Efficient approach to 1,1′-bisindoles via copper(I)-catalyzed double domino reaction

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Supporting Information

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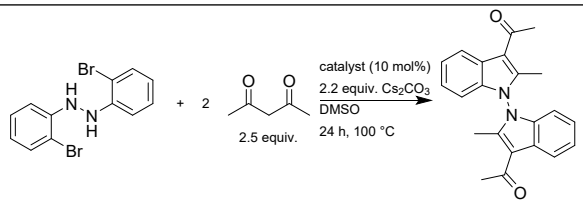
Optimization of the metal species

Table S1 Optimization of the catalyst [a,b]



1a **2a** **4a**

Entry	Catalyst (10 mol%)	Yield[a] (%)
11*	MoO_3	-
12*	Ferrocene carboxaldehyde $C_{11}H_{10}FeO$	-
13*	$FeCl_3$	-
14*	$RhCl_3\ H_2O$	-
15*	$IrCl_3$	-
16*	$NiSO_4\ 6H_2O$	-
17*	$Pd(OAc)_2$	-
18*	$Pd(OAc)_2$, DABCO (3 equiv.)	-
19*	Pd(dba), NaOAc (6 equiv.), XPhos (20 mol%)	-
20*	$PdCl_2(PPh_3)_2$	-
21*	$PdCl_2$	-
22*	$PtCl_2$	-
23*	AgSbCl	-
24*	$AuCl_3$	-
25*	$AuCl_3$ / AgSbCl = 1:1 (5% each)	-
26*	$AuCl(PPh_3)$	-
27*	$AuCl(PPh_3)$ / AgSbCl = 1:1 (5% each)	-
28*	$ZnCl_2$	-
29*	$InCl_3$	-
30*	$AlCl_3$	-

[a] For entries 1-10 see paper.

[b] 0.25 mmol **1a** were reacted in 1 mL DMSO. All yields refer to isolated yields.

