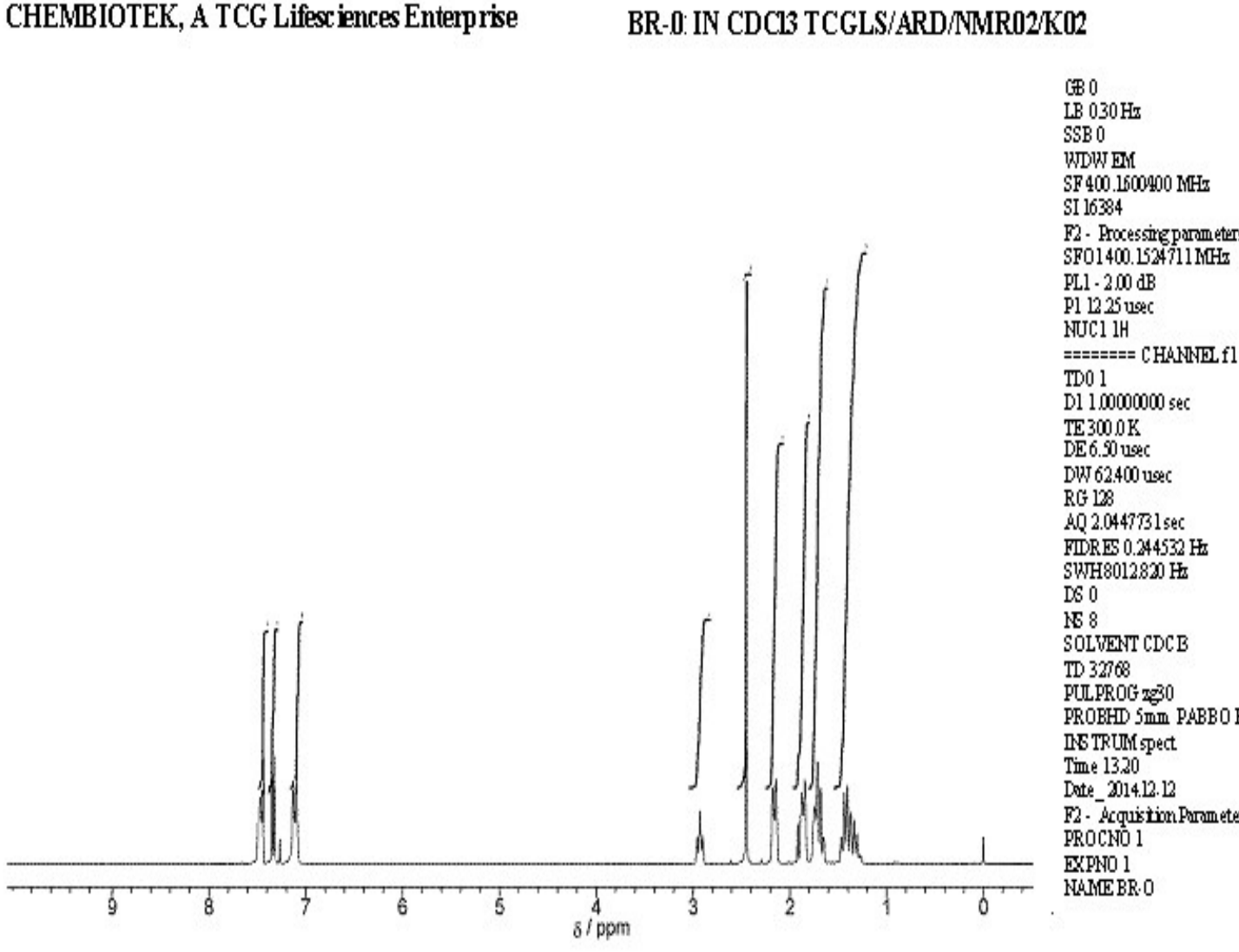
Compound 3p. ^{13}C NMR Spectrum ($CDCl_3$).