NMR spectra of nitrobenzenes 5e and 5f

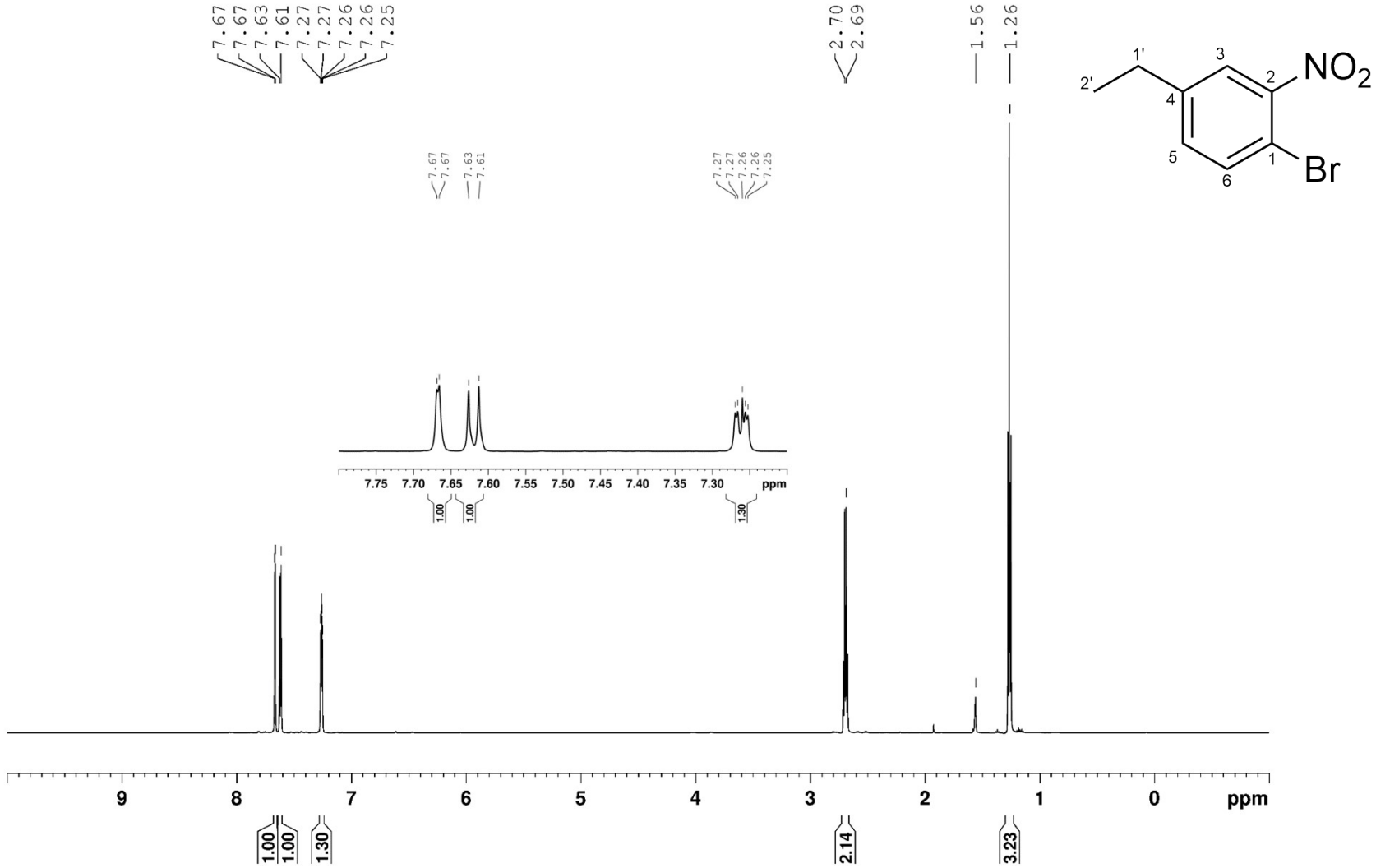


Fig. 1 ^{1}H (600 MHz) NMR spectra of **5e** in $CDCl_3$

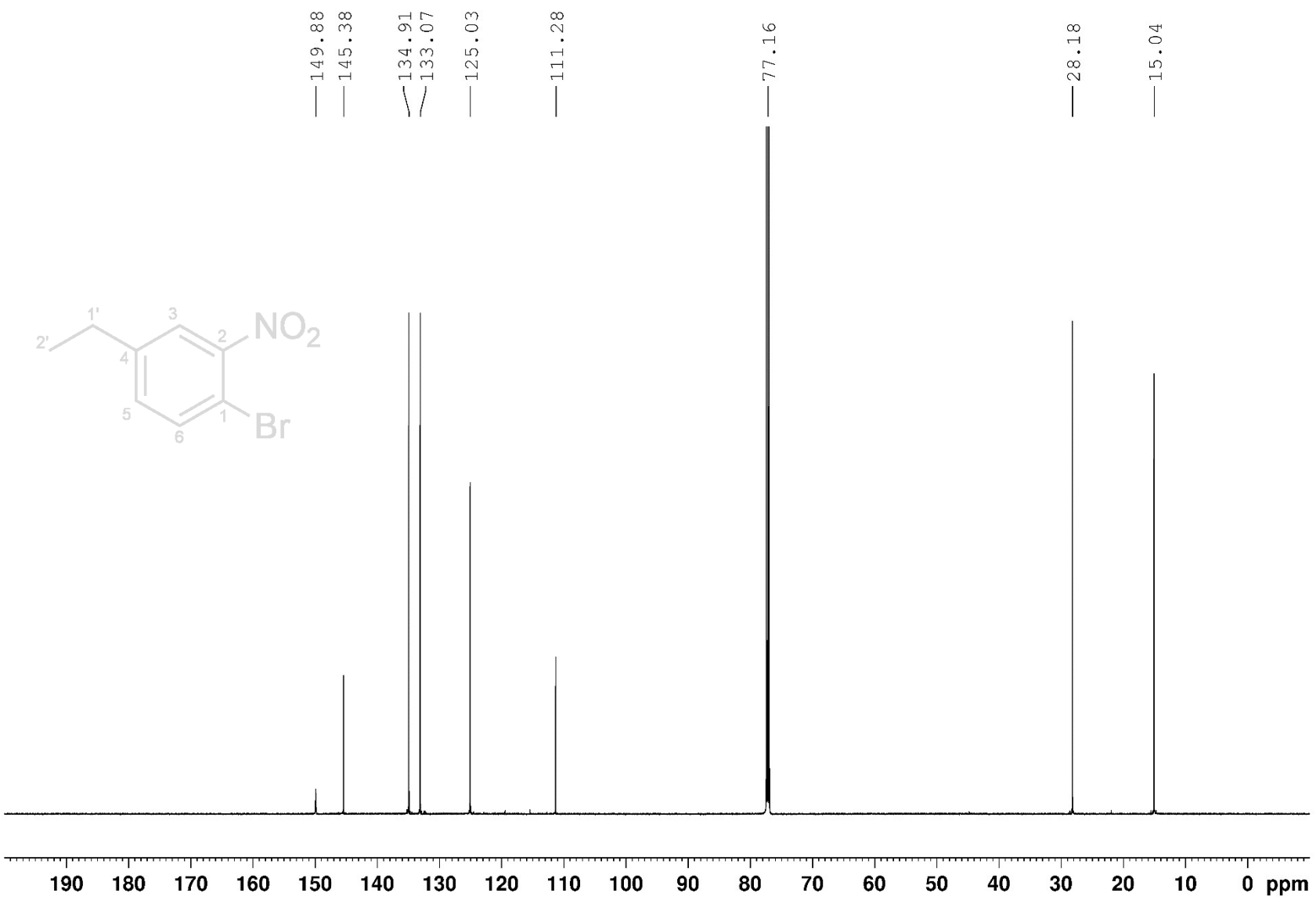


Fig. 2 ^{13}C (150 MHz) NMR spectra of **5e** in $CDCl_3$

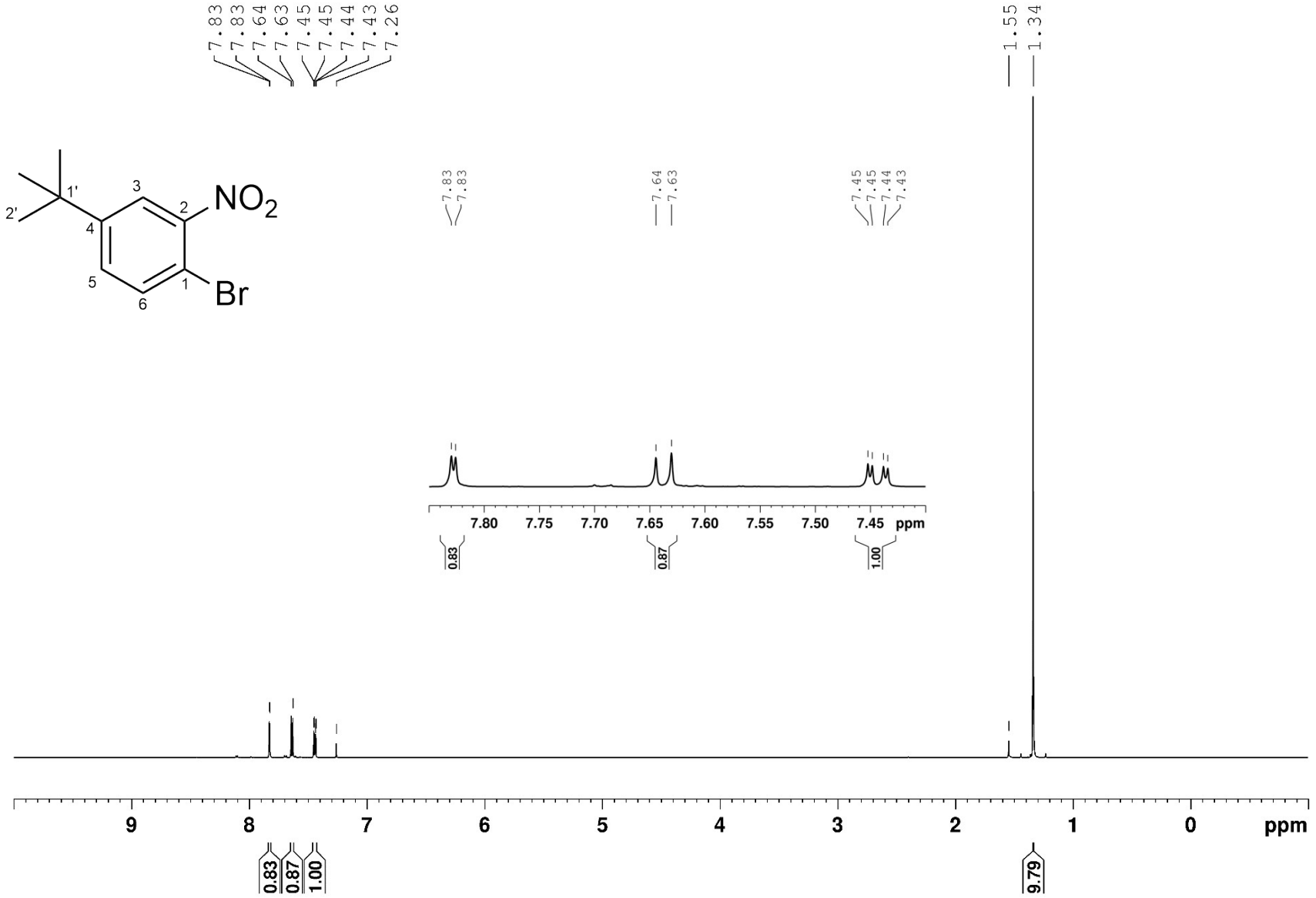


Fig. 3 ^{1}H (600 MHz) spectra of **5f** in $CDCl_3$

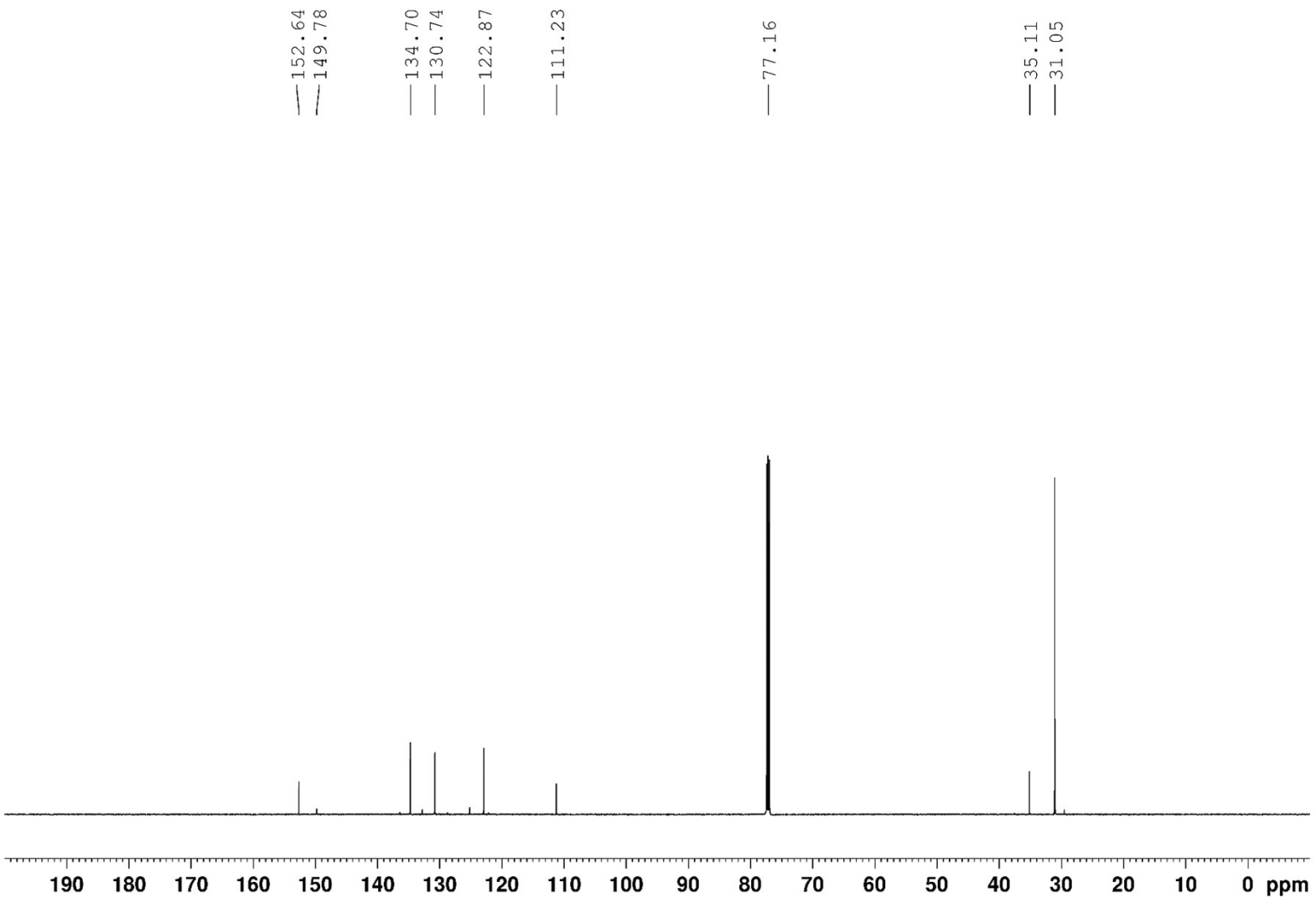


Fig. 4 ^{13}C (150 MHz) NMR spectra of **5f** in $CDCl_3$

NMR spectra of hydrazines 1a-h

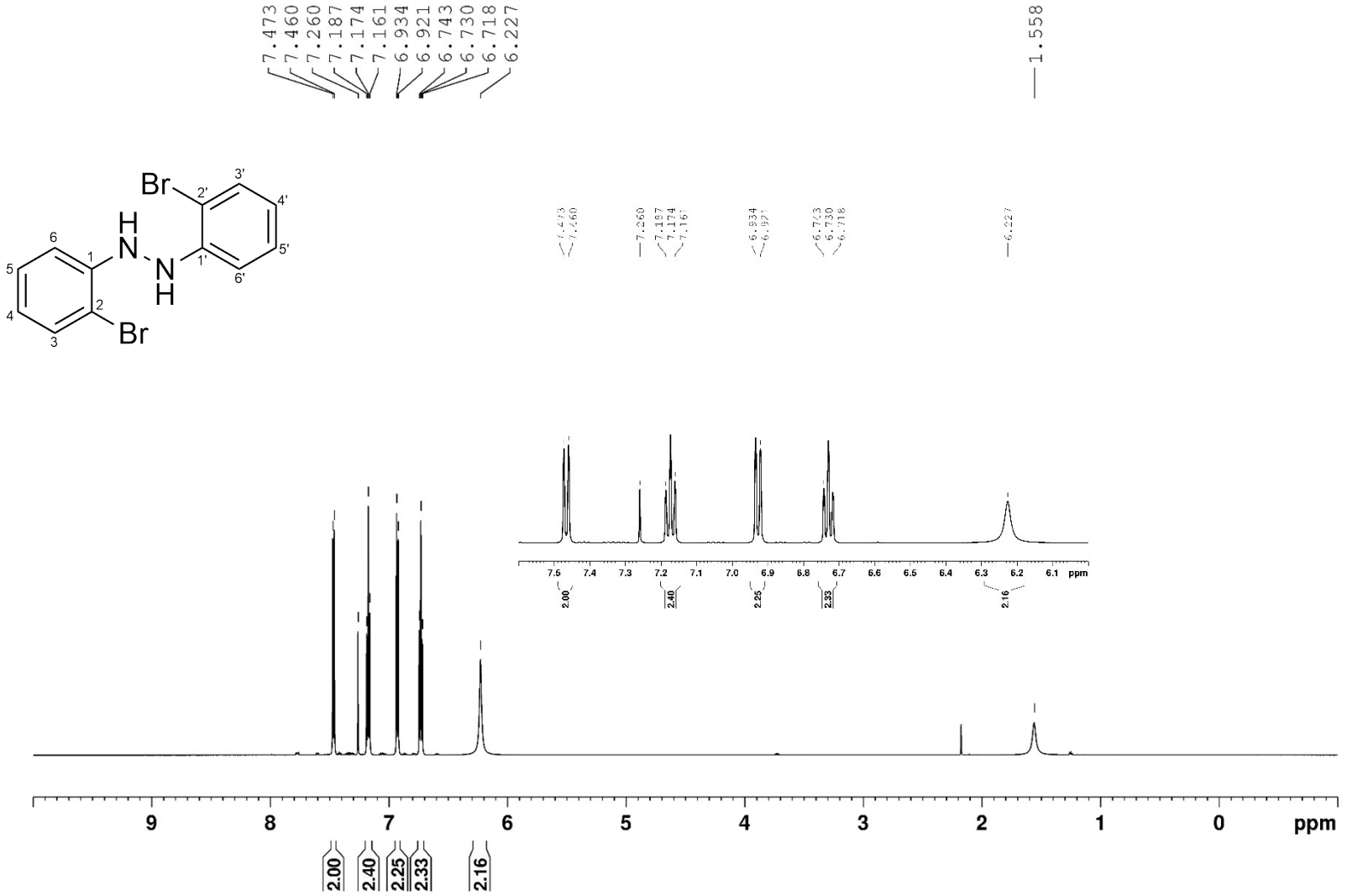


Fig. 5 ^{1}H (150 MHz) NMR spectra of **1a** in $CDCl_3$

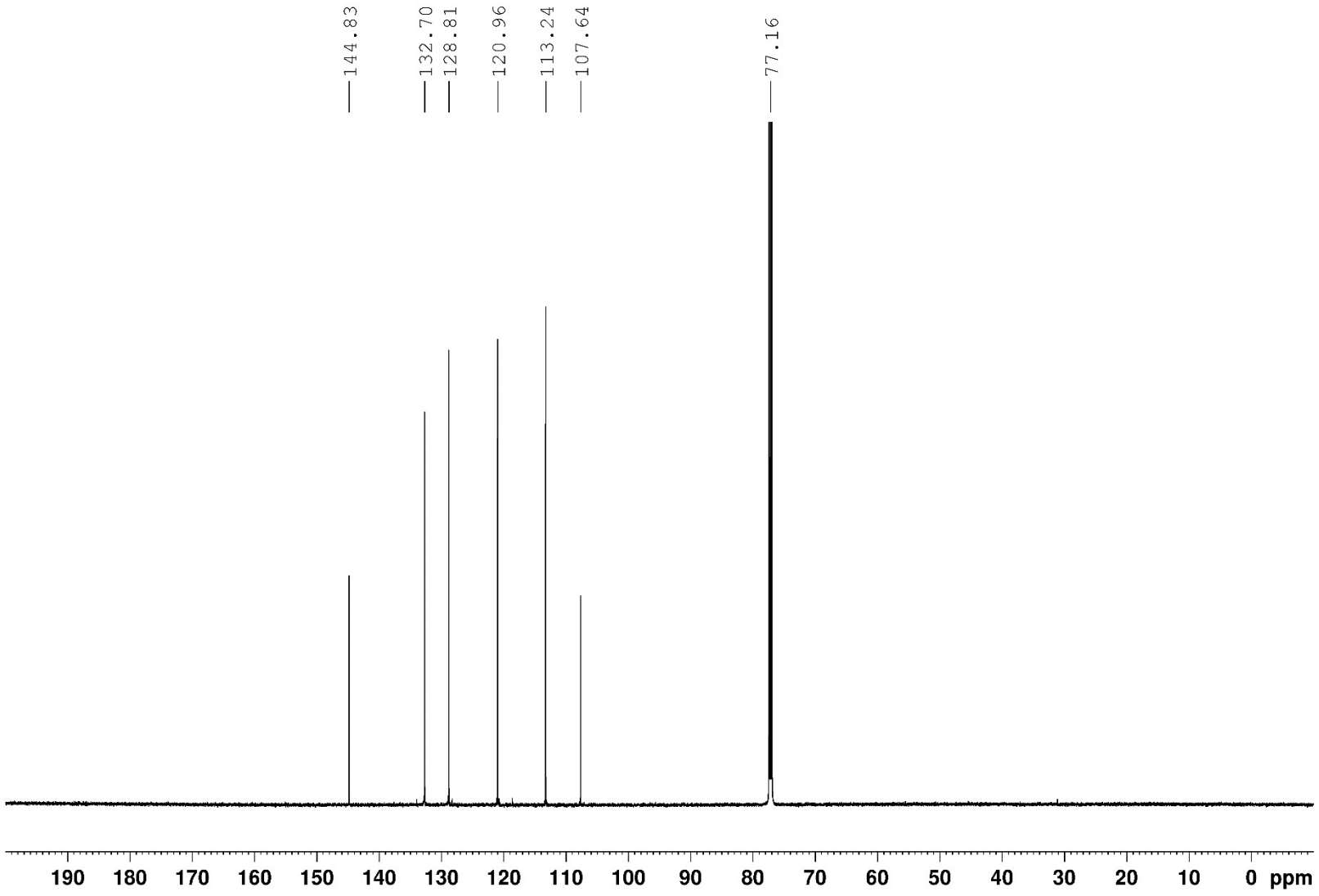


Fig. 6 ^{13}C (150 MHz) NMR spectra of **1a** in $CDCl_3$

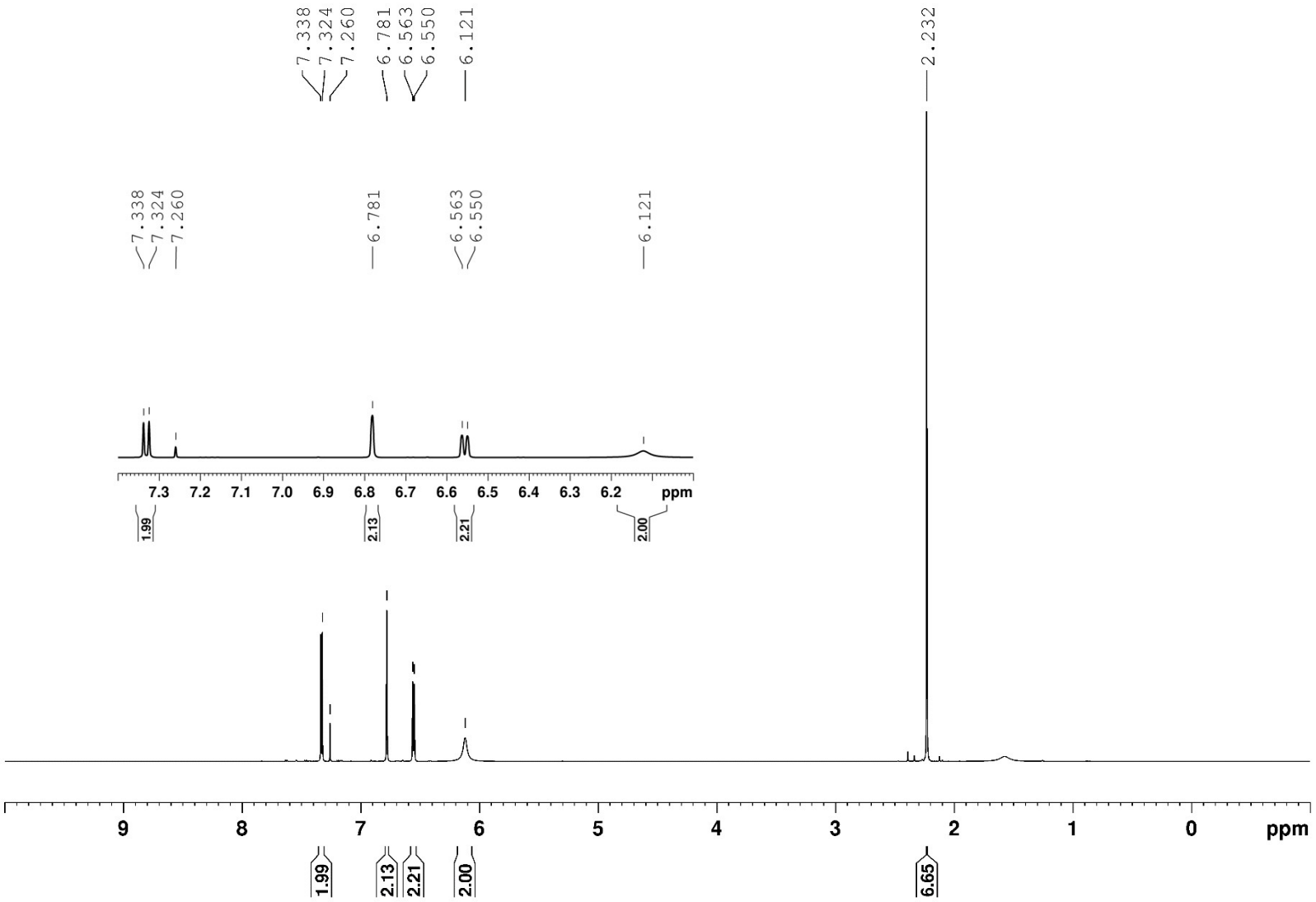


Fig. 7 ^{1}H (600 MHz) NMR spectra of **1b** in $CDCl_3$

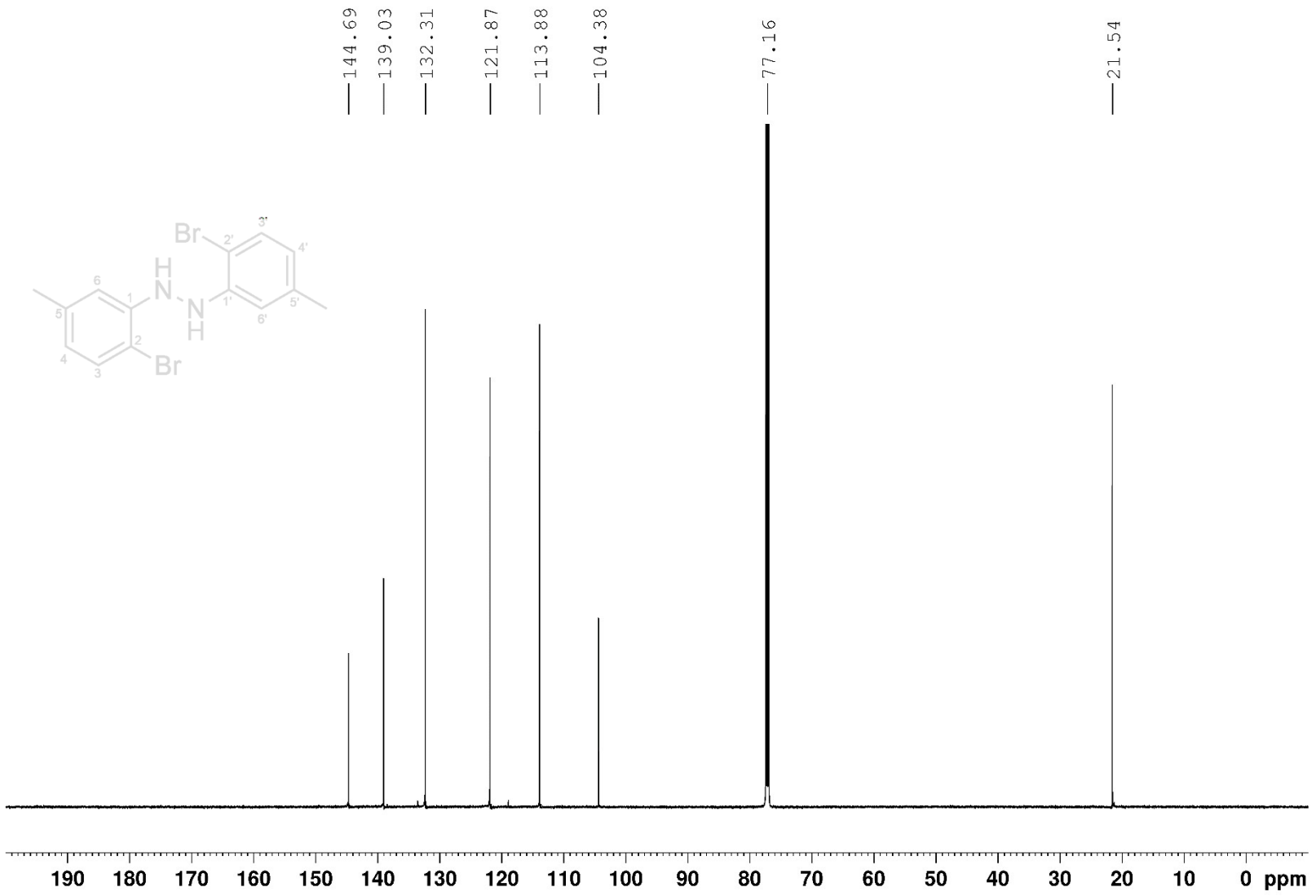


Fig. 8 ^{13}C (150 MHz) NMR spectra of **1b** in $CDCl_3$

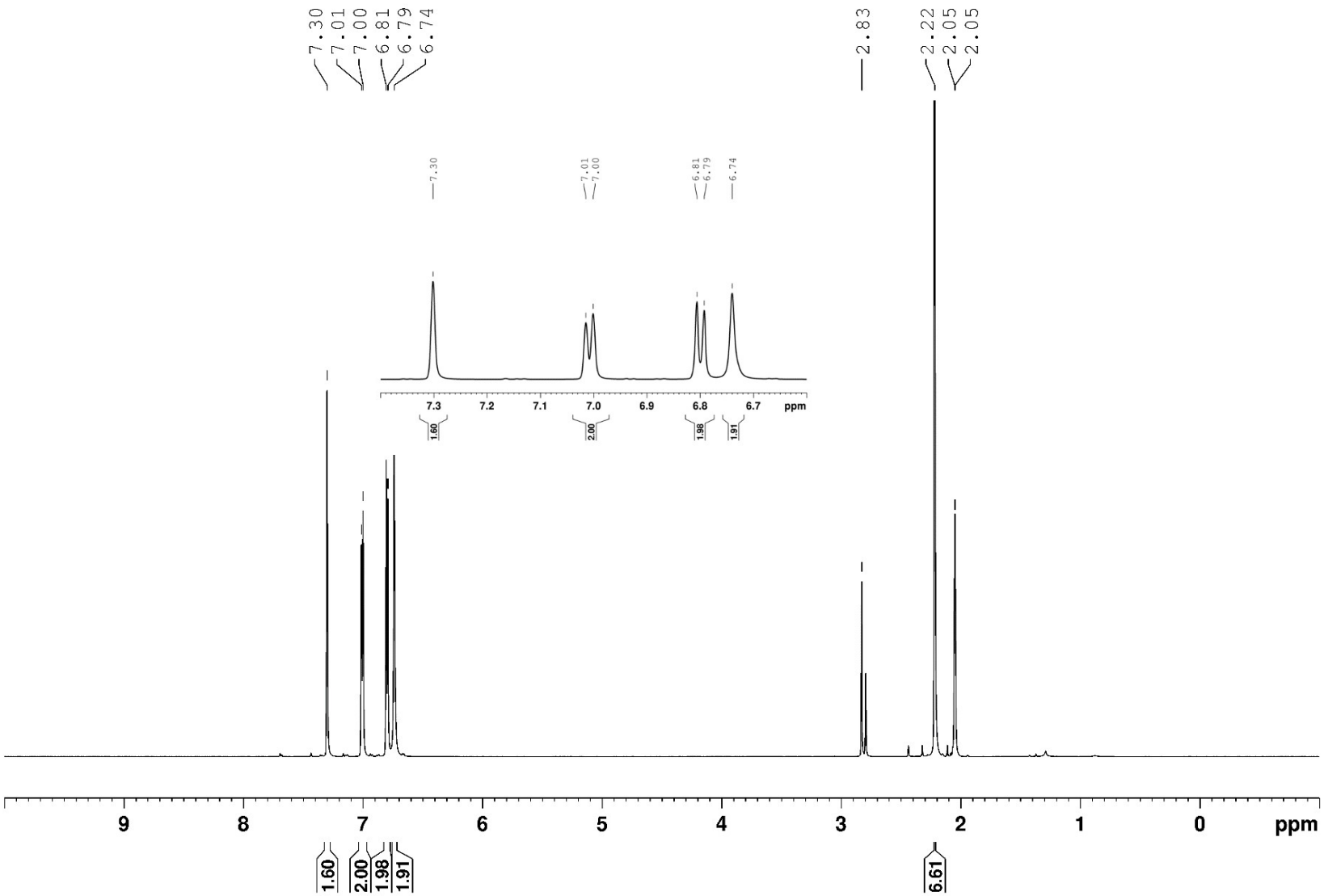


Fig. 9 ^{1}H (600 MHz) NMR spectra of **1c** in $(CD_3)_2CO$

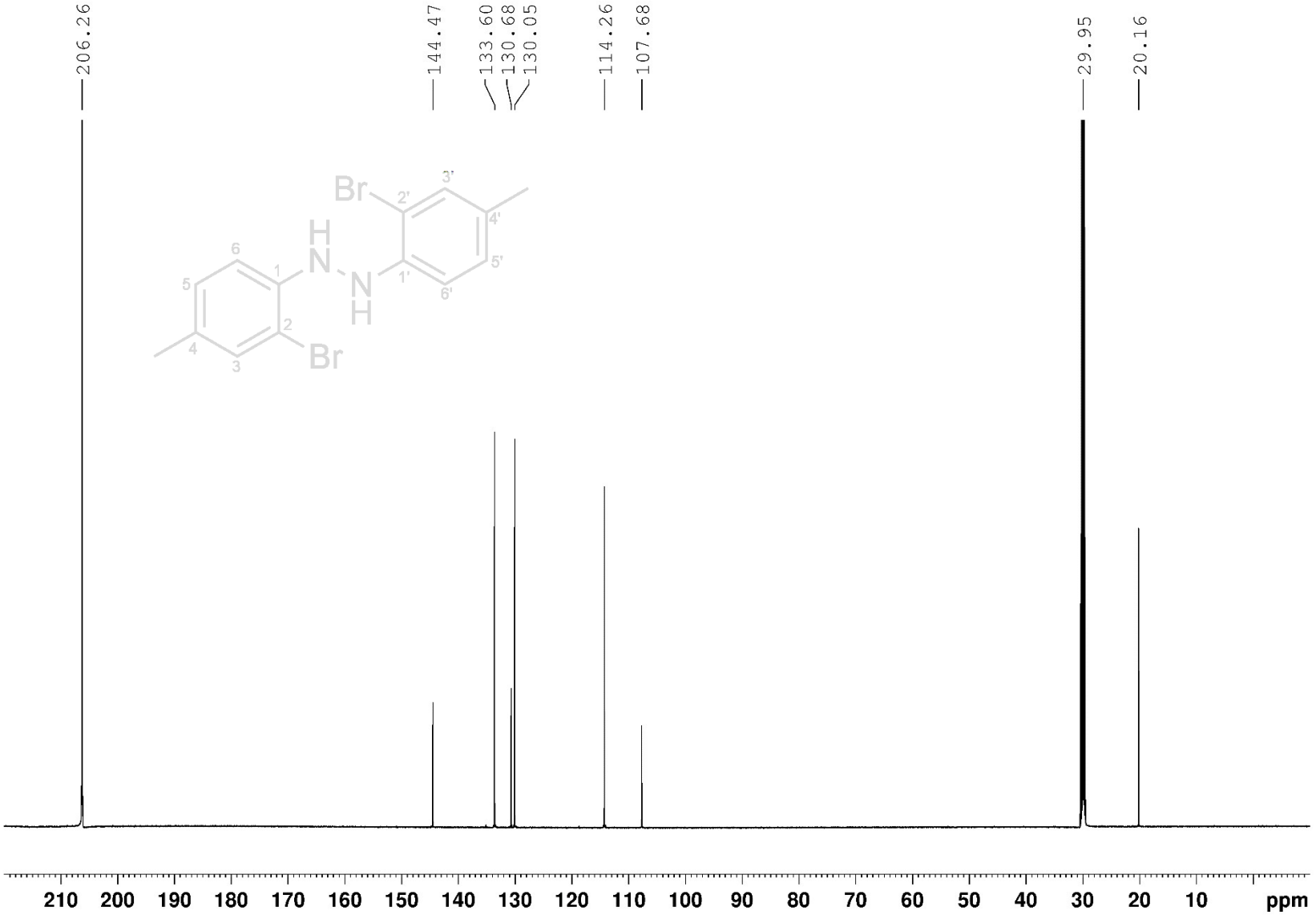


Fig. 10 ^{13}C (150 MHz) NMR spectra of **1c** in $(CD_3)_2CO$

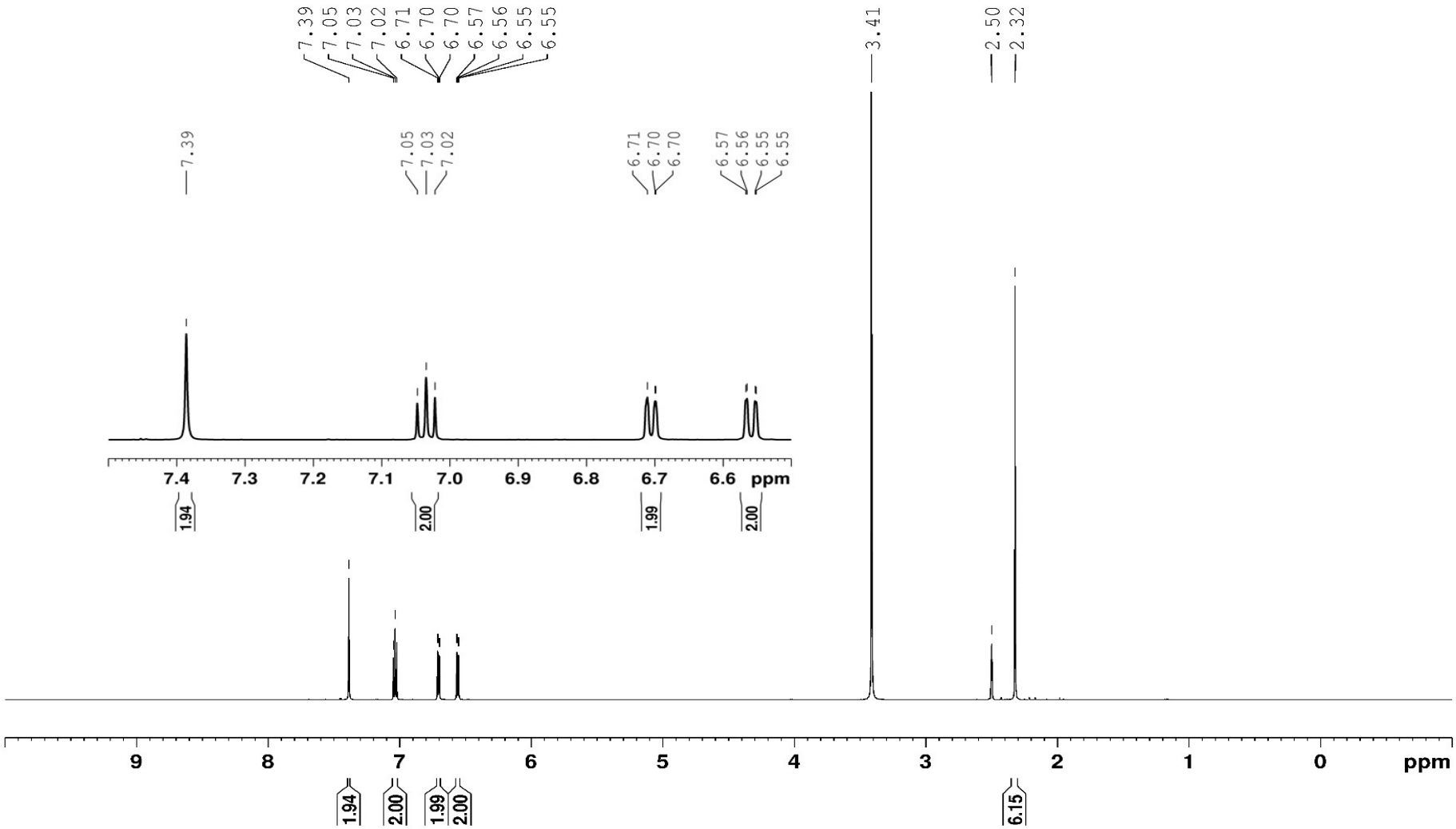


Fig. 11 ^{1}H (600 MHz) NMR spectra of **1d** in $(CD_3)_2SO$

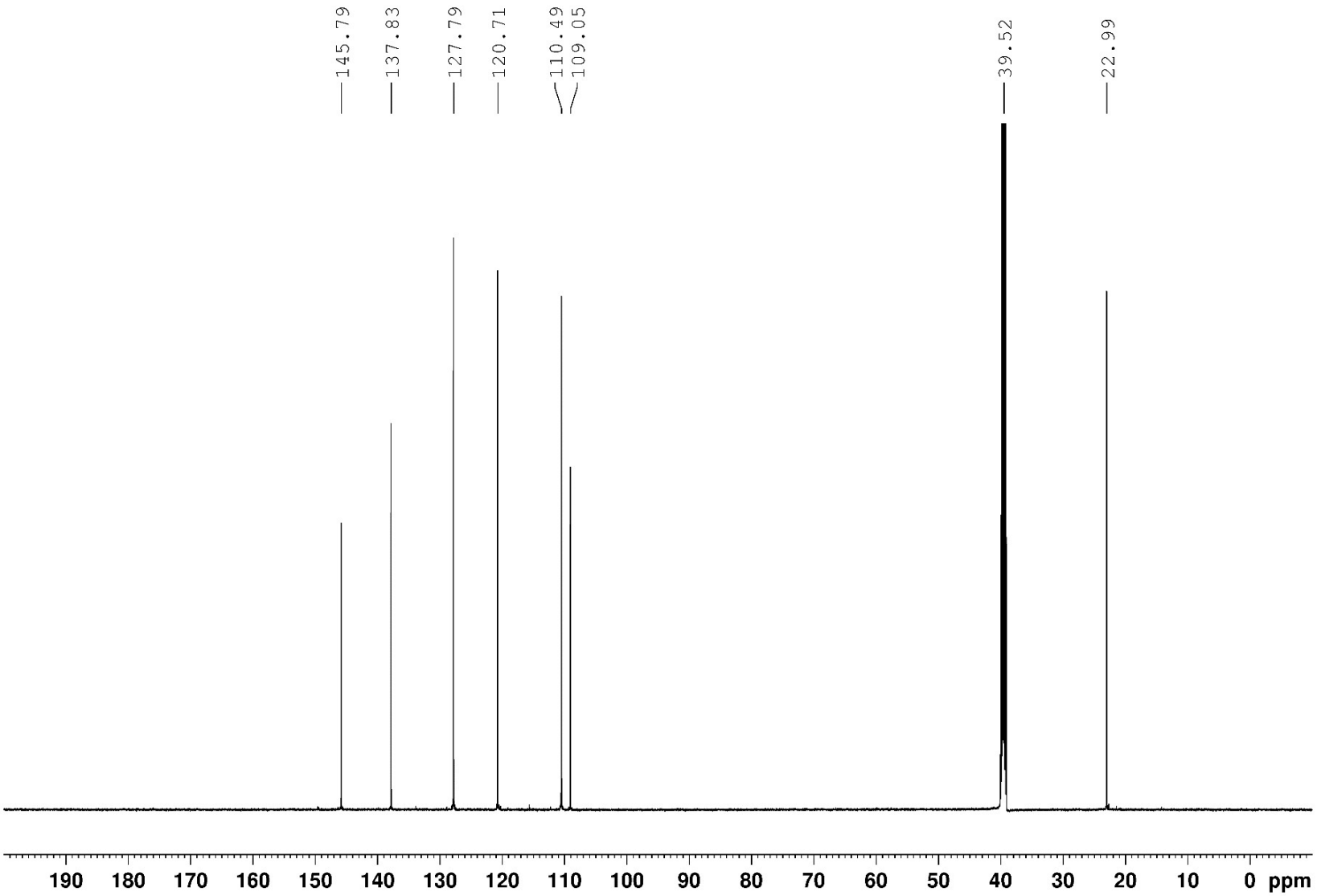


Fig. 12 ^{13}C (150 MHz) NMR spectra of **1d** in $(CD_3)_2SO$

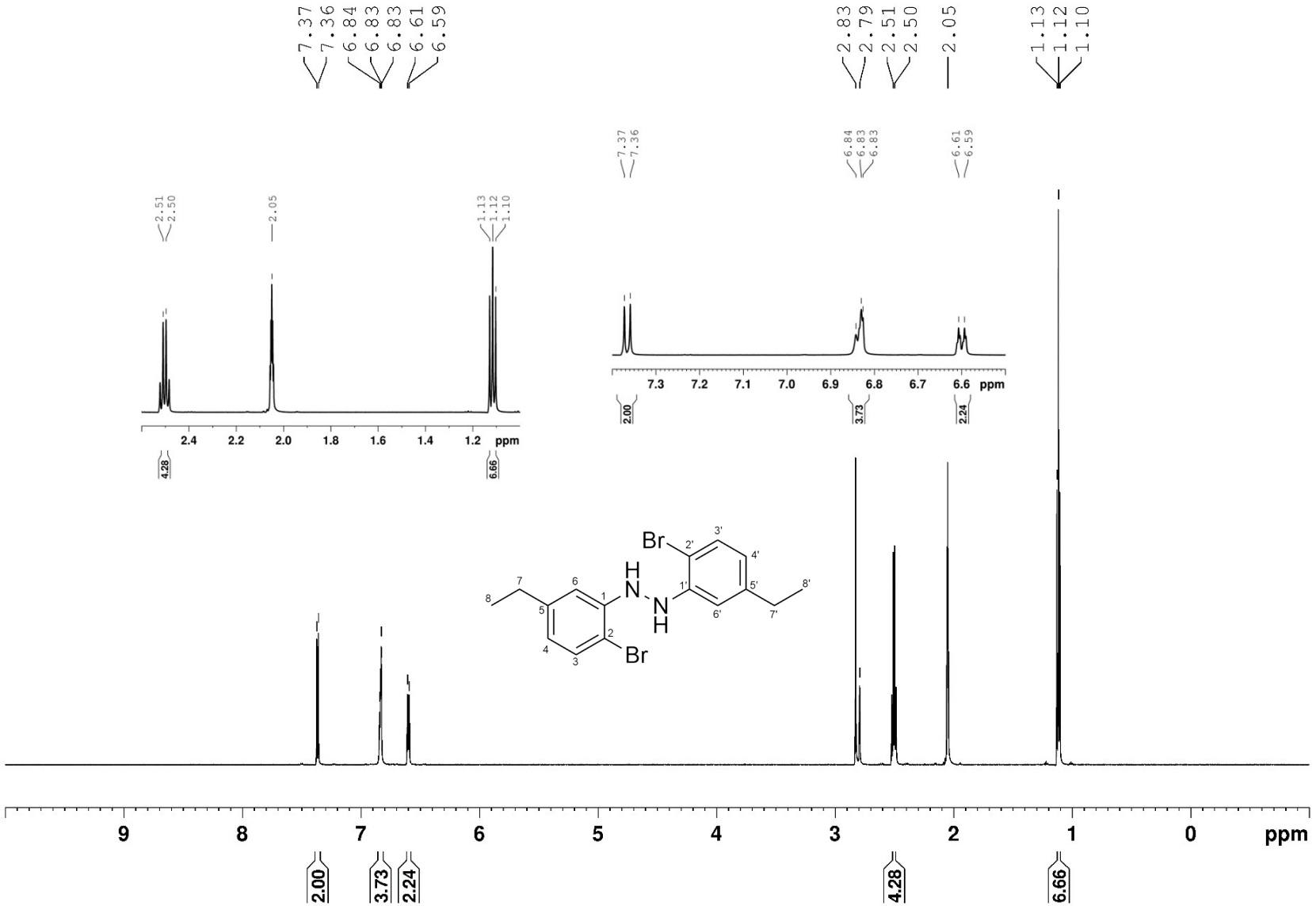


Fig. 13 ^{1}H (600 MHz) NMR spectra of **1e** in $(CD_3)_2CO$

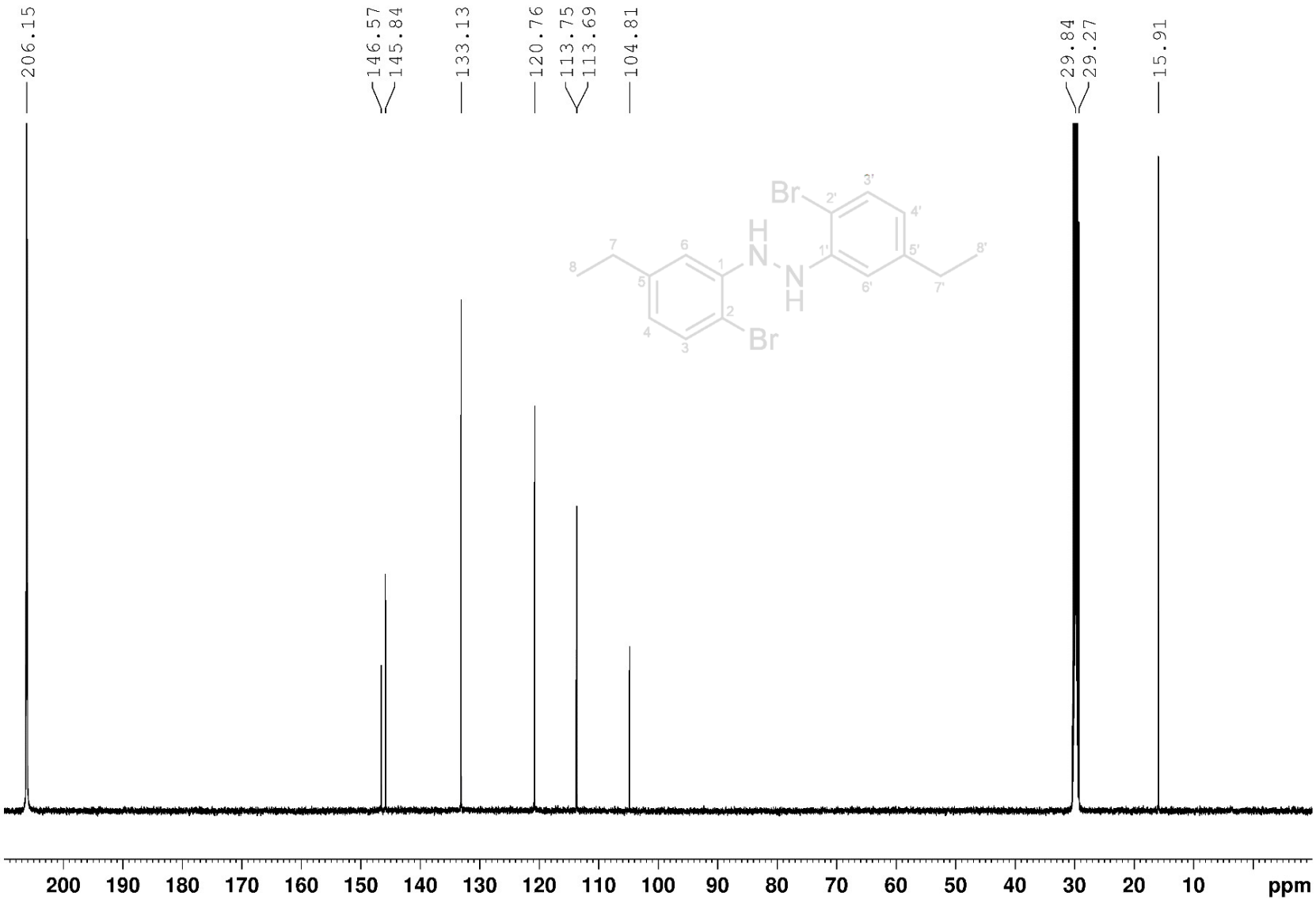


Fig. 14 ^{13}C (150 MHz) NMR spectra of **1e** in $(CD_3)_2CO$

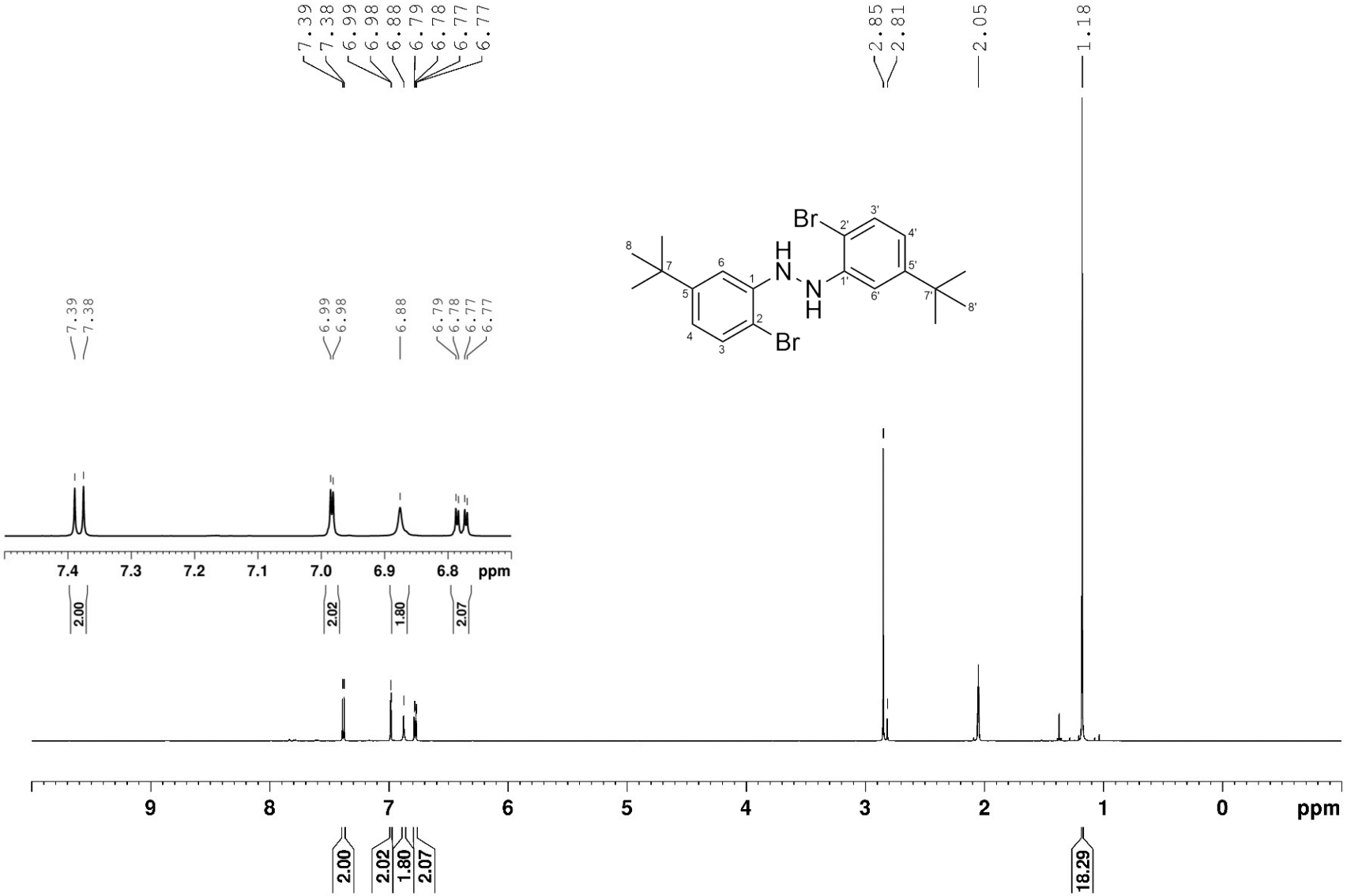


Fig. 15 ^{1}H (600 MHz) NMR spectra of **1f** in $(CD_3)_2CO$

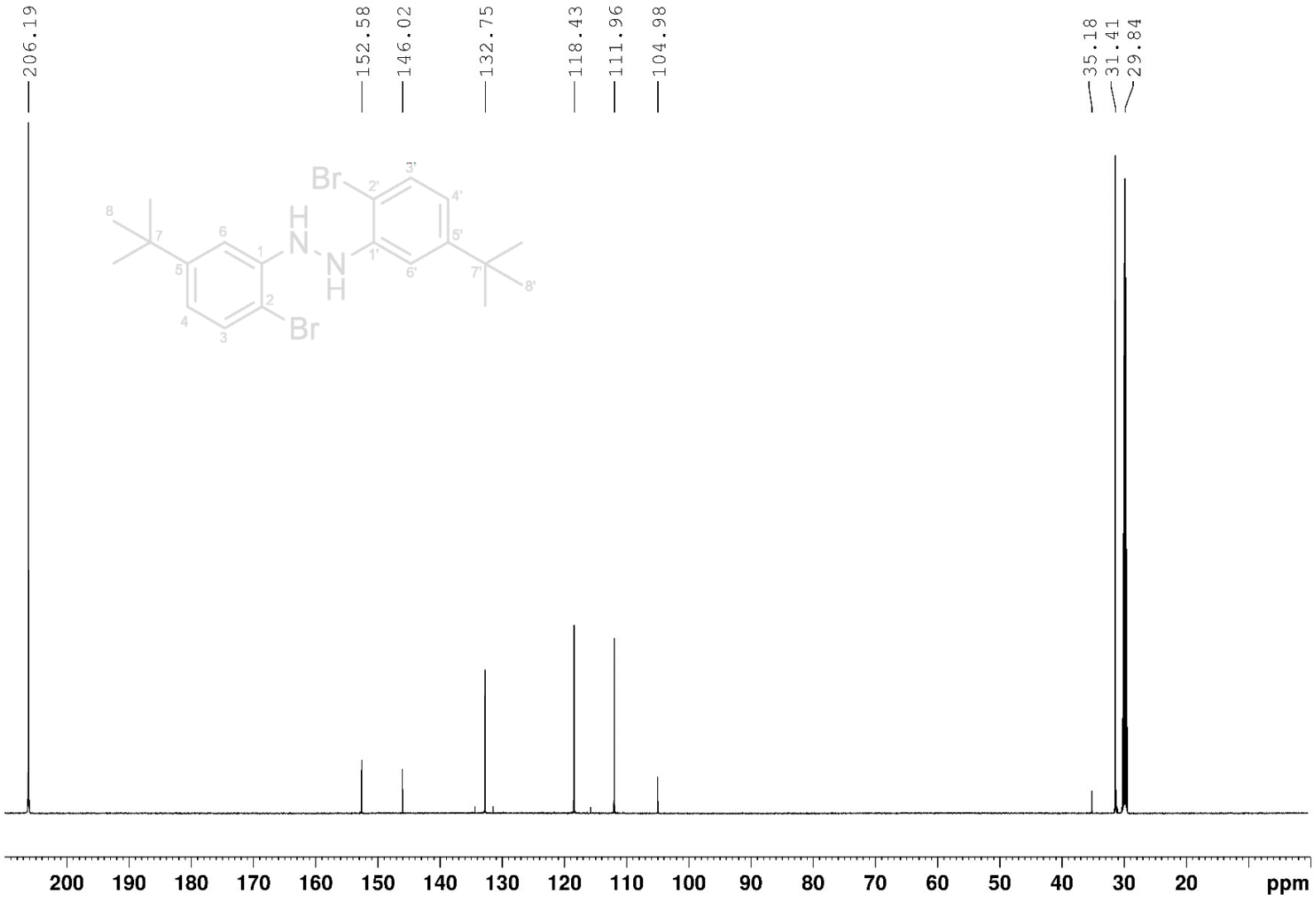


Fig. 16 ^{13}C (150 MHz) NMR spectra of **1f** in $(CD_3)_2CO$

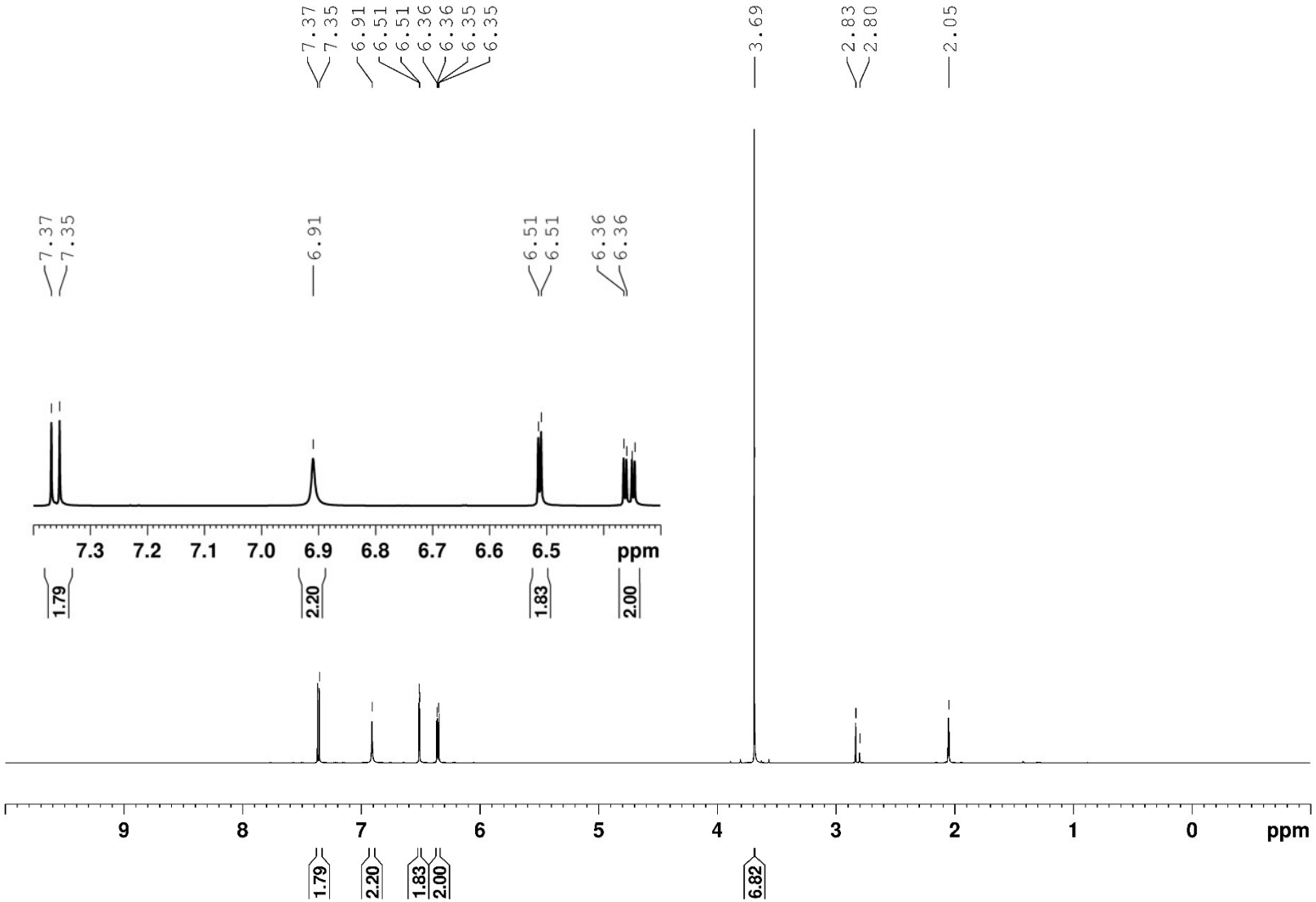


Fig. 17 ^{1}H (600 MHz) NMR spectra of **1g** in $(CD_3)_2CO$

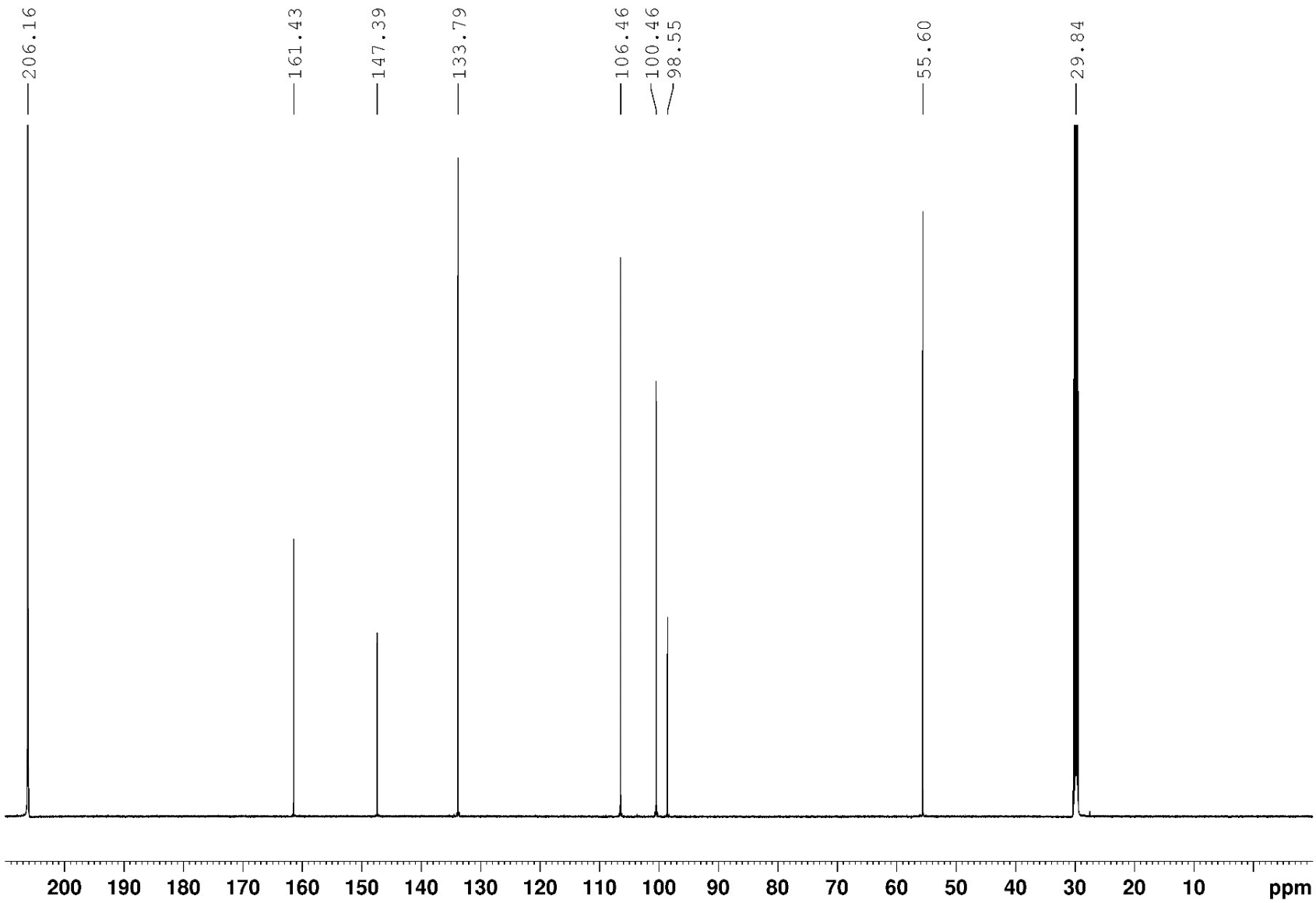