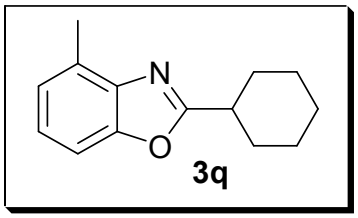


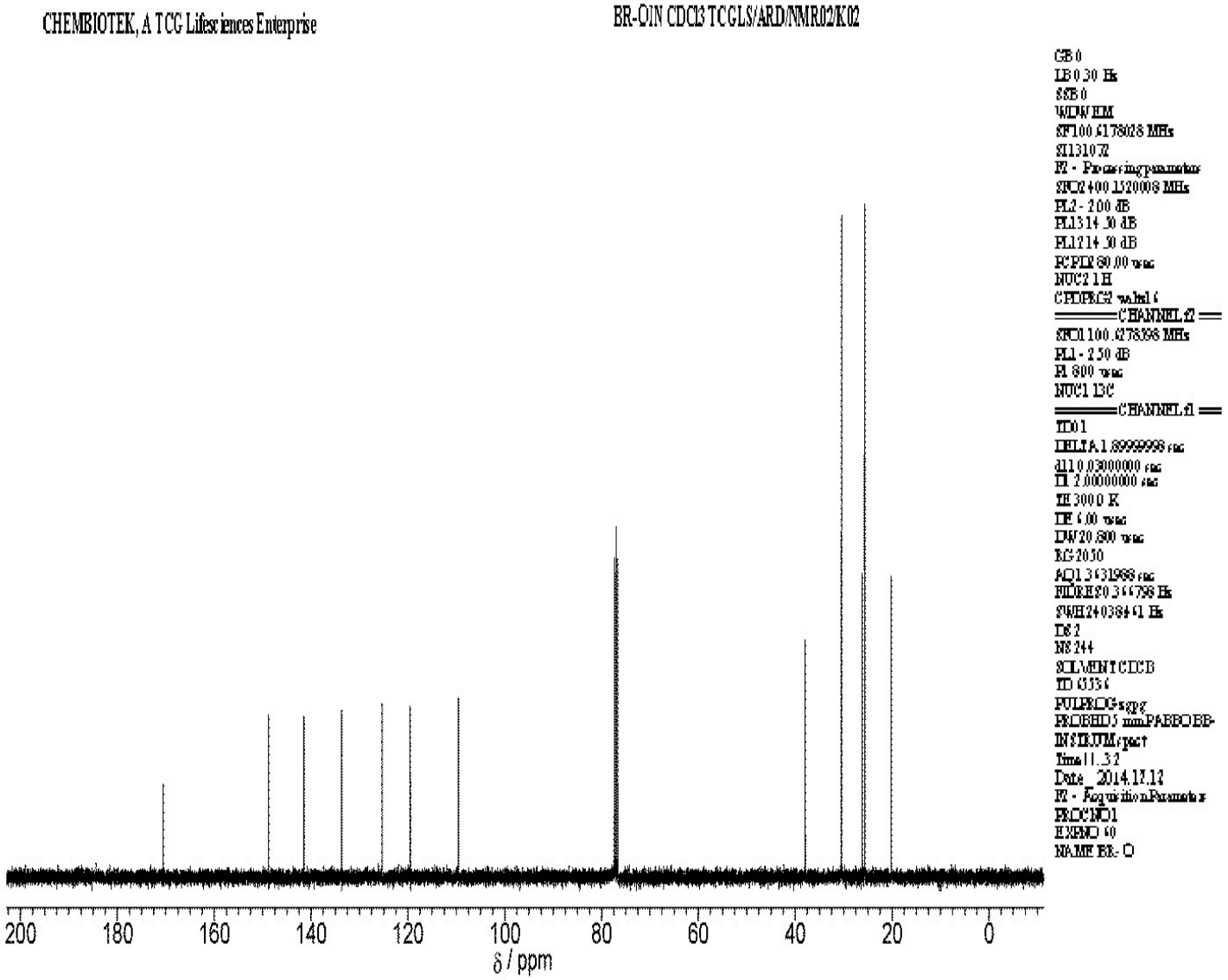
Compound 3q. ^{1}H NMR Spectrum ($CDCl_3$).





Compound 3q. ^{13}C NMR Spectrum ($CDCl_3$).





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