Fig. 18 ^{13}C (150 MHz) NMR spectra of **1g** in $(CD_3)_2CO$

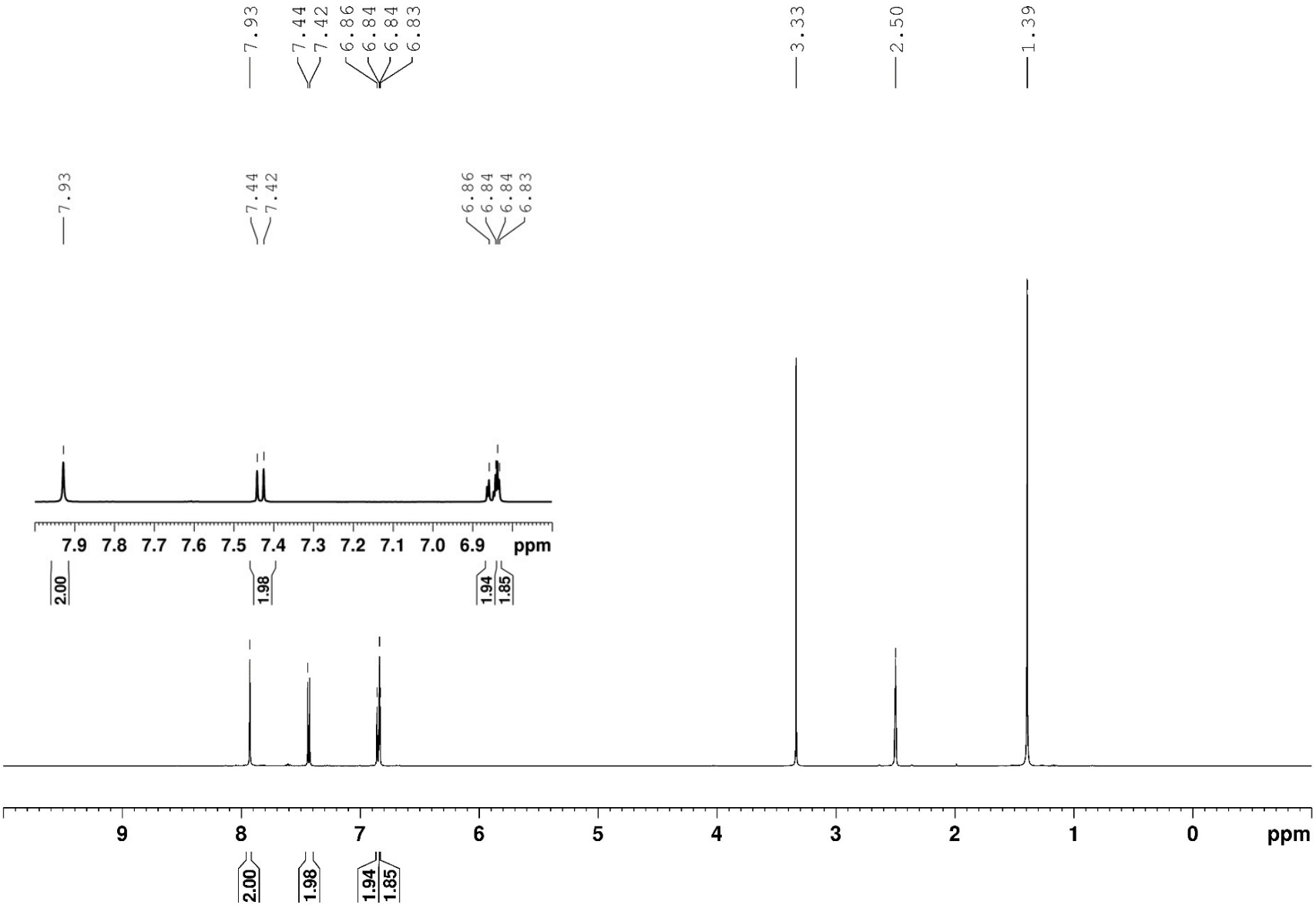


Fig. 19 ^{1}H (500 MHz) NMR spectra of **1h** in $(CD_3)_2SO$

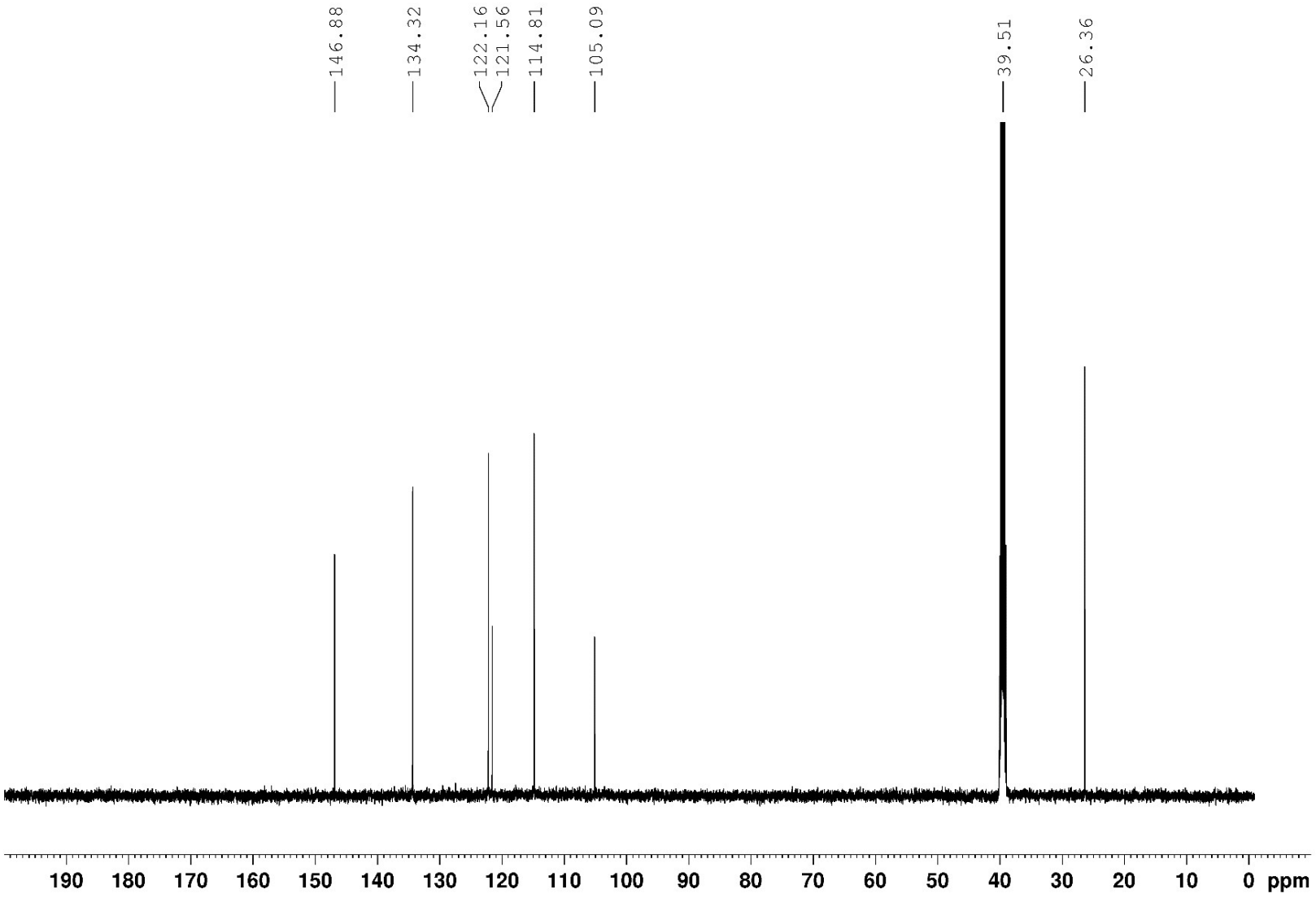


Fig. 20 ^{13}C (125 MHz) NMR spectra of **1h** in $(CD_3)_2SO$

NMR spectra of bisindoles 4a-l, 8h und 8j

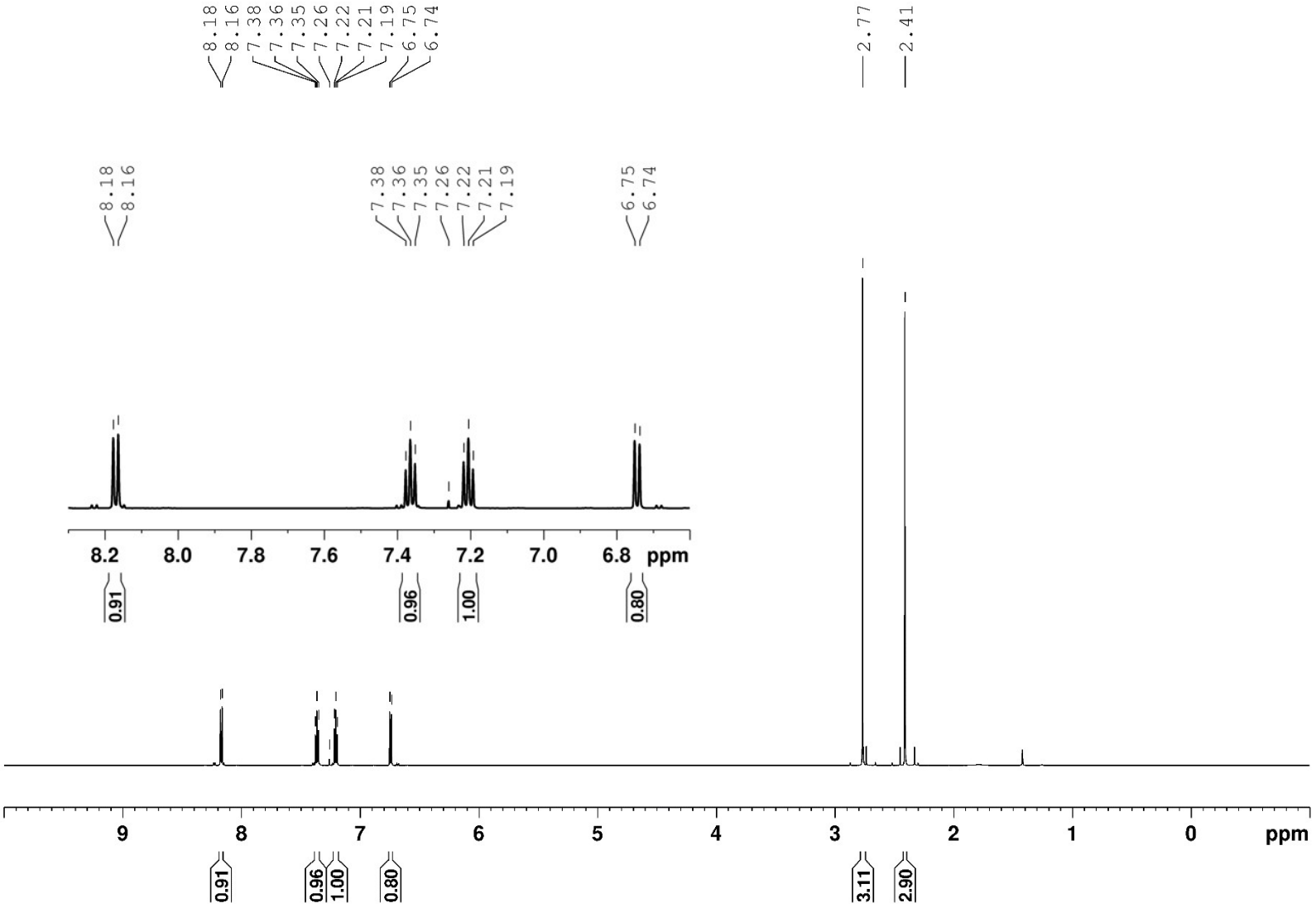


Fig. 21 ^{1}H (600 MHz) NMR spectra of **4a** in $CDCl_3$

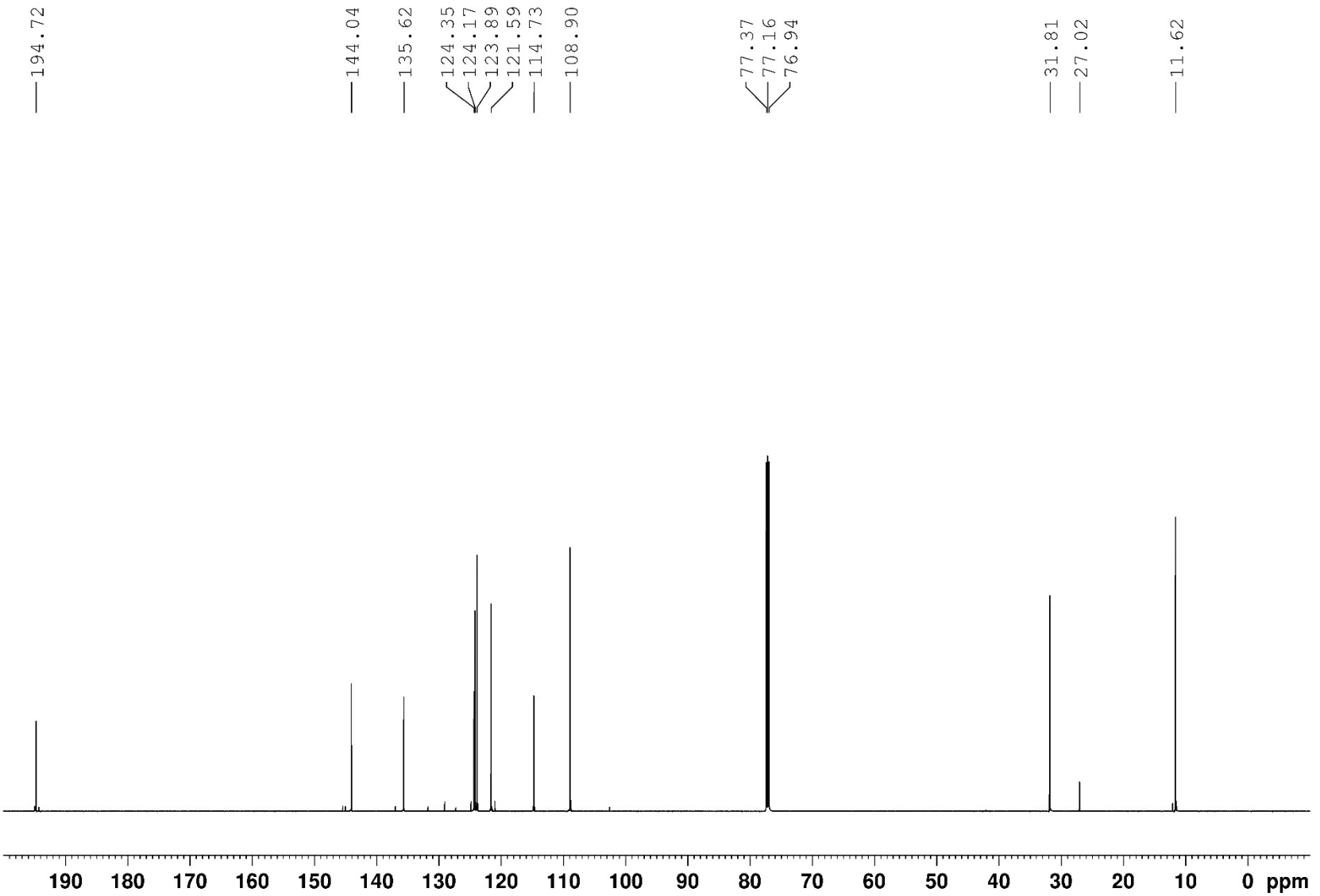


Fig. 22 ^{13}C (150 MHz) NMR spectra of **4a** in $CDCl_3$

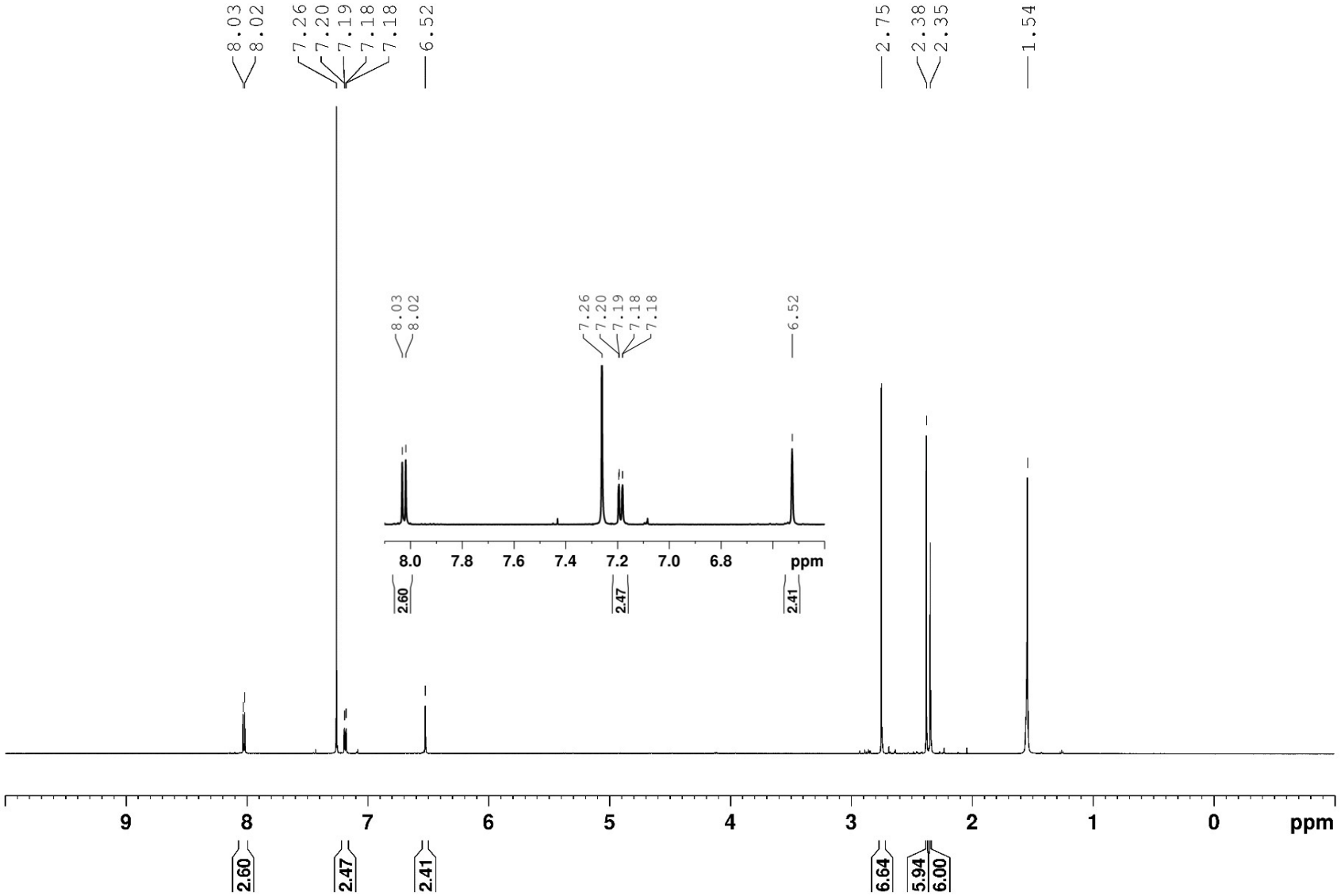


Fig. 23 ^{1}H (600 MHz) NMR spectra of **4b** in $CDCl_3$

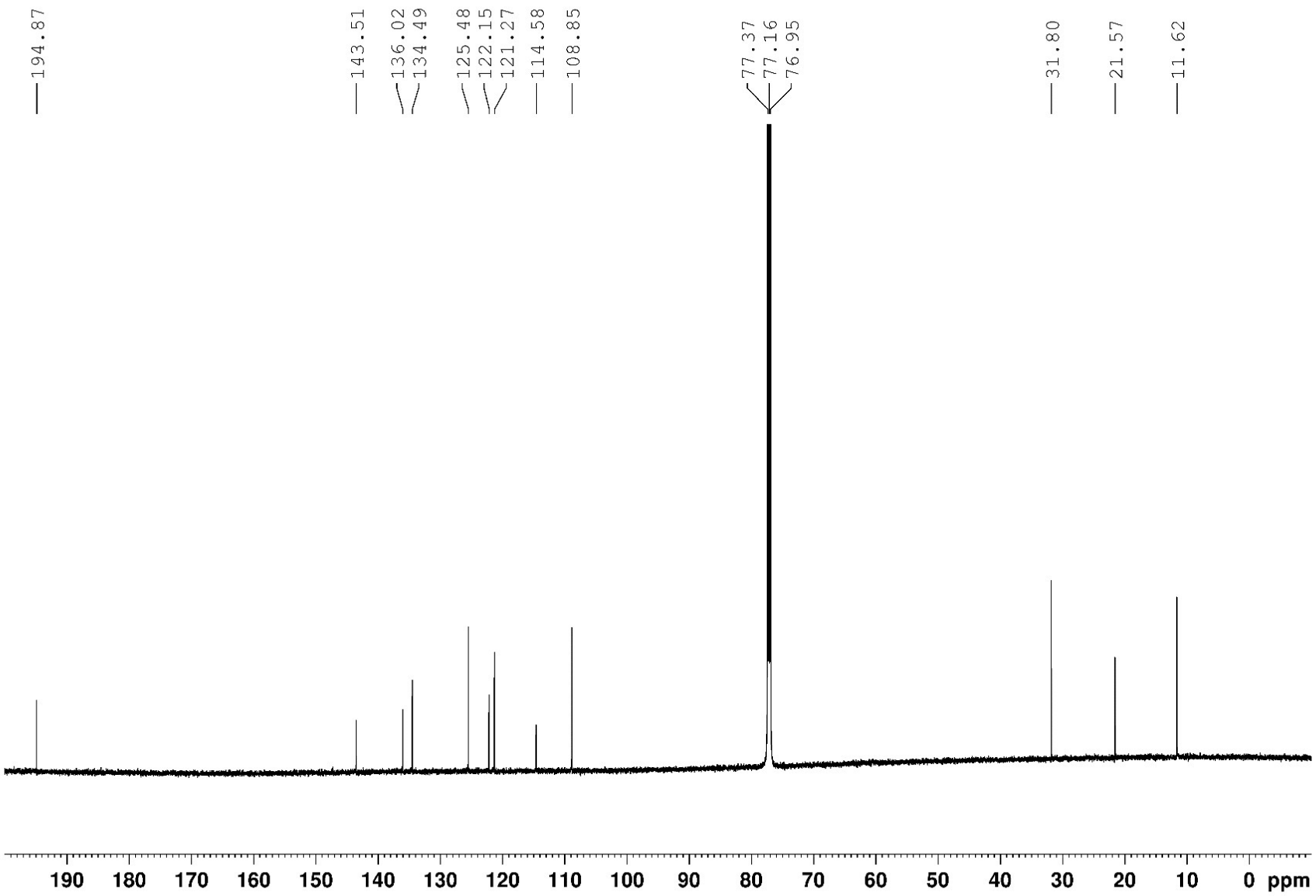


Fig. 24 ^{13}C (150 MHz) NMR spectra of **4b** in $CDCl_3$

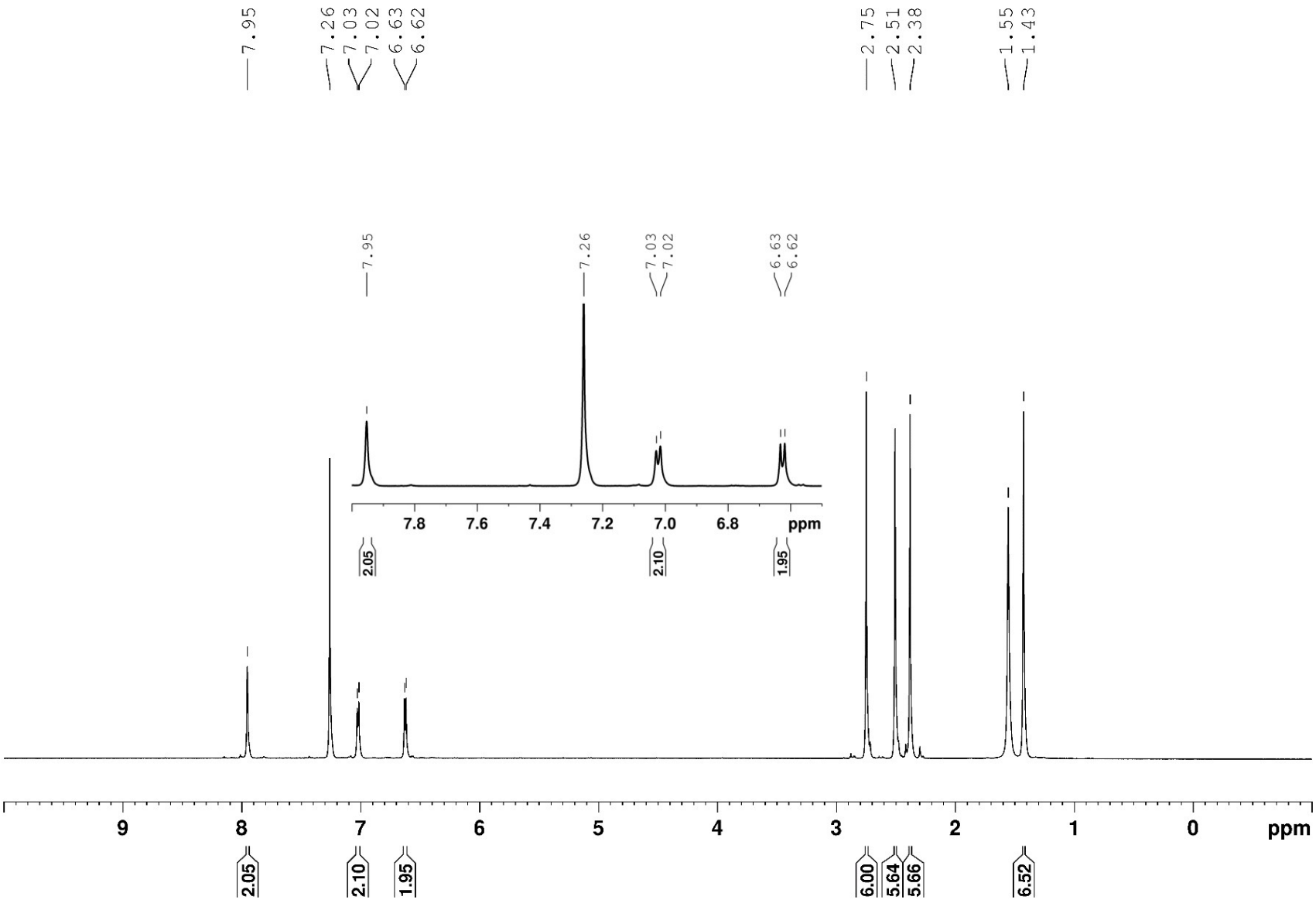


Fig. 25 ^{1}H (600 MHz) NMR spectra of **4c** in $CDCl_3$

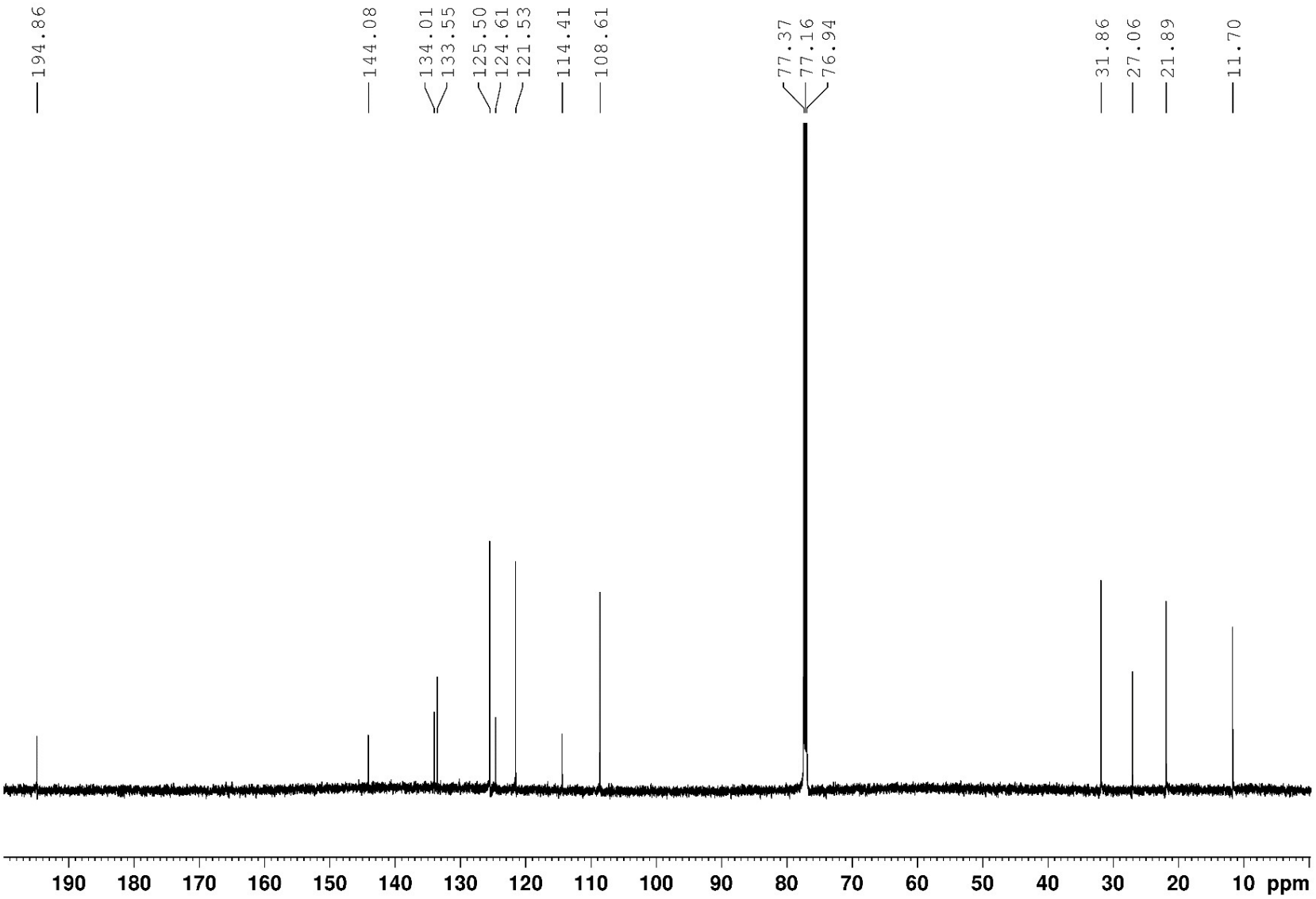


Fig. 26 ^{13}C (150 MHz) NMR spectra of **4c** in $CDCl_3$

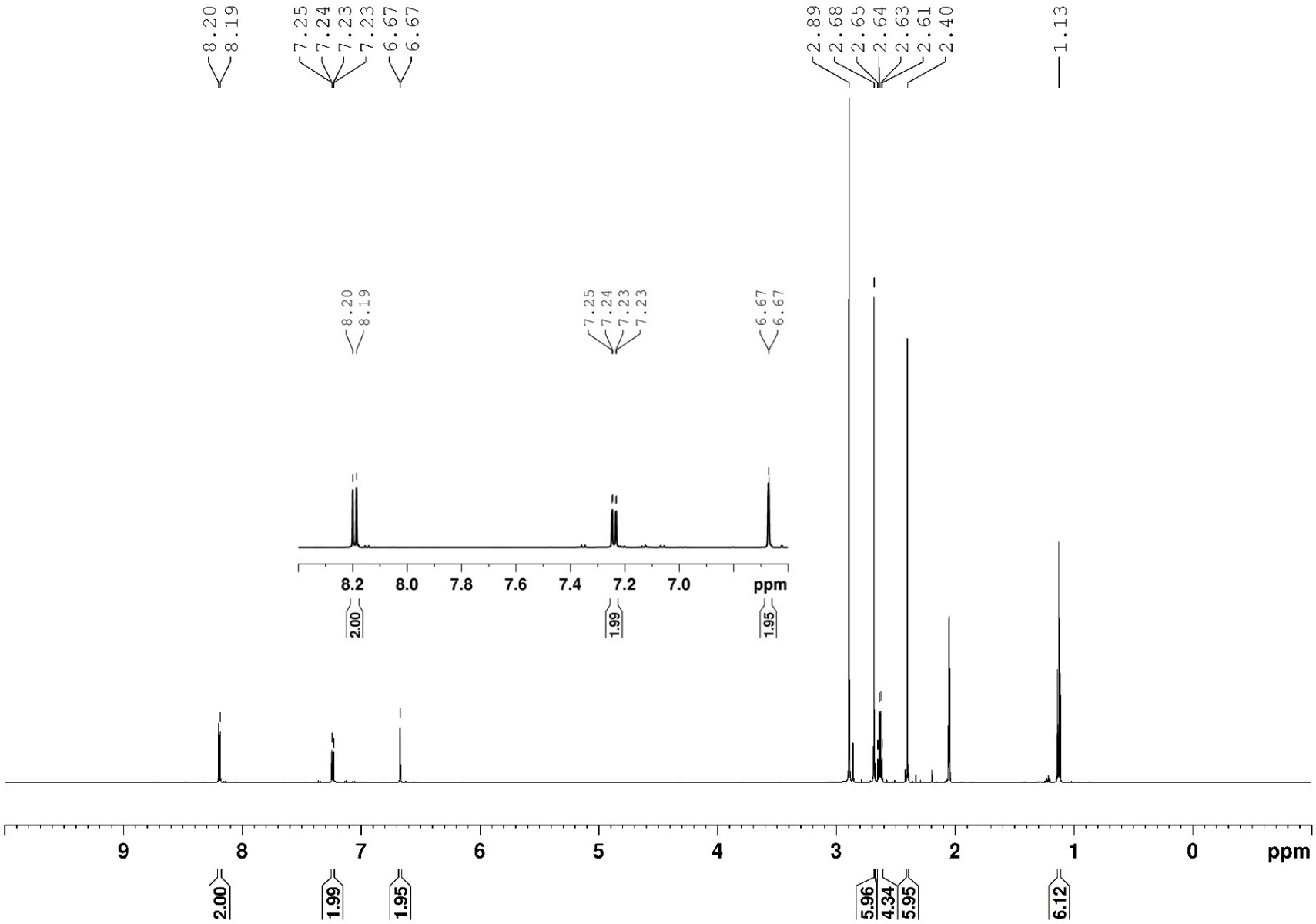


Fig. 27 ^{1}H (600 MHz) NMR spectra of **4e** in $(CD_3)_2CO$

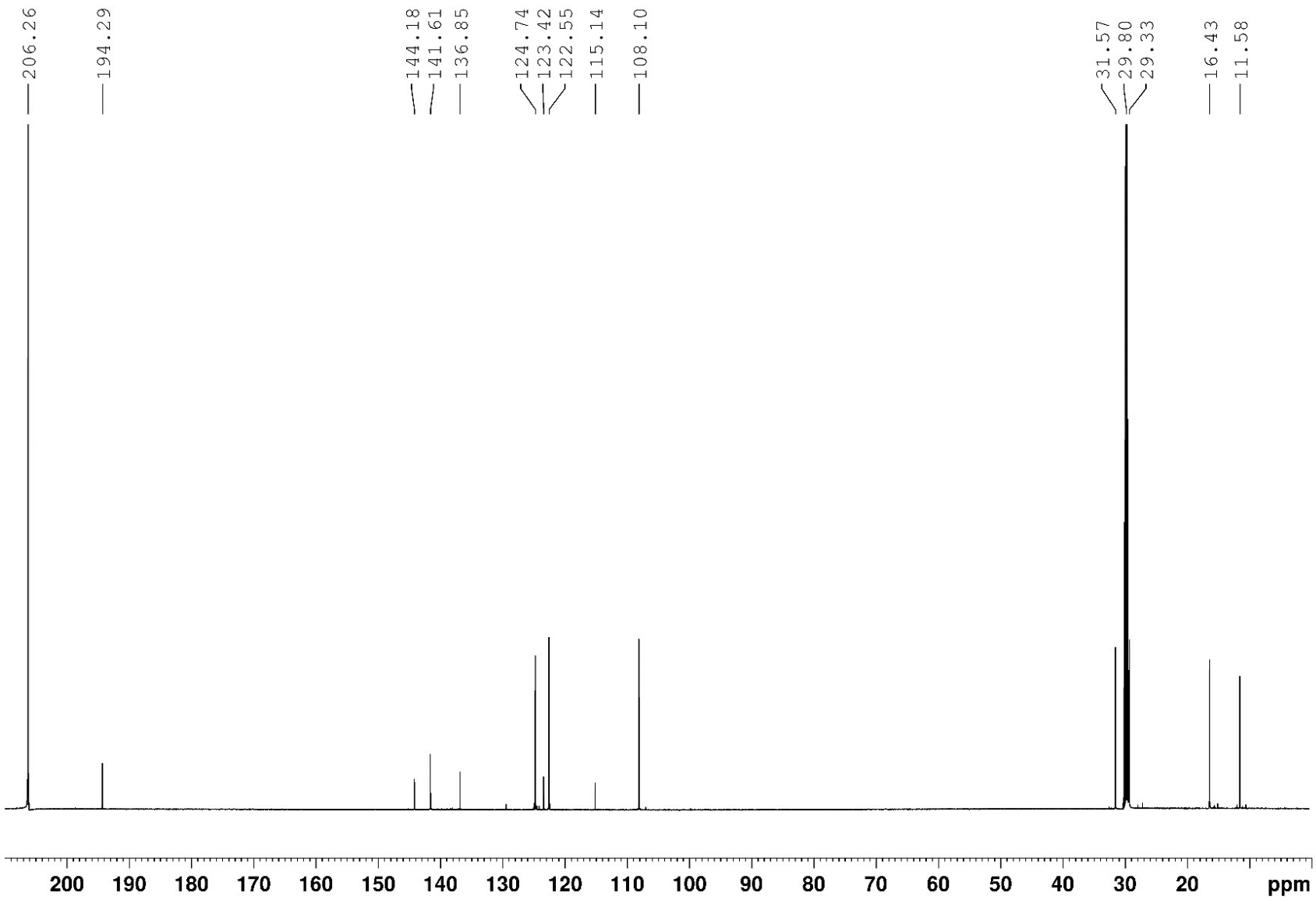


Fig. 28 ^{13}C (150 MHz) NMR spectra of **4e** in $(CD_3)_2CO$

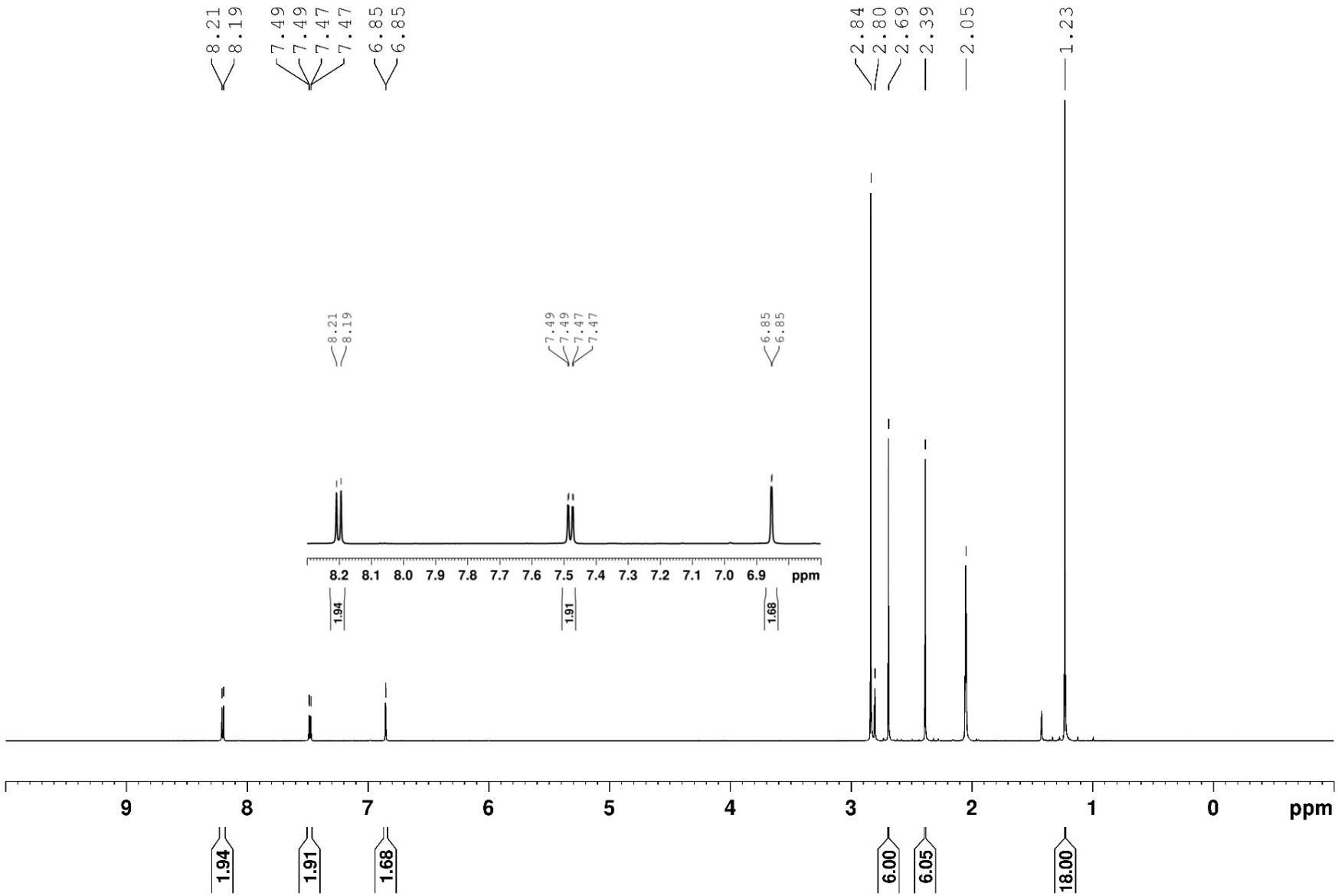


Fig. 29 ^{1}H (600 MHz) NMR spectra of **4f** in $(CD_3)_2CO$

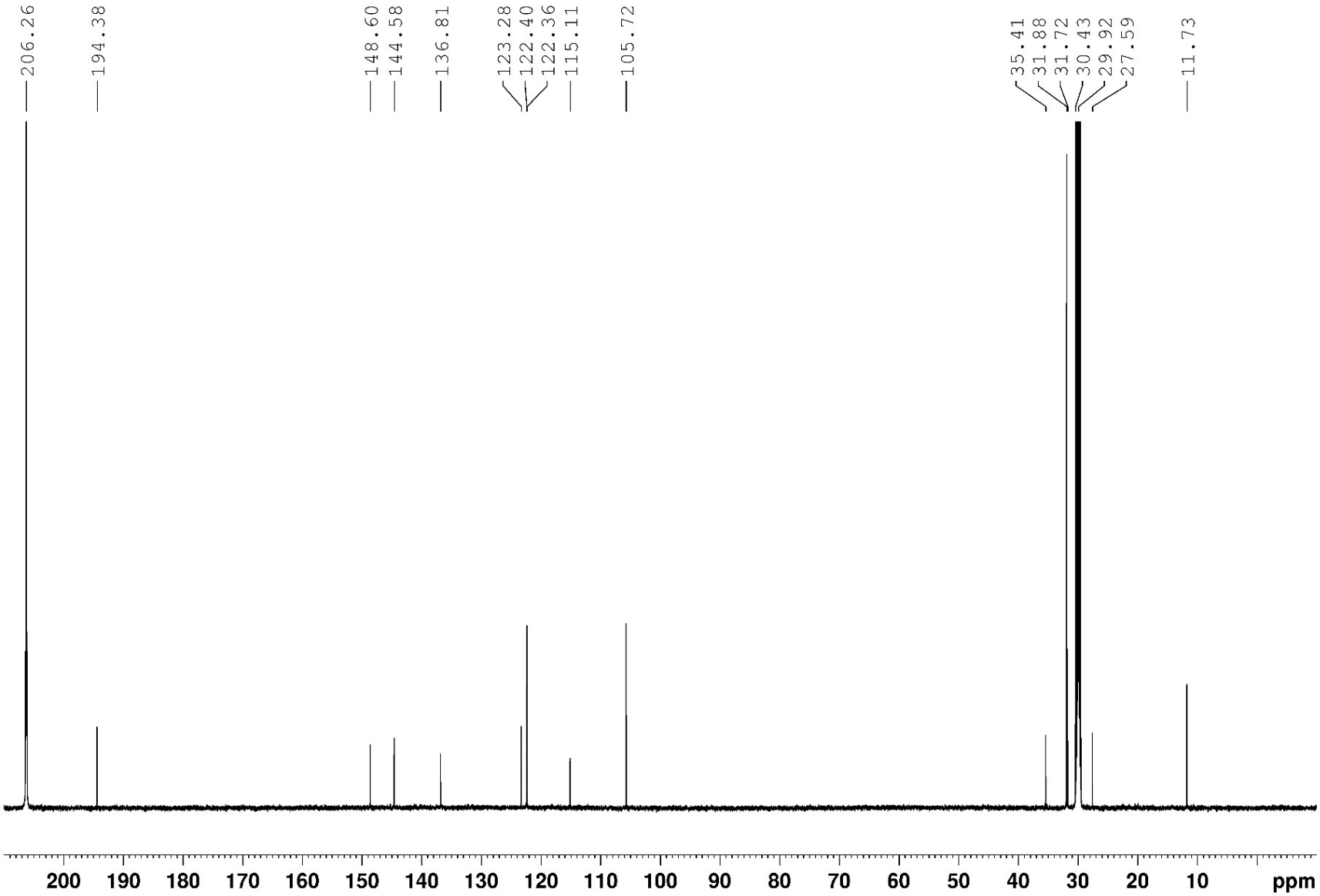


Fig. 30 ^{13}C (150MHz) NMR spectra of **4f** in $(CD_3)_2CO$

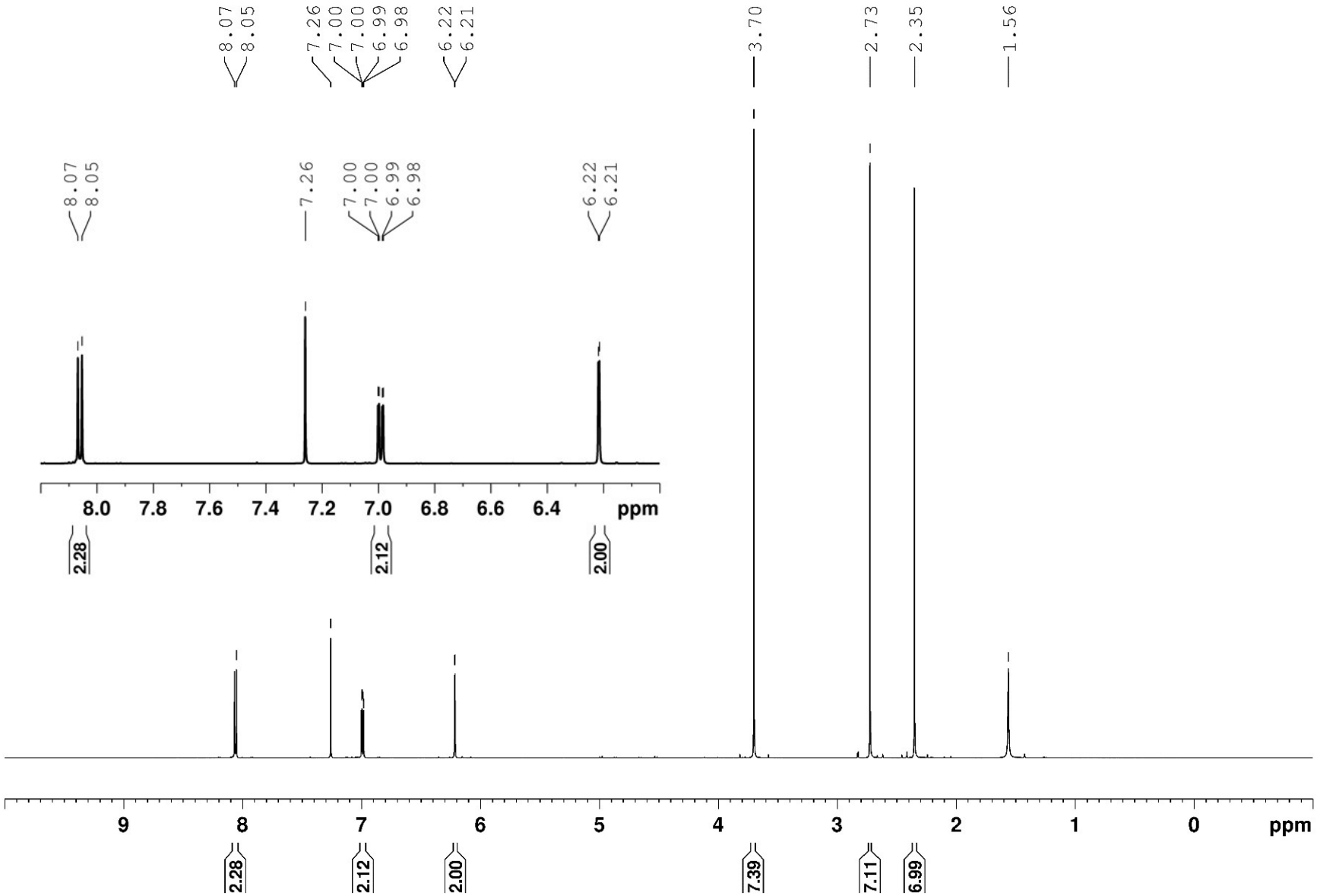


Fig. 31 ^{1}H (600 MHz) NMR spectra of **4g** in $CDCl_3$

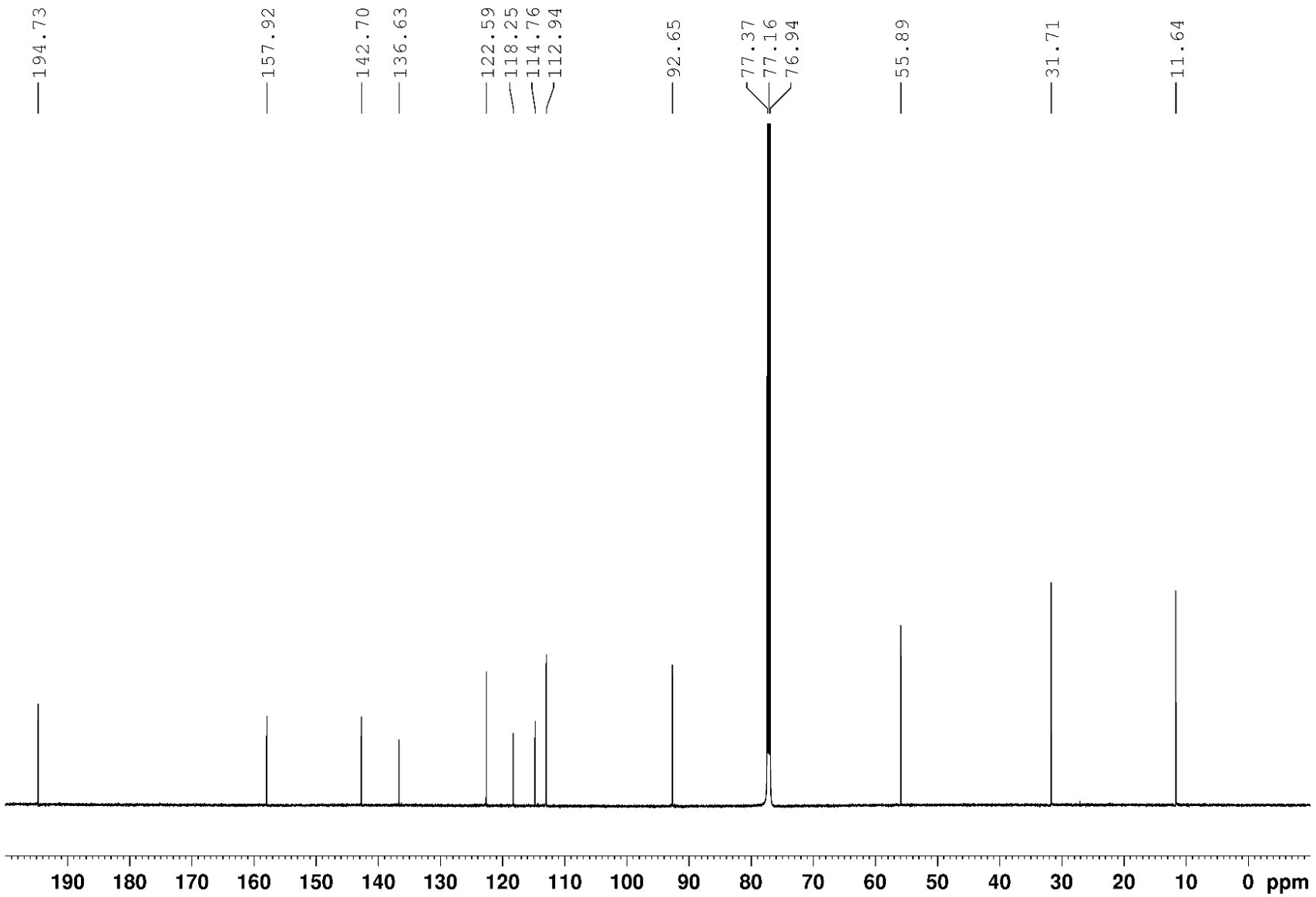


Fig. 32 ^{13}C (150MHz) NMR spectra of **4g** in $CDCl_3$

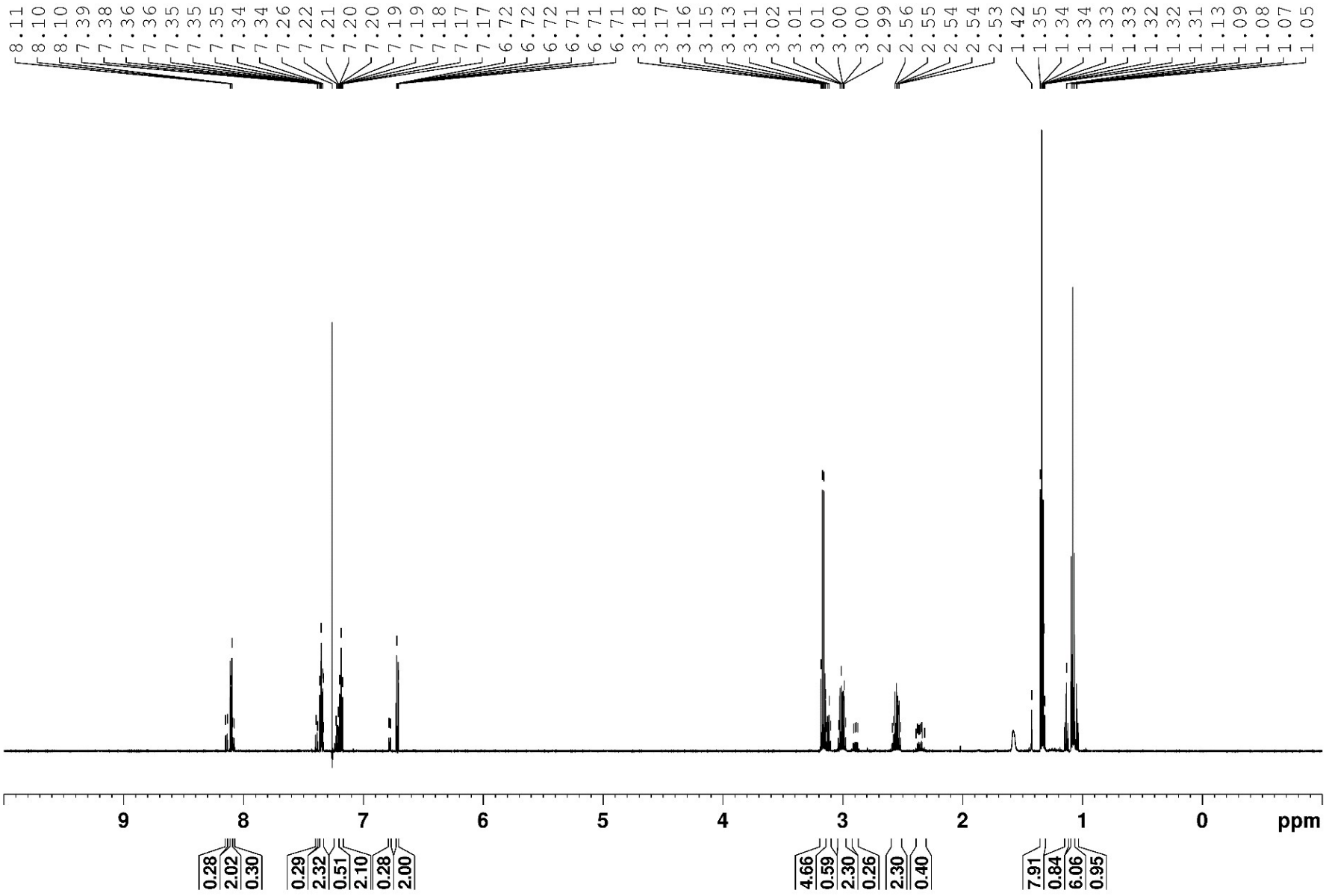


Fig. 33 ^{1}H (600 MHz) NMR spectra of **4h** and **8h** in $CDCl_3$

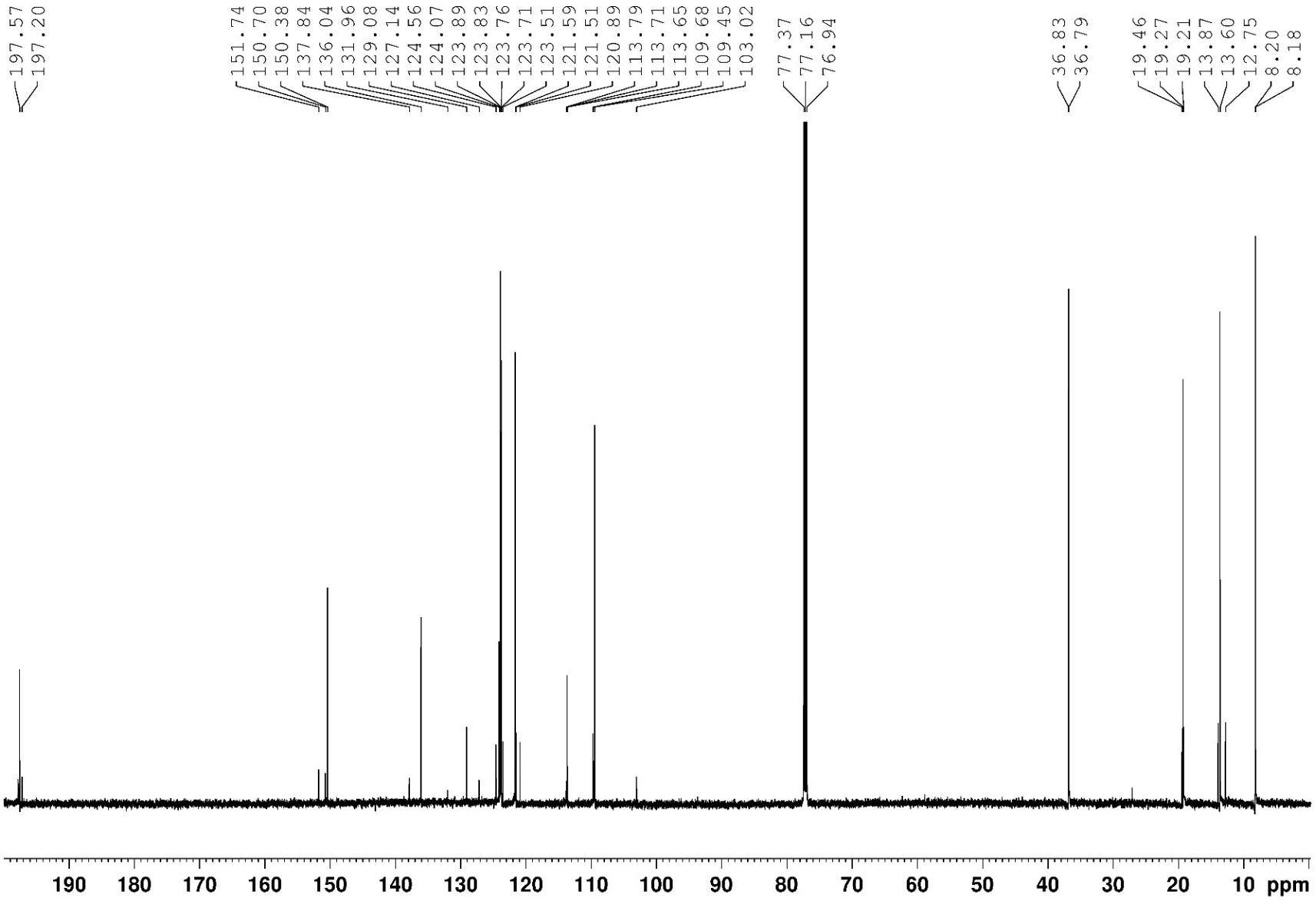


Fig. 34 ^{13}C (150 MHz) NMR spectra of **4h** and **8h** in $CDCl_3$

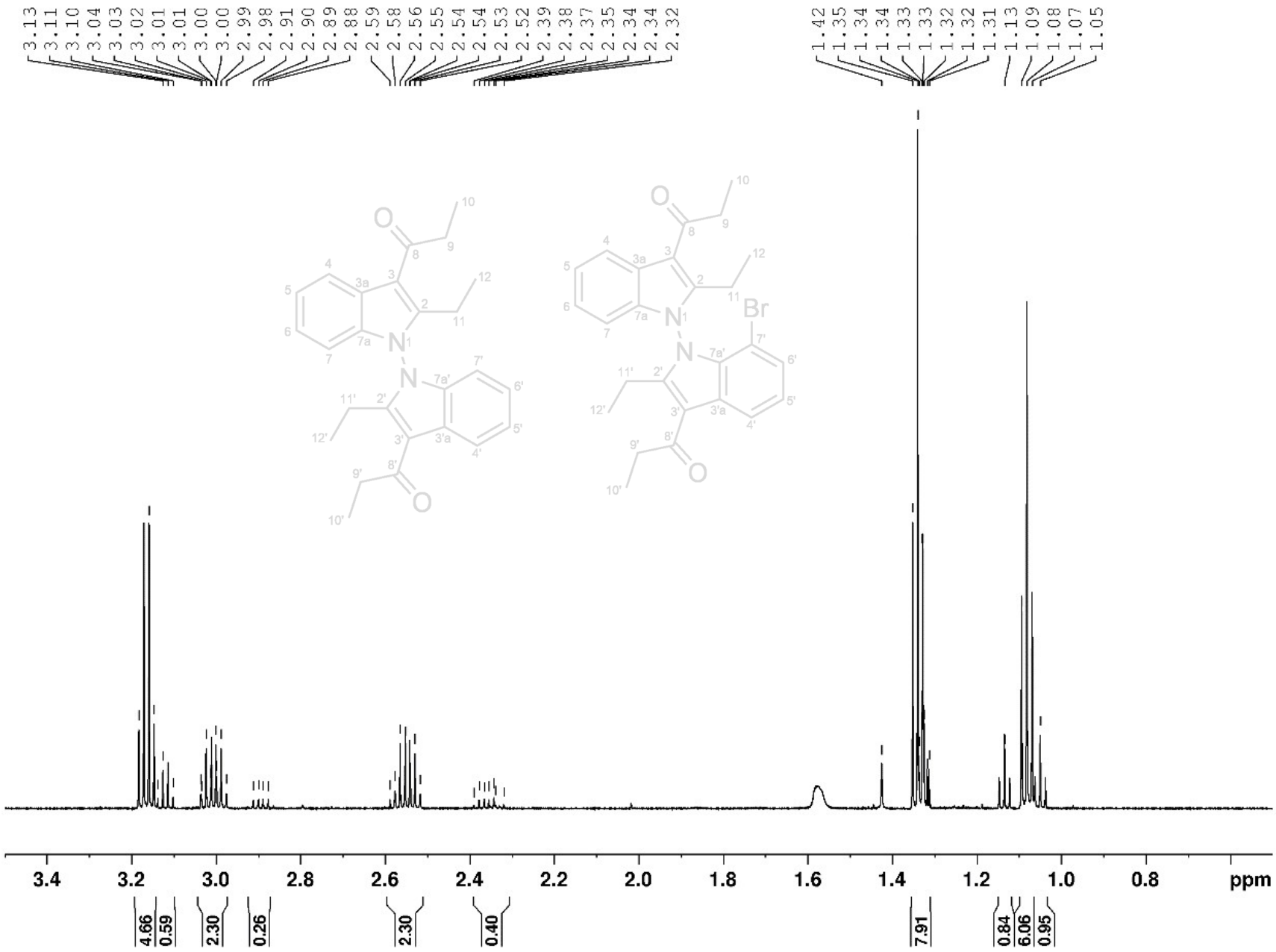


Fig. 35 Excerpts from the ^{1}H (600 MHz) NMR spectra of **4h** and **8h** in $CDCl_3$

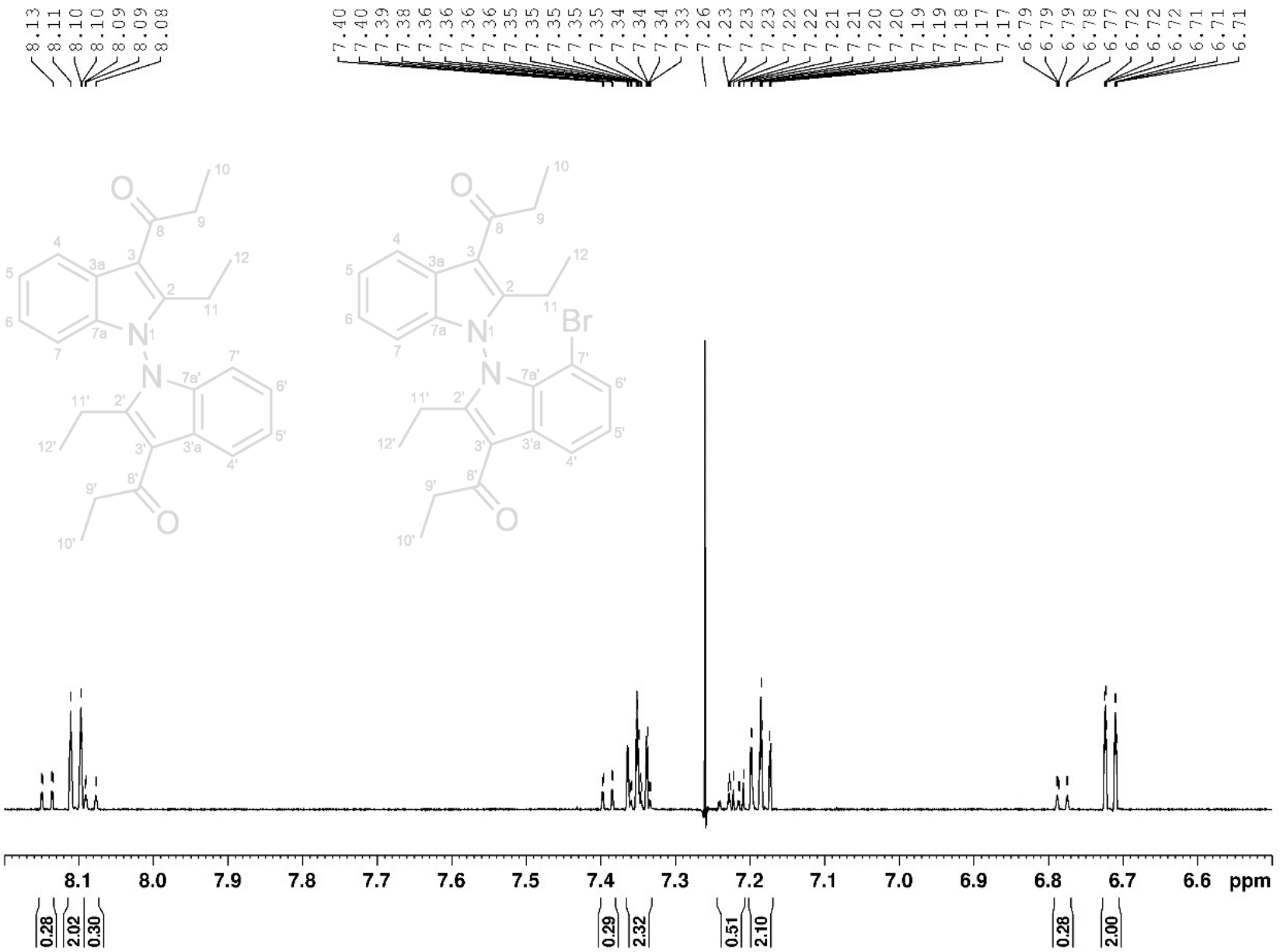


Fig. 36 Excerpts from the ^{1}H (600 MHz) NMR spectra of **4h** and **8h** in $CDCl_3$

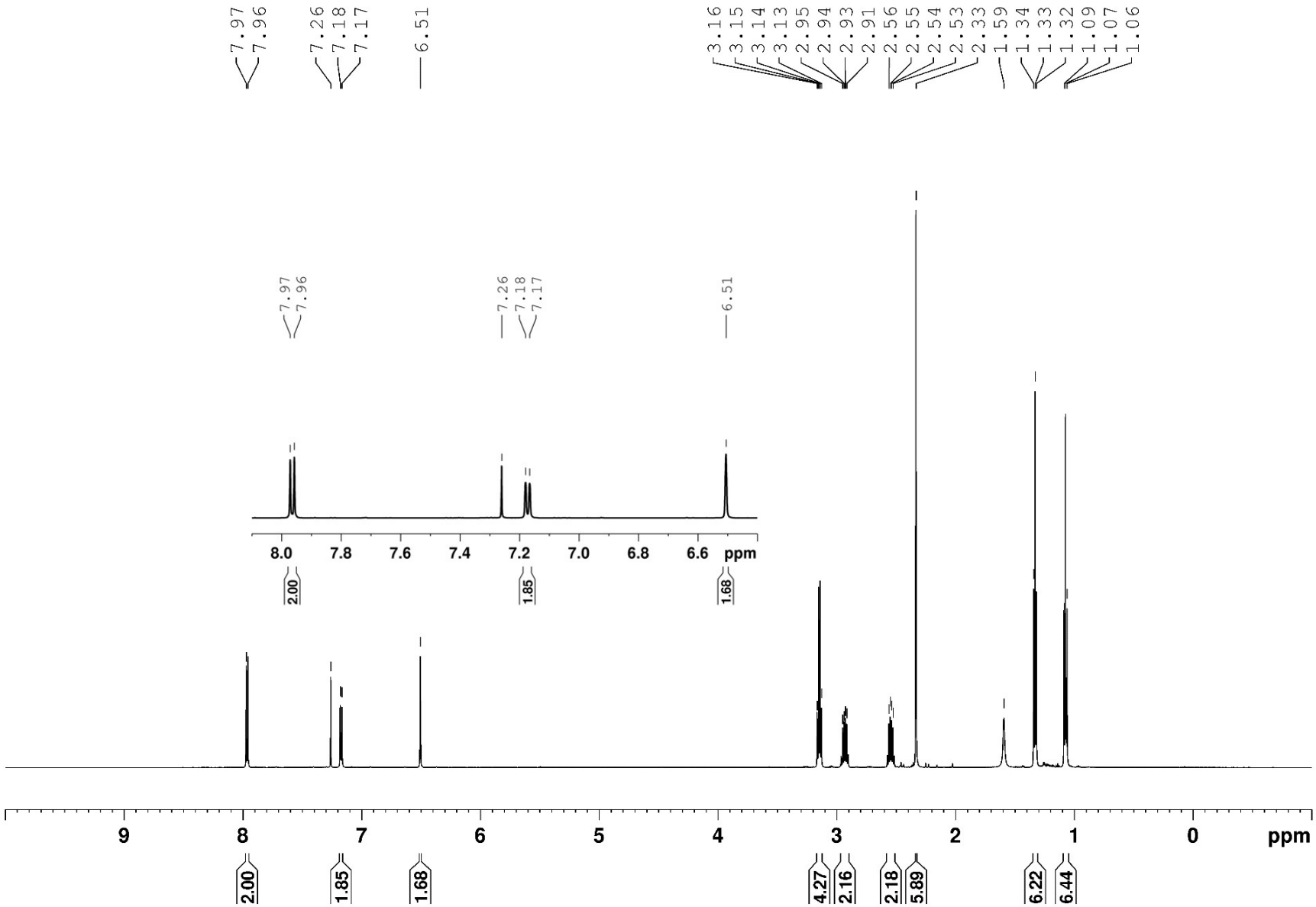


Fig. 37 ^{1}H (600 MHz) NMR spectra of **4i** in $CDCl_3$

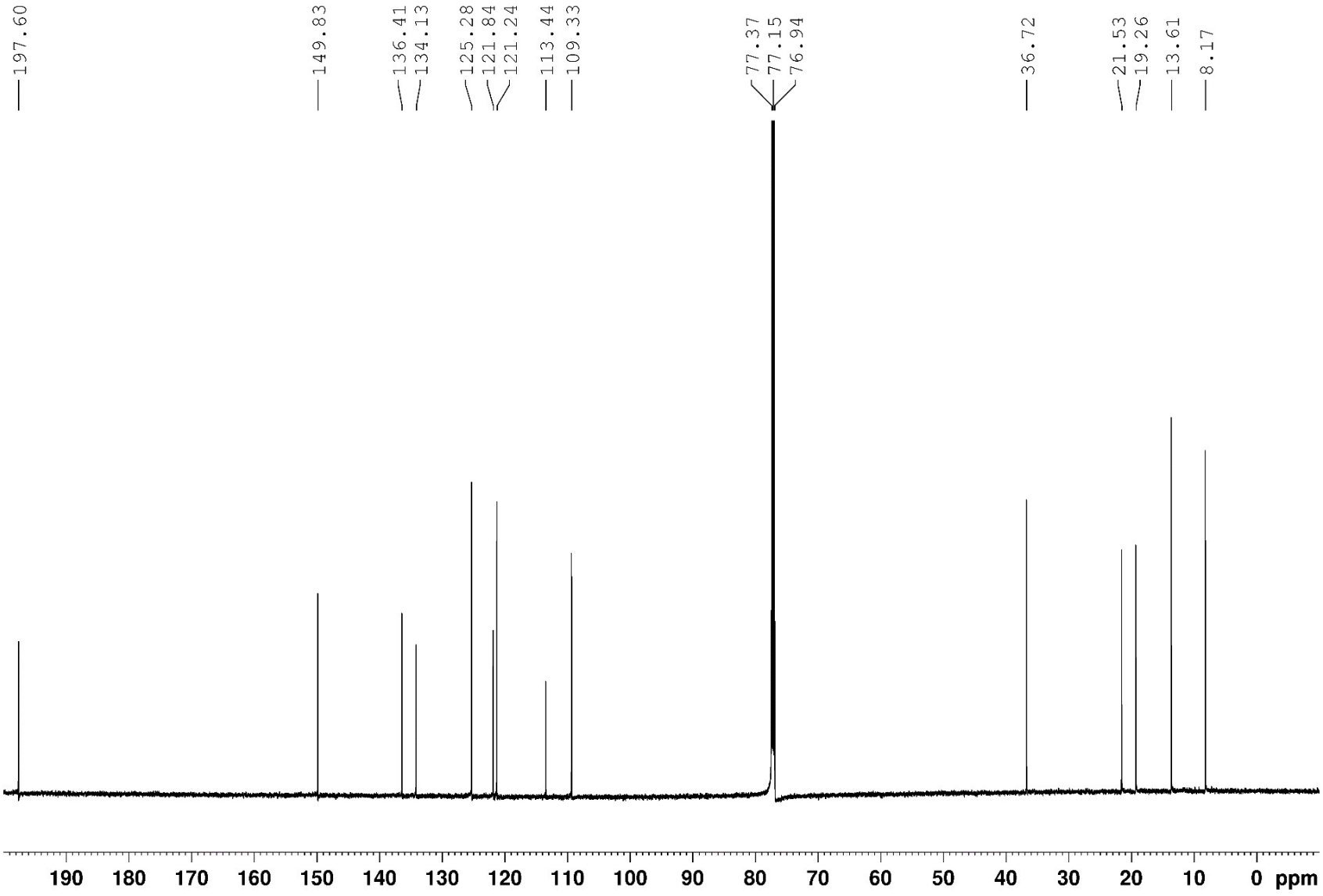


Fig. 38 ^{13}C (150 MHz) NMR spectra of **4i** in $CDCl_3$

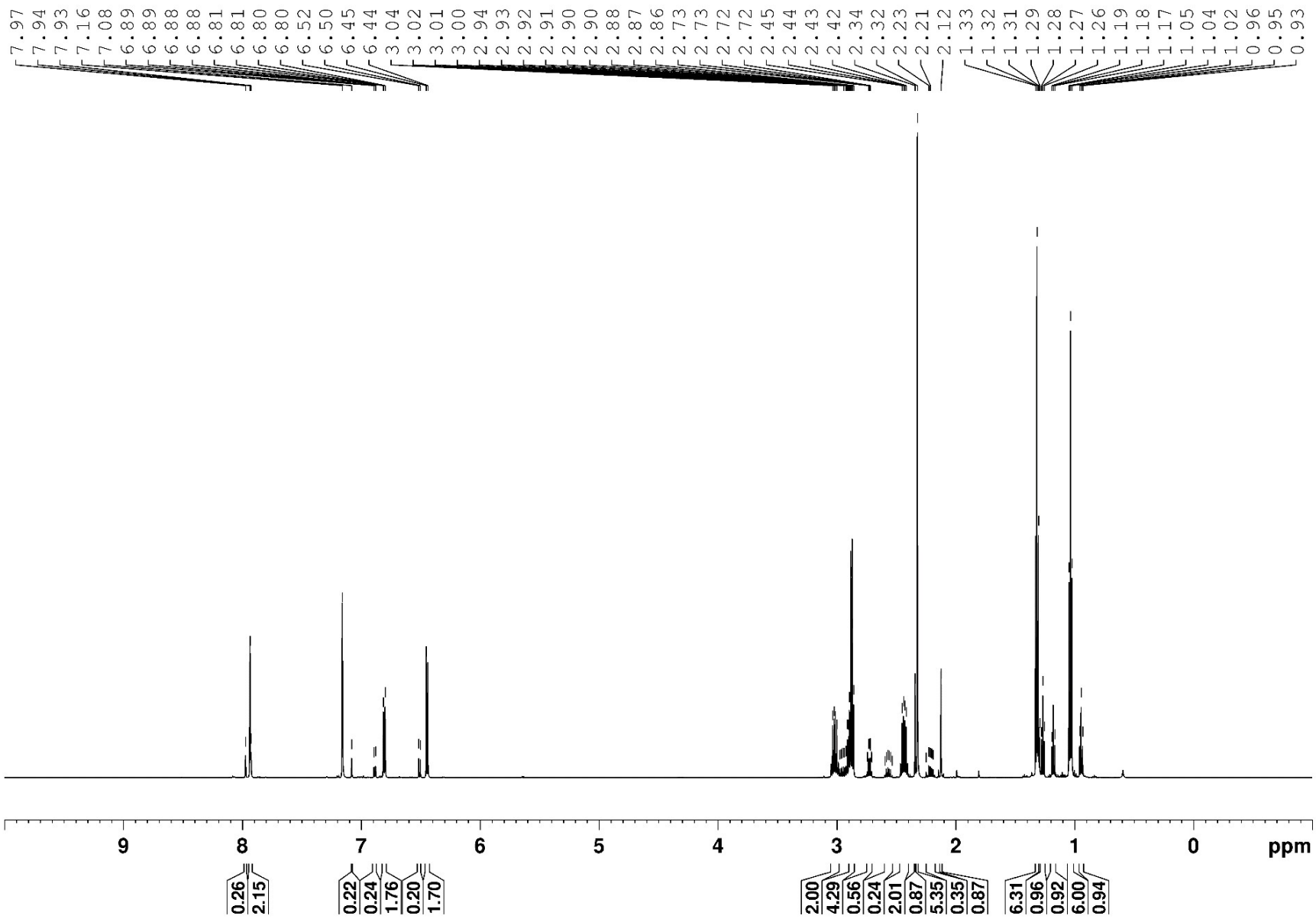


Fig. 39 ^{1}H (600 MHz) NMR spectra of **4j** and **8j** in C_6D_6

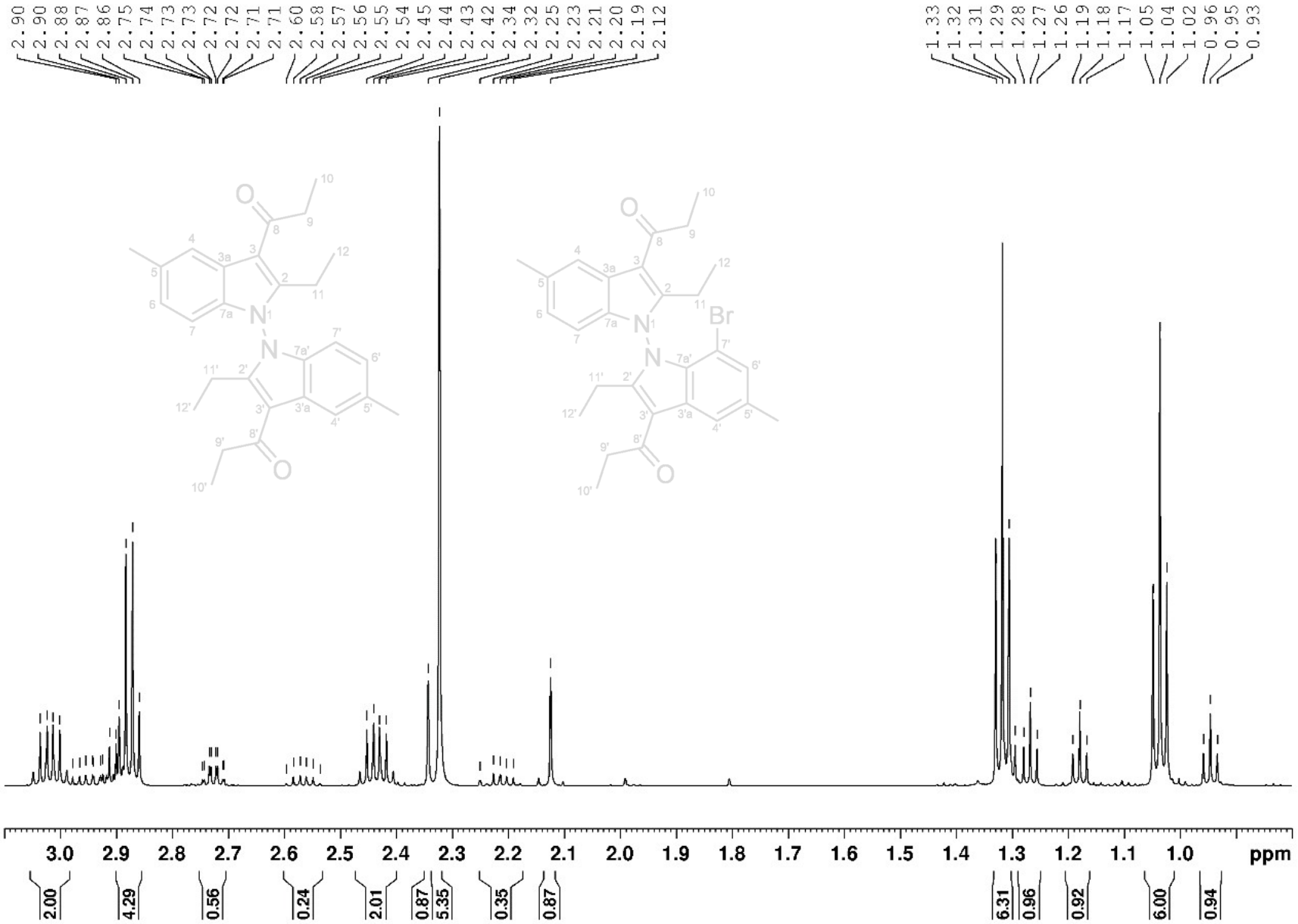


Fig. 40 Excerpts from the ^{1}H (600 MHz) NMR spectra of **4j** and **8j** in C_6D_6

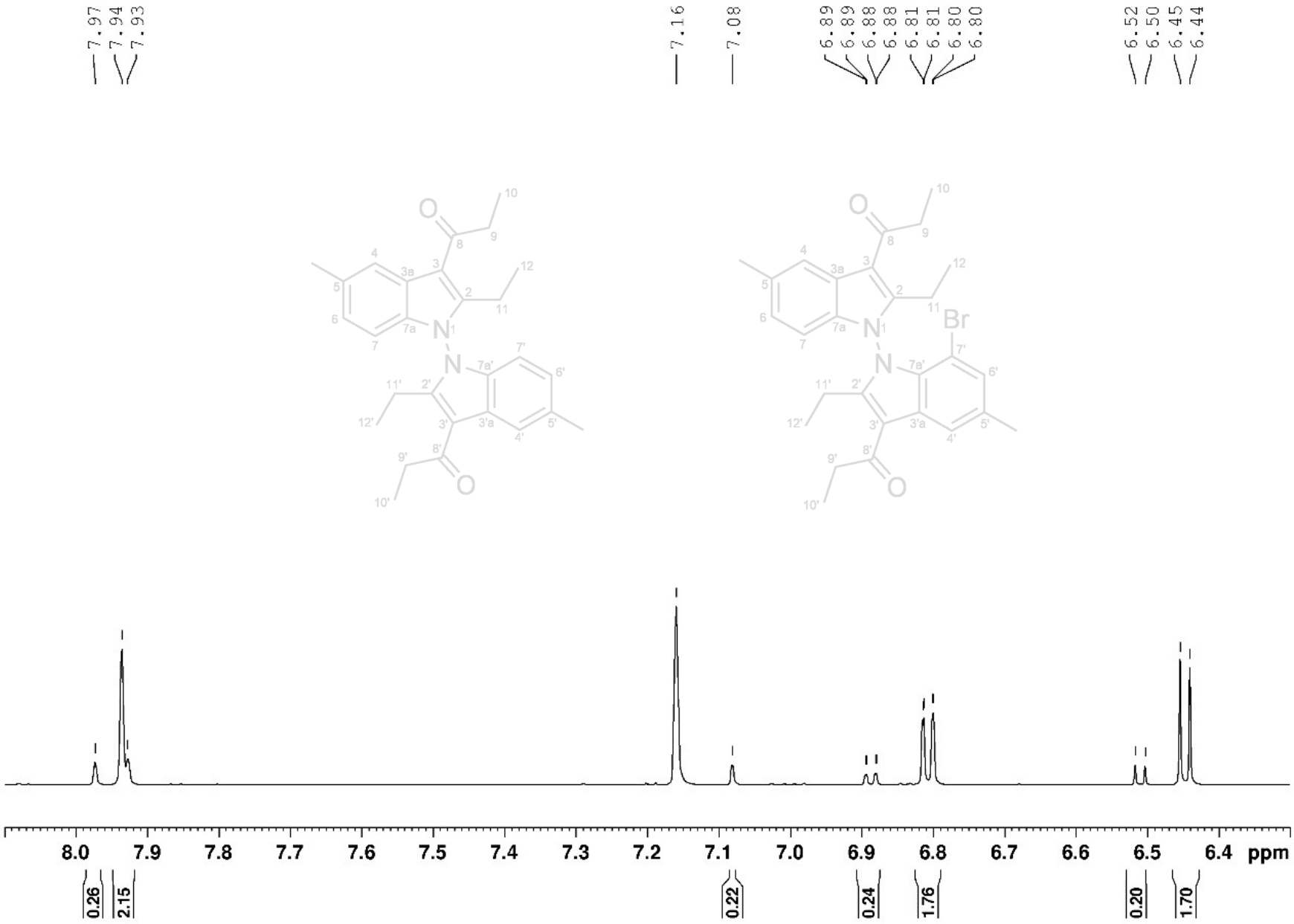


Fig. 41 Excerpts from the ^{1}H (600 MHz) NMR spectra of **4j** and **8j** in C_6D_6

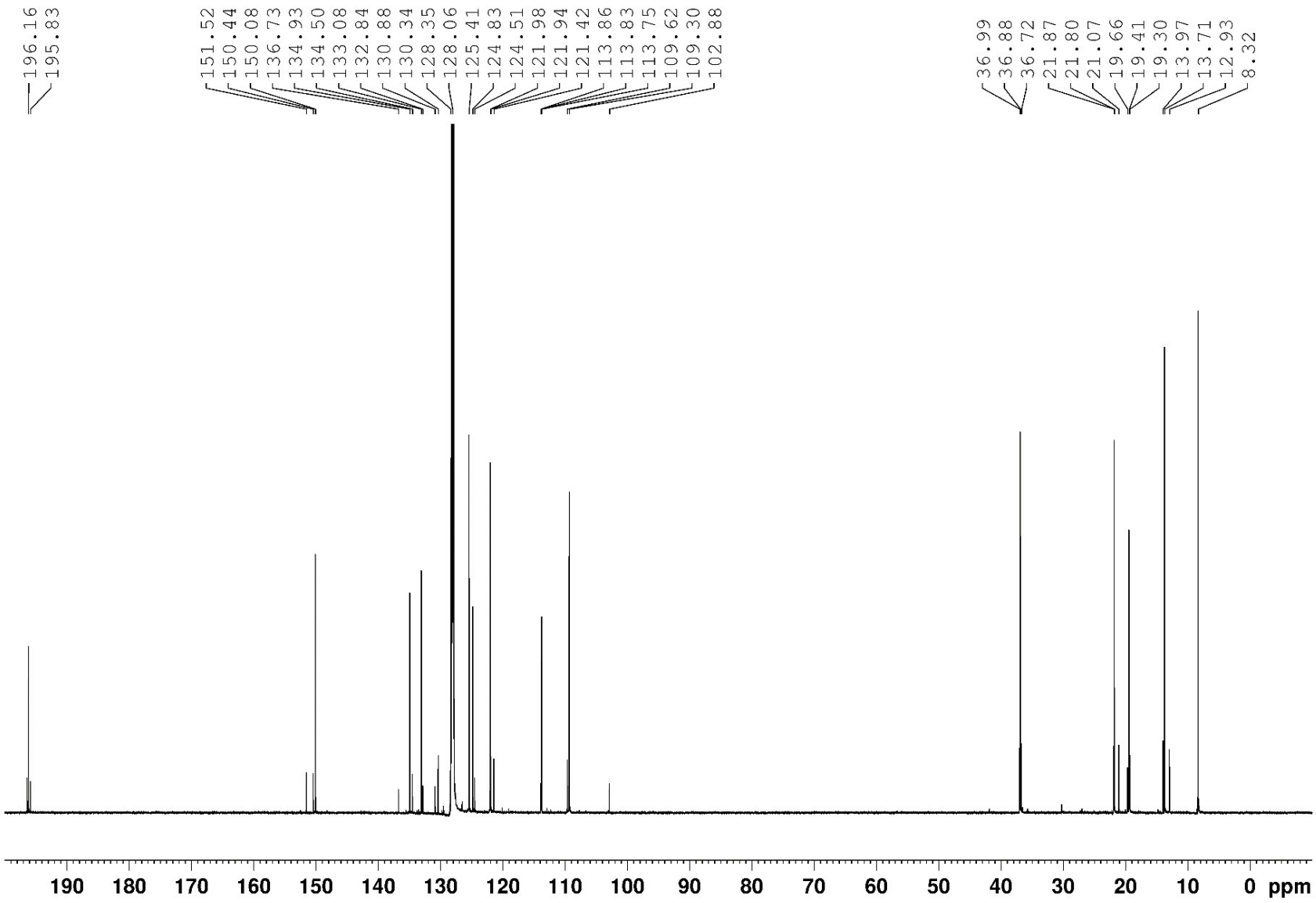


Fig. 42 ^{13}C (150 MHz) NMR spectra of **4j** and **8j** in C_6D_6

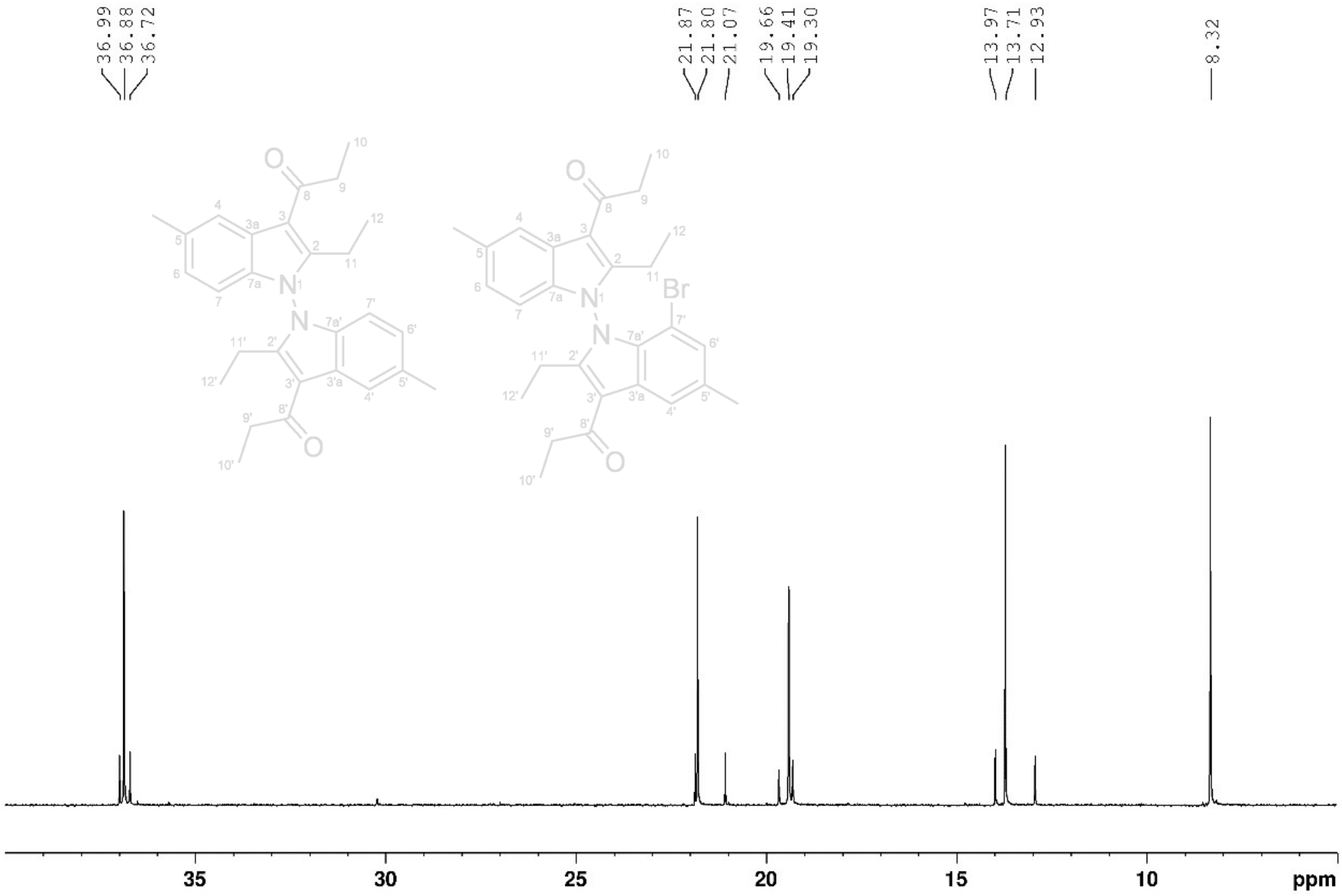


Fig. 43 Excerpts from the ^{13}C (600 MHz) NMR spectra of **4j** and **8j** in C_6D_6

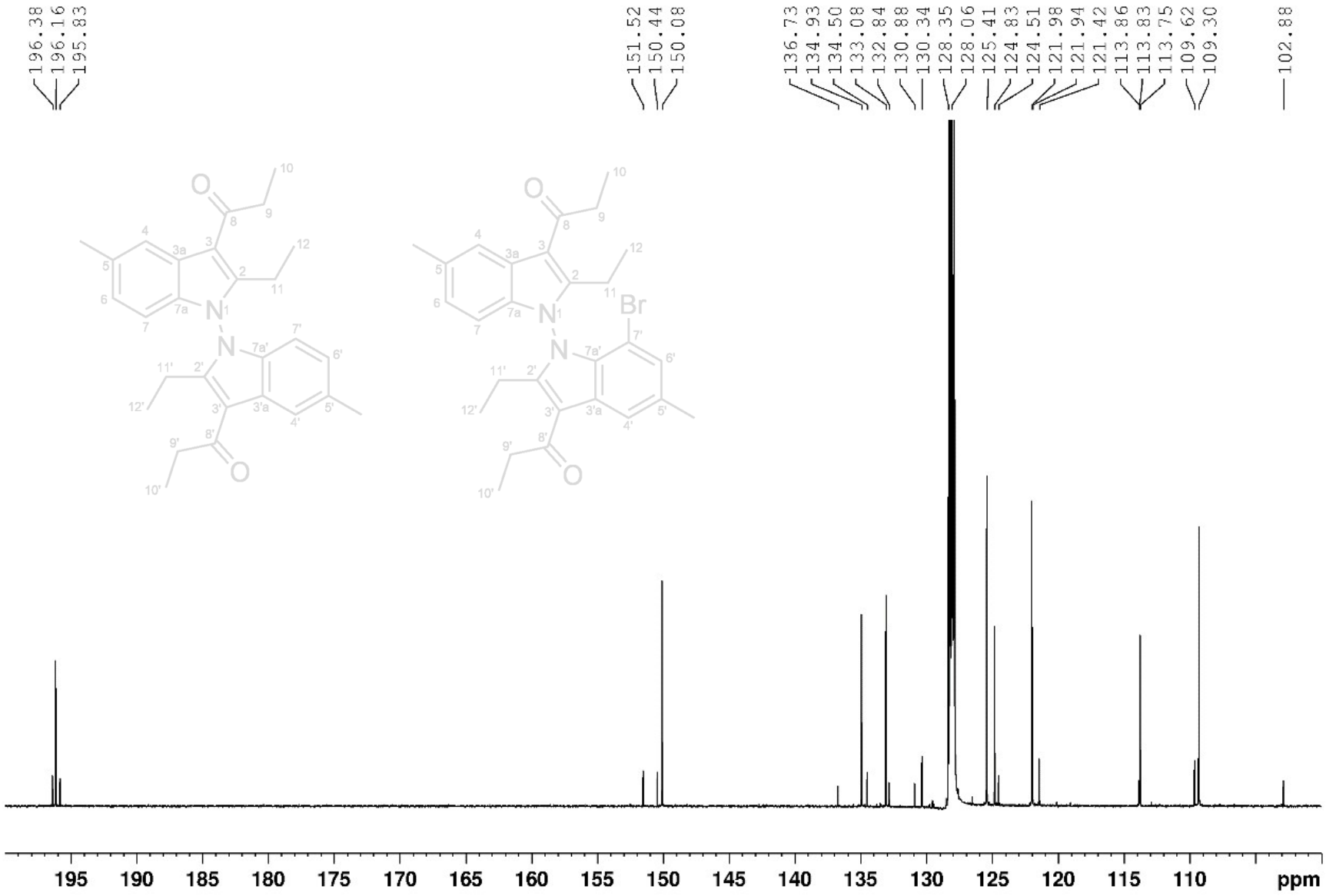


Fig. 44 Excerpts from the ^{13}C (600 MHz) NMR spectra of **4j** and **8j** in C_6D_6

NMR spectra of the indoles 3k, 3l and 7d

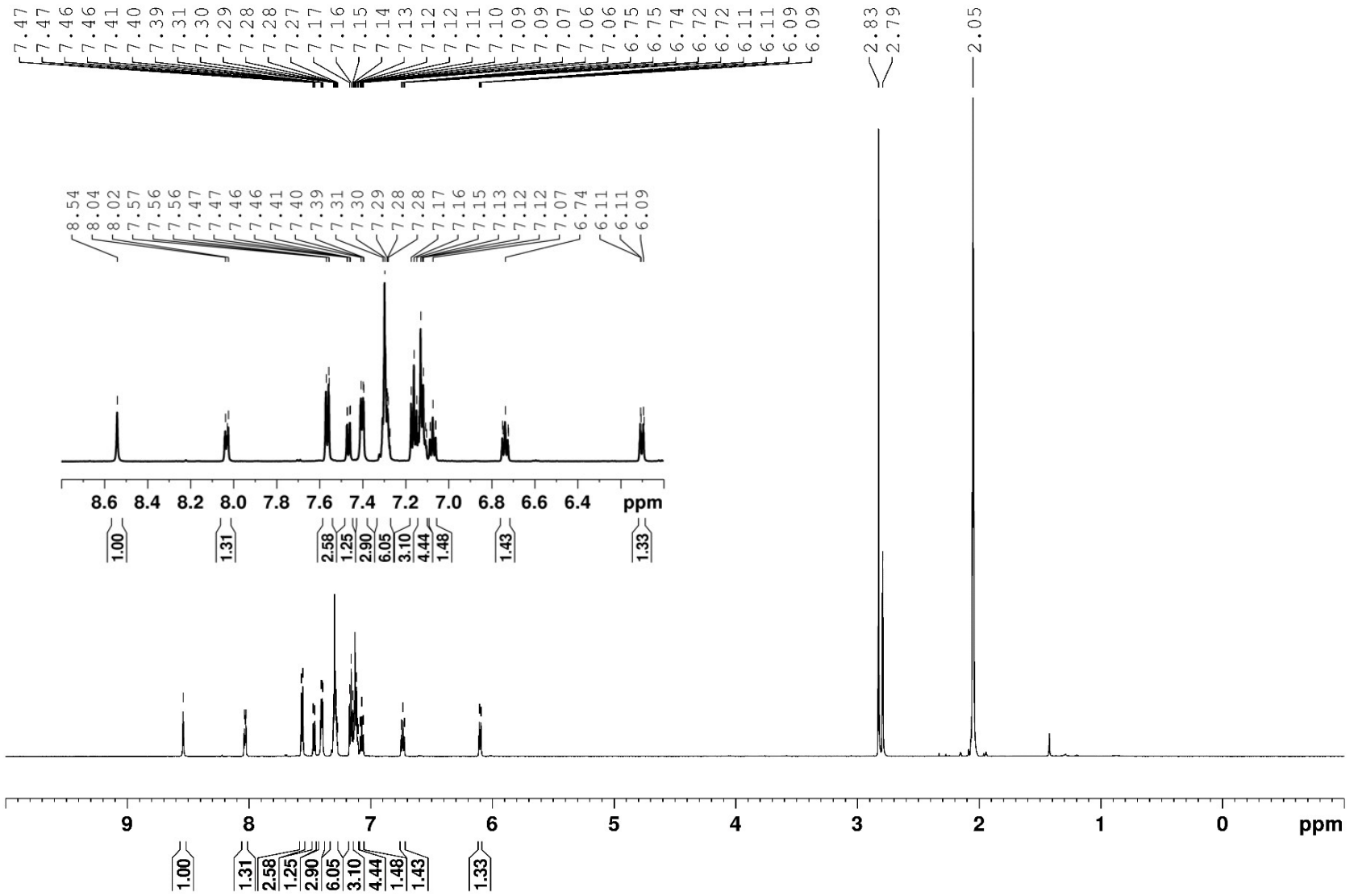


Fig. 45 ^{1}H (600 MHz) NMR spectra of **3k** in $(CD_3)_2CO$

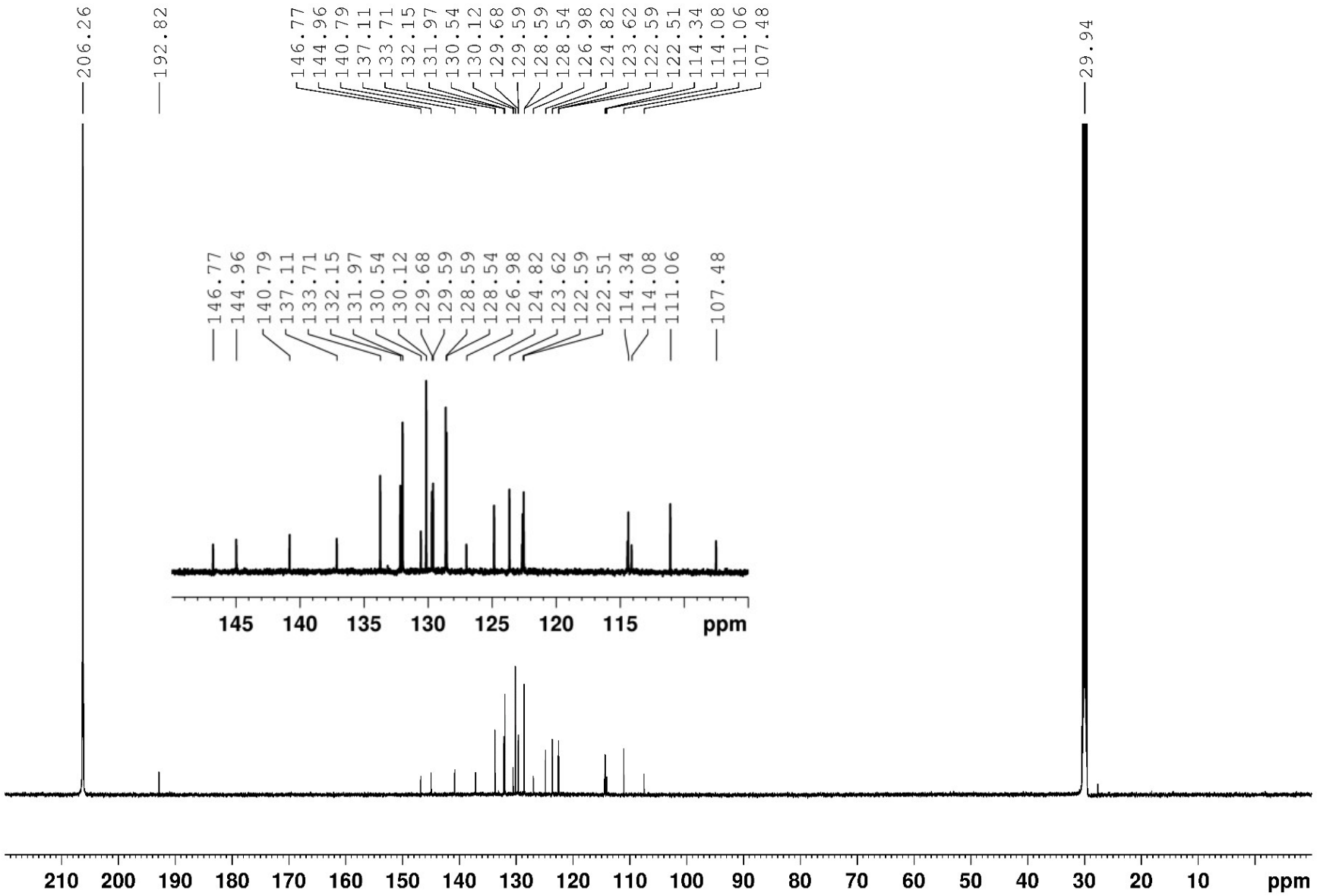


Fig. 46 ^{13}C (150MHz) NMR spectra of **3k** in $(CD_3)_2CO$

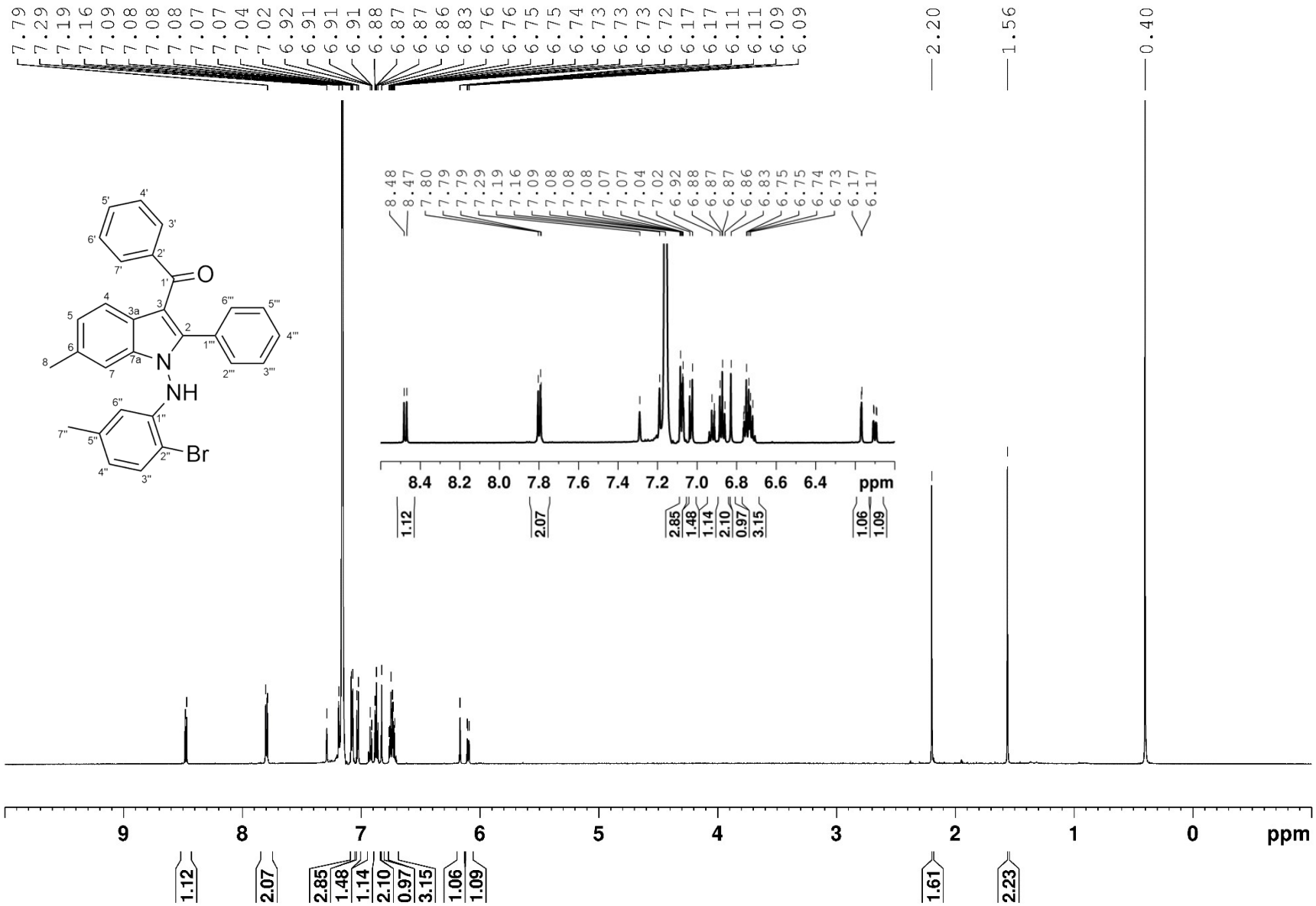


Fig. 47 ^{1}H (600 MHz) NMR spectra of **3l** in C_6D_6

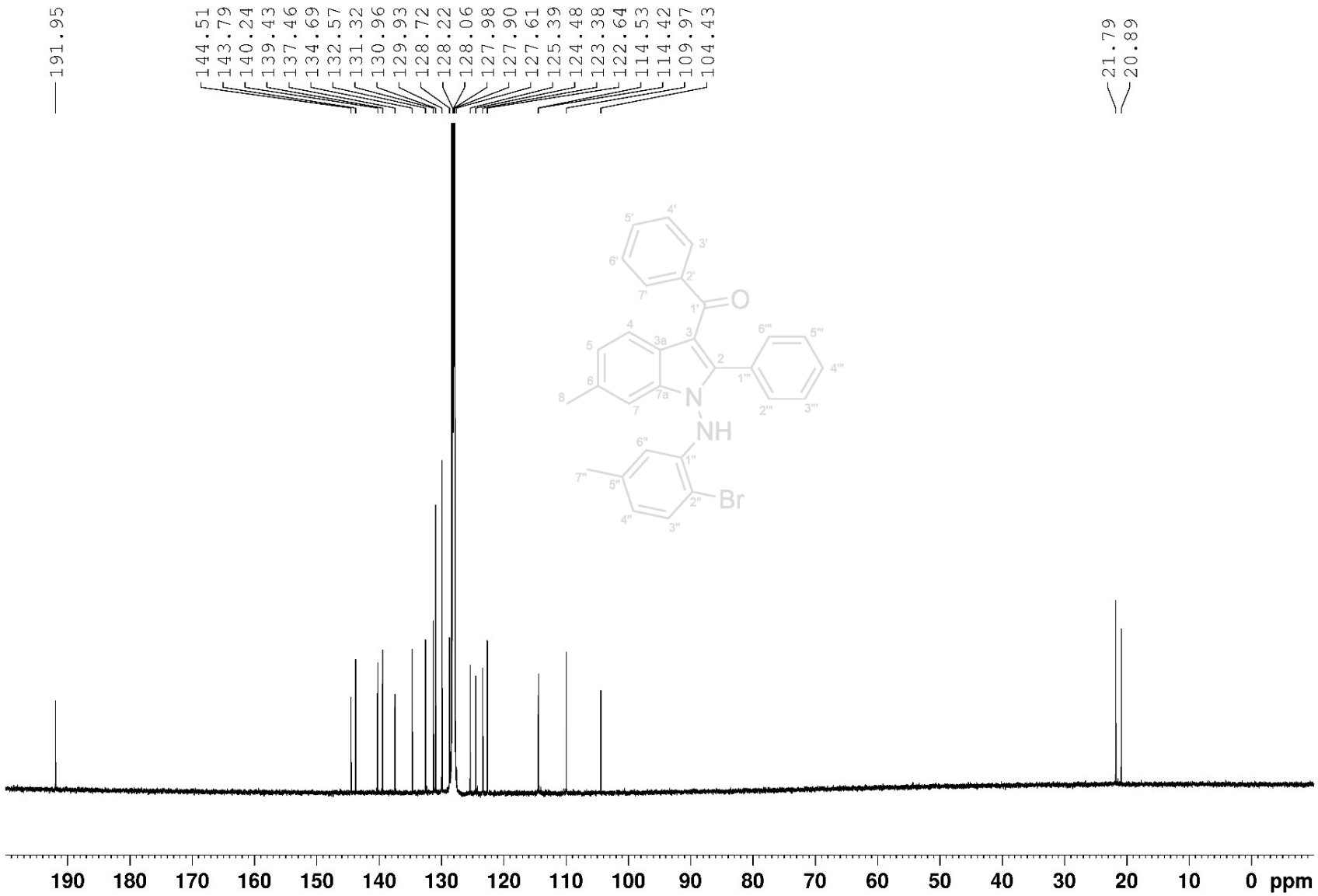


Fig. 48 ^{13}C (150MHz) NMR spectra of **3l** in C_6D_6

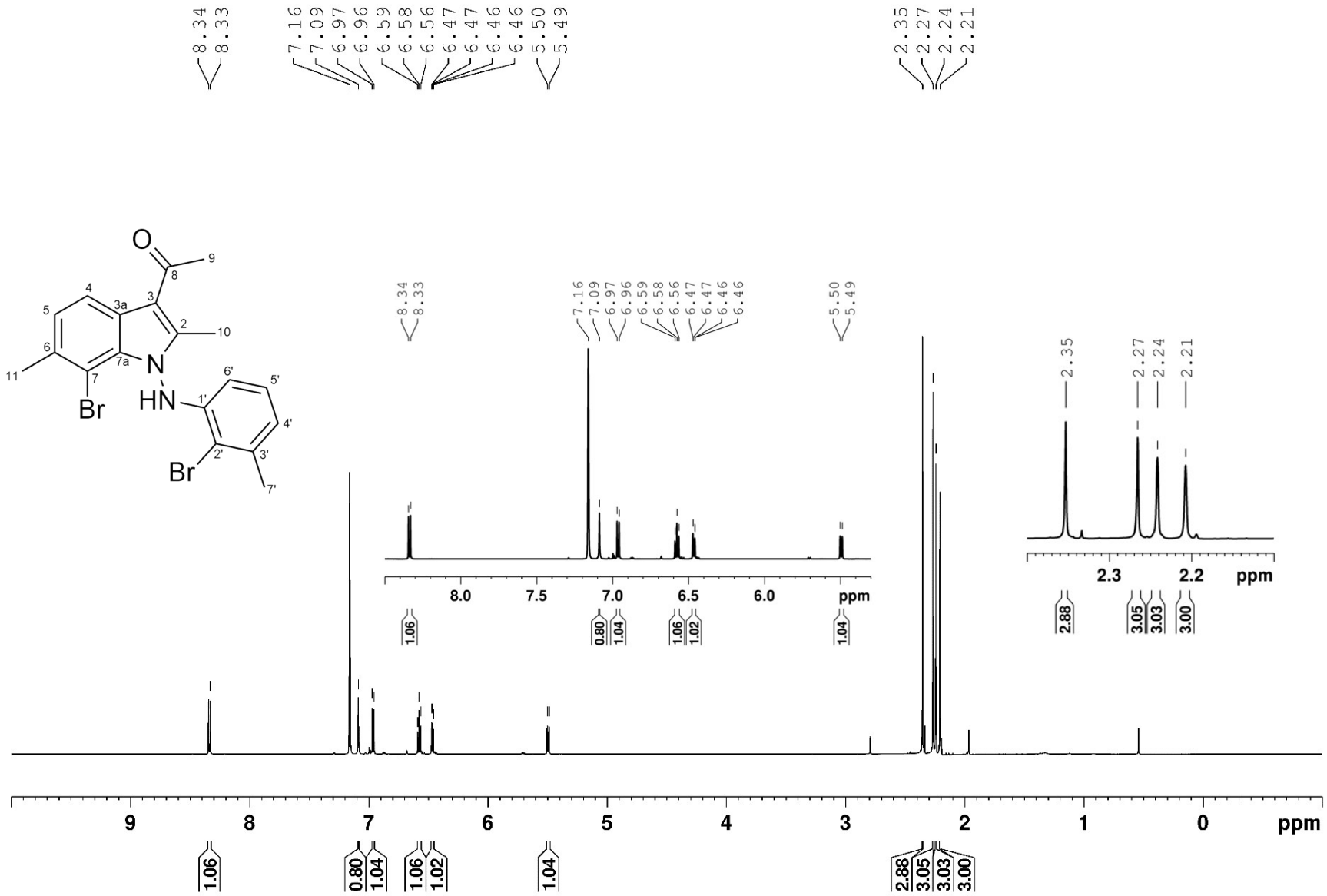


Fig. 49 ^{1}H (600 MHz) spectra of **7d** in C_6D_6

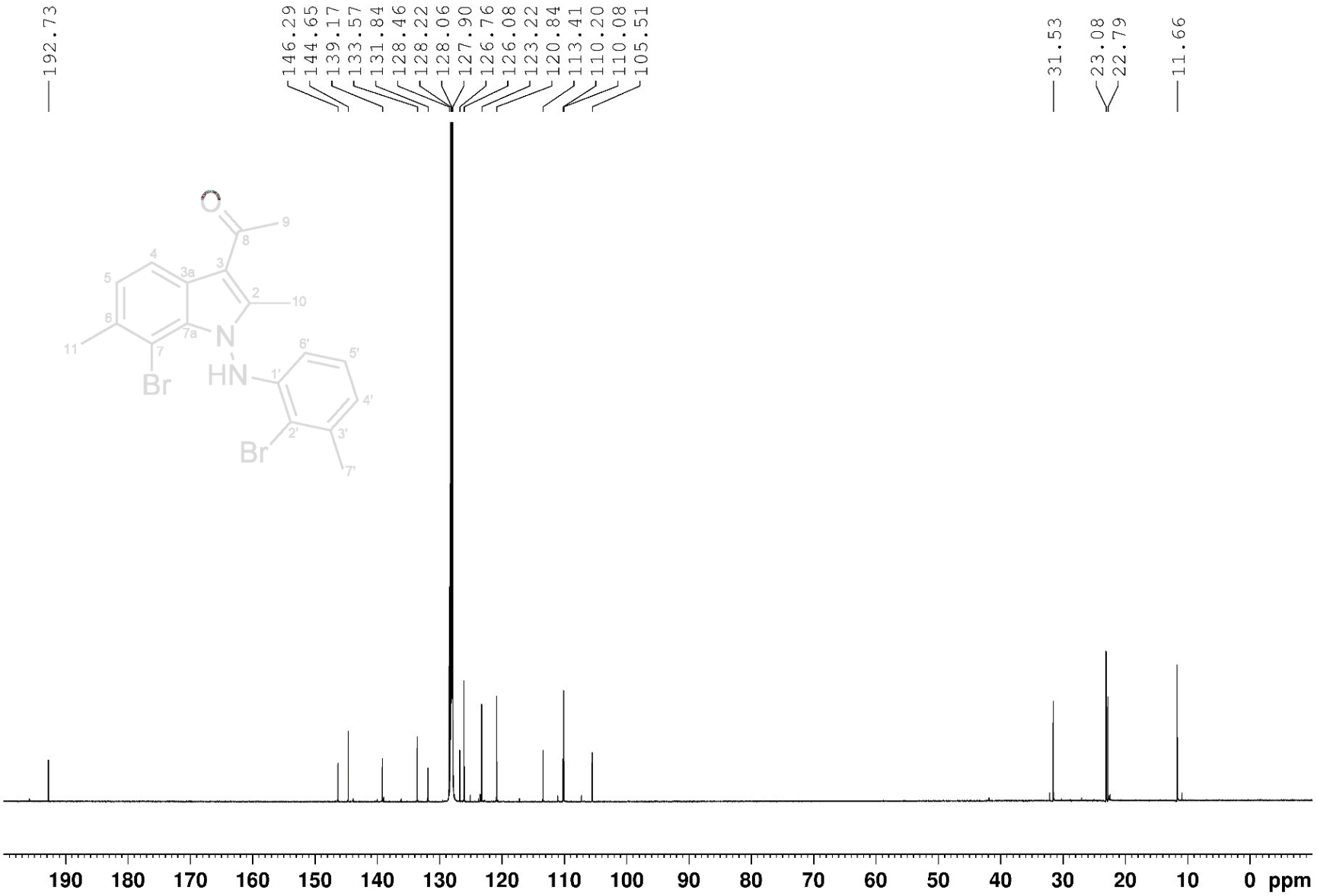
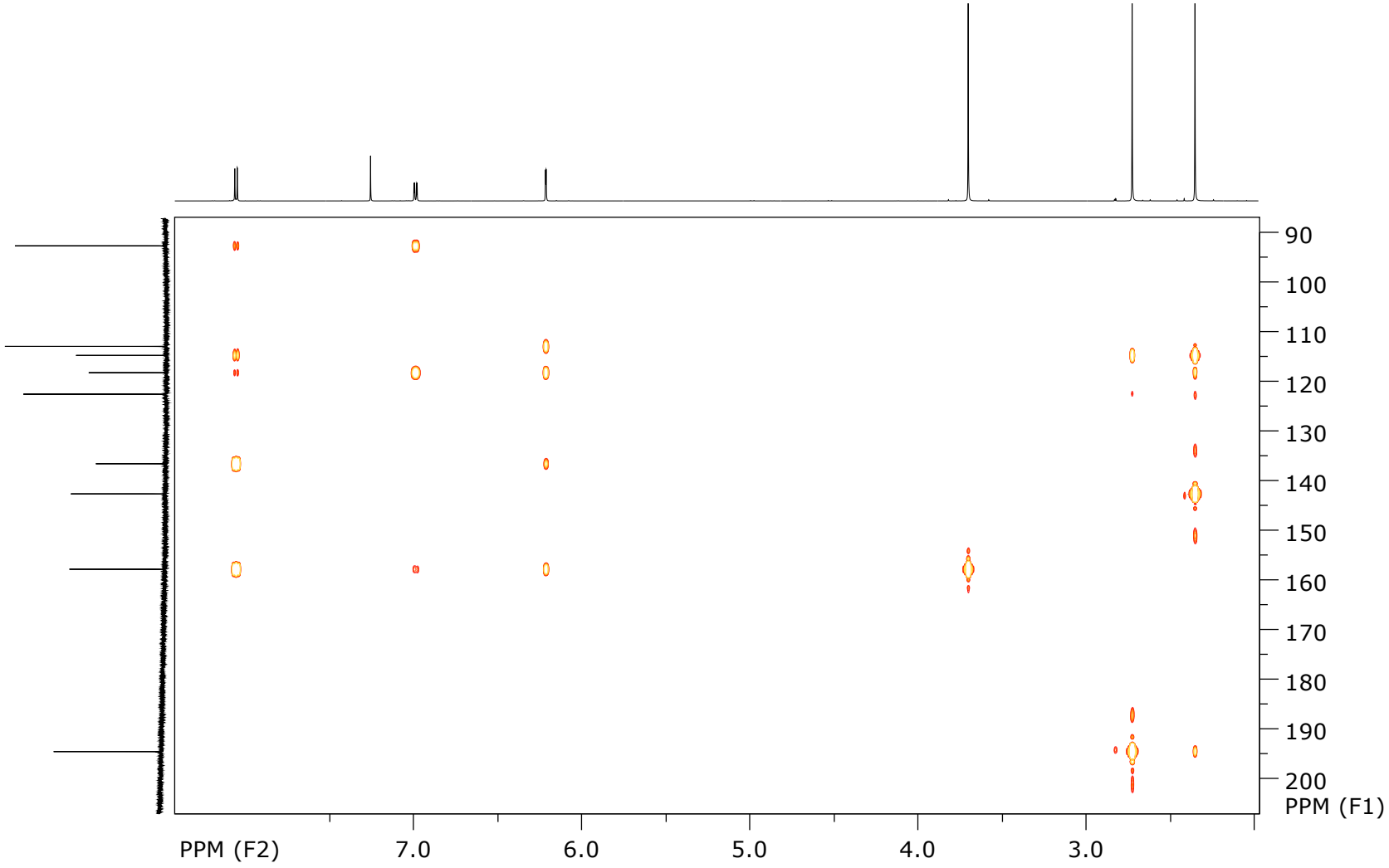


Fig. 50 ^{13}C (150MHz) NMR spectra of **7d** in C_6D_6

Important spectra for the structure elucidation of 4h/8h and 4g



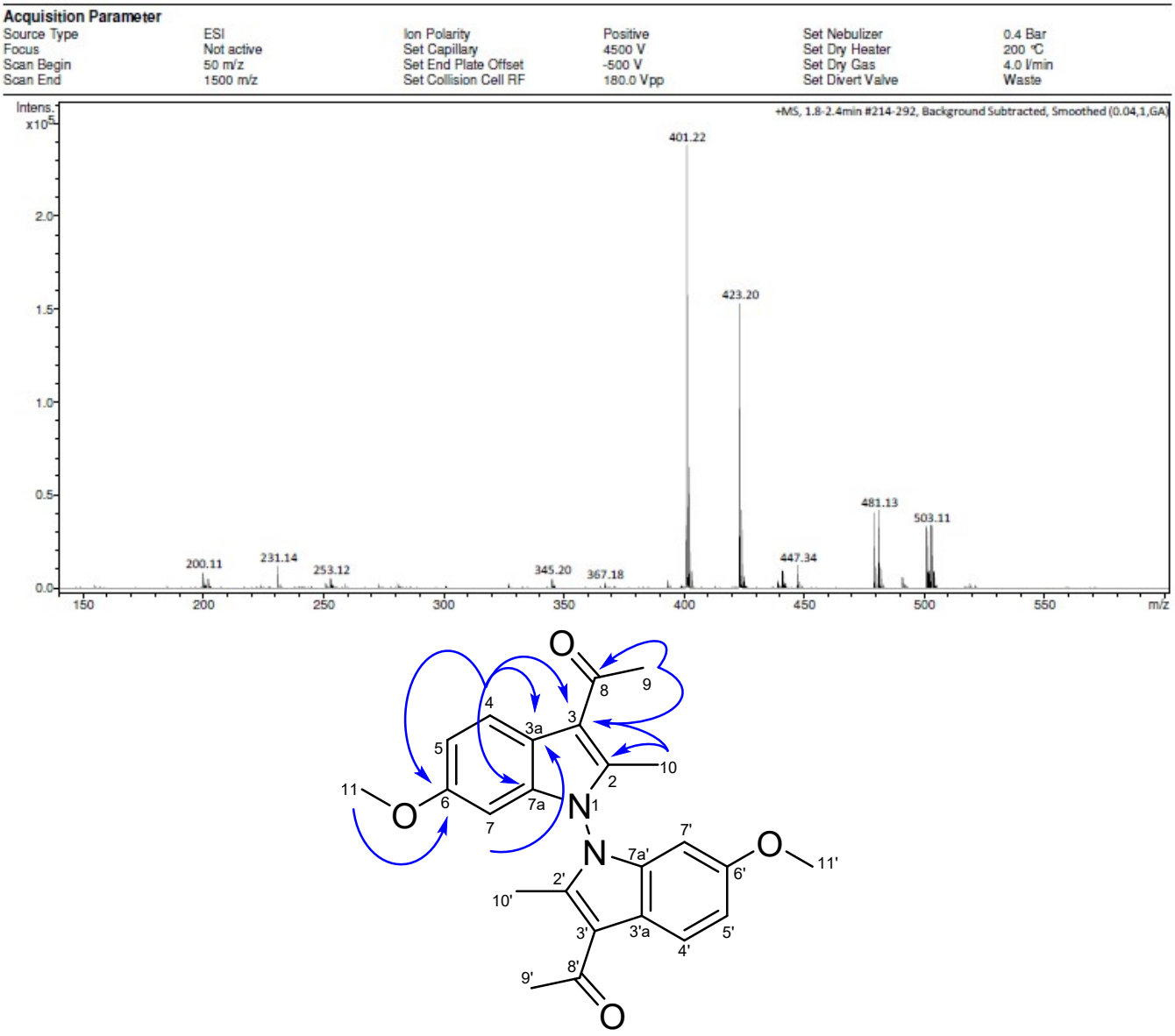
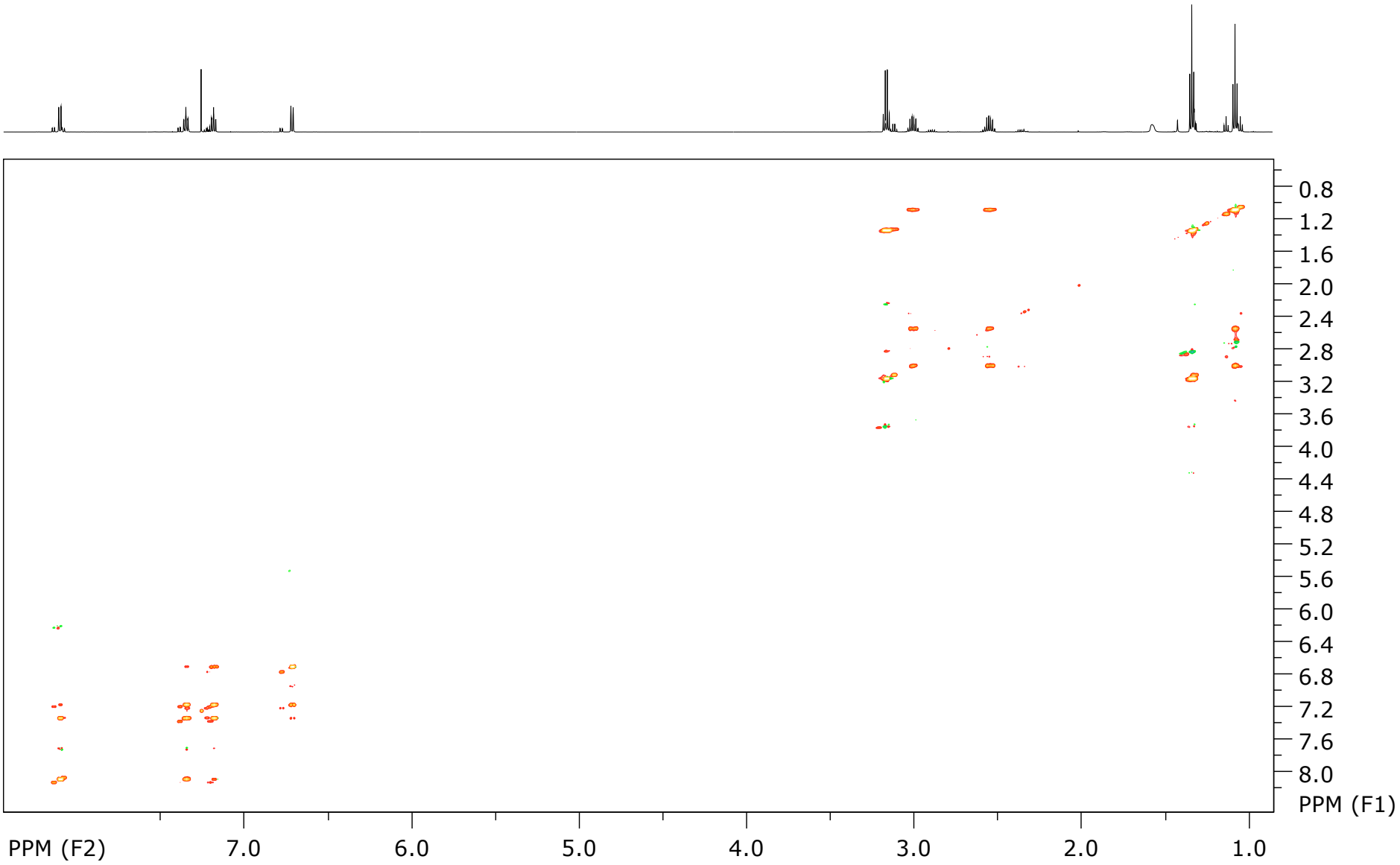
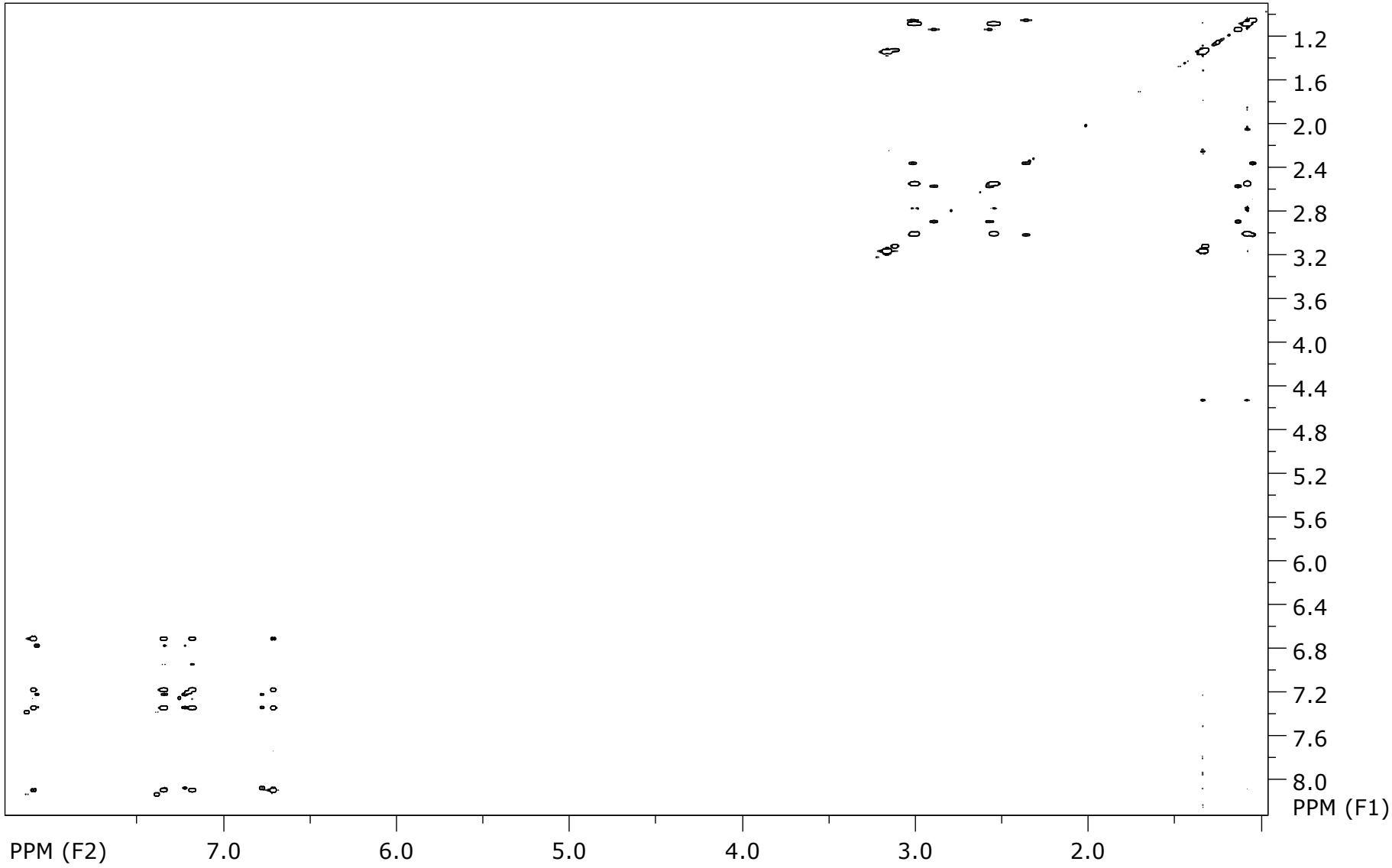


Fig. 51 $^{1}H,^{13}C$ HMBC spectrum in $CDCl_3$ at 600 MHz (top), ESI MS spectrum (middle) and chemical structure of **4g** with HMBC correlations (blue arrows)

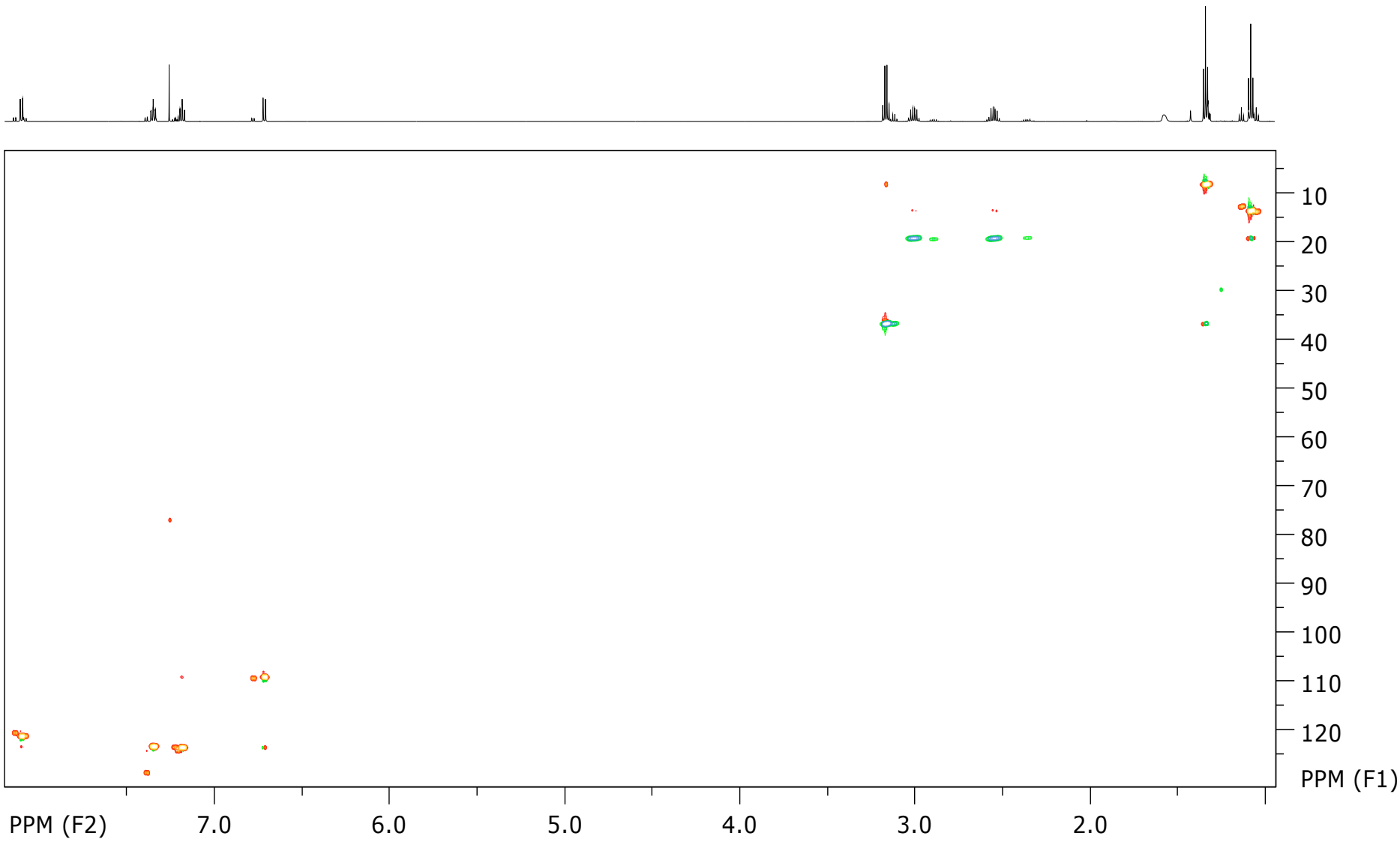
A


0.8
1.2
1.6
2.0
2.4
2.8
3.2
3.6
4.0
4.4
4.8
5.2
5.6
6.0
6.4
6.8
7.2
7.6
8.0
PPM (F1)
PPM (F2)
7.0
6.0
5.0
4.0
3.0
2.0
1.0

B


1.2
1.6
2.0
2.4
2.8
3.2
3.6
4.0
4.4
4.8
5.2
5.6
6.0
6.4
6.8
7.2
7.6
8.0
PPM (F1)
PPM (F2)
7.0
6.0
5.0
4.0
3.0
2.0

c



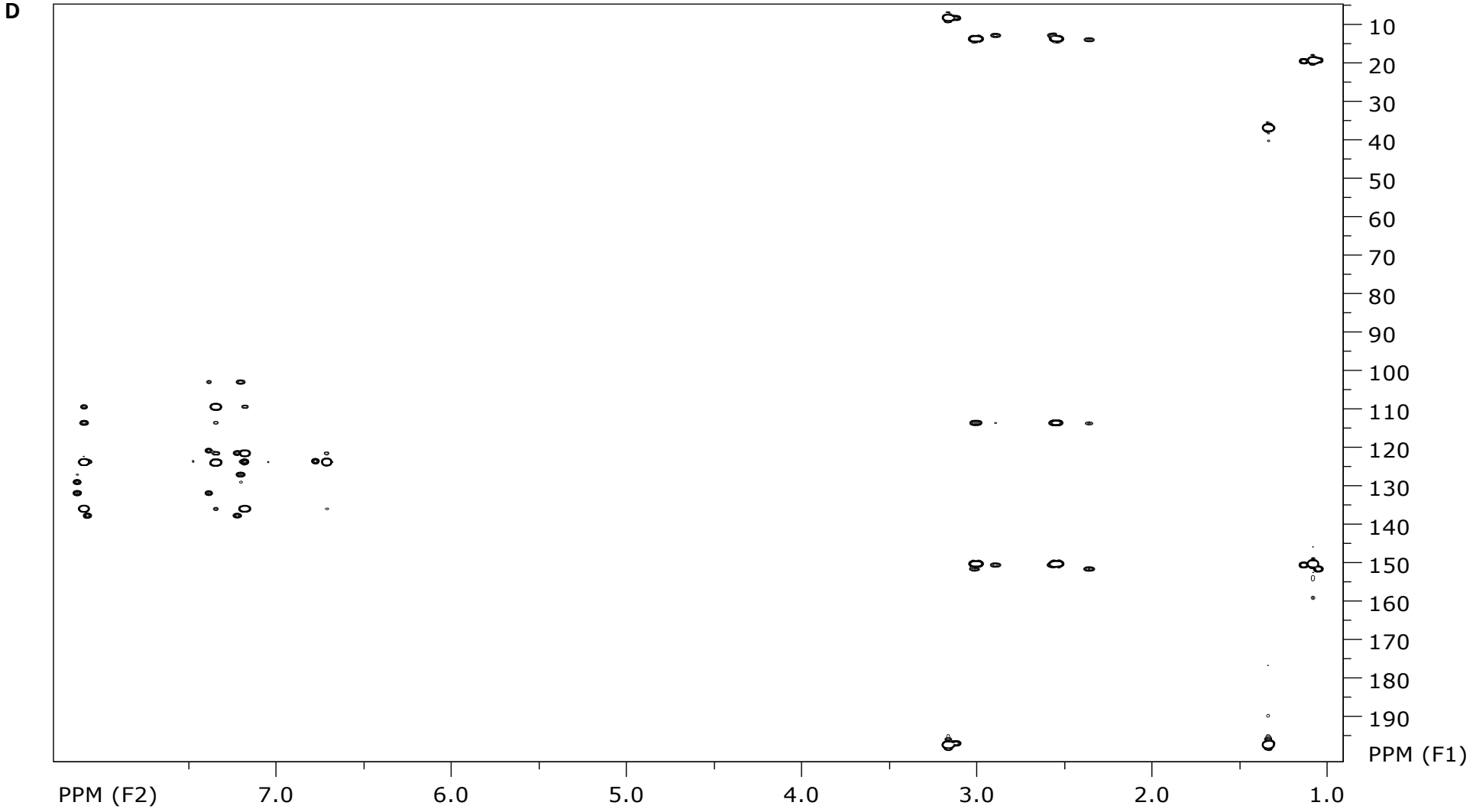


Fig. 52 PSYCHE CLIP COSY spectrum (A), PSYCHE TOCSY spectrum (B), $^{1}H,^{13}C$ HSQC spectrum (C) and $^{1}H,^{13}C$ HMBC spectrum (D) of **4h/8h** in $CDCl_3$ at 600 MHz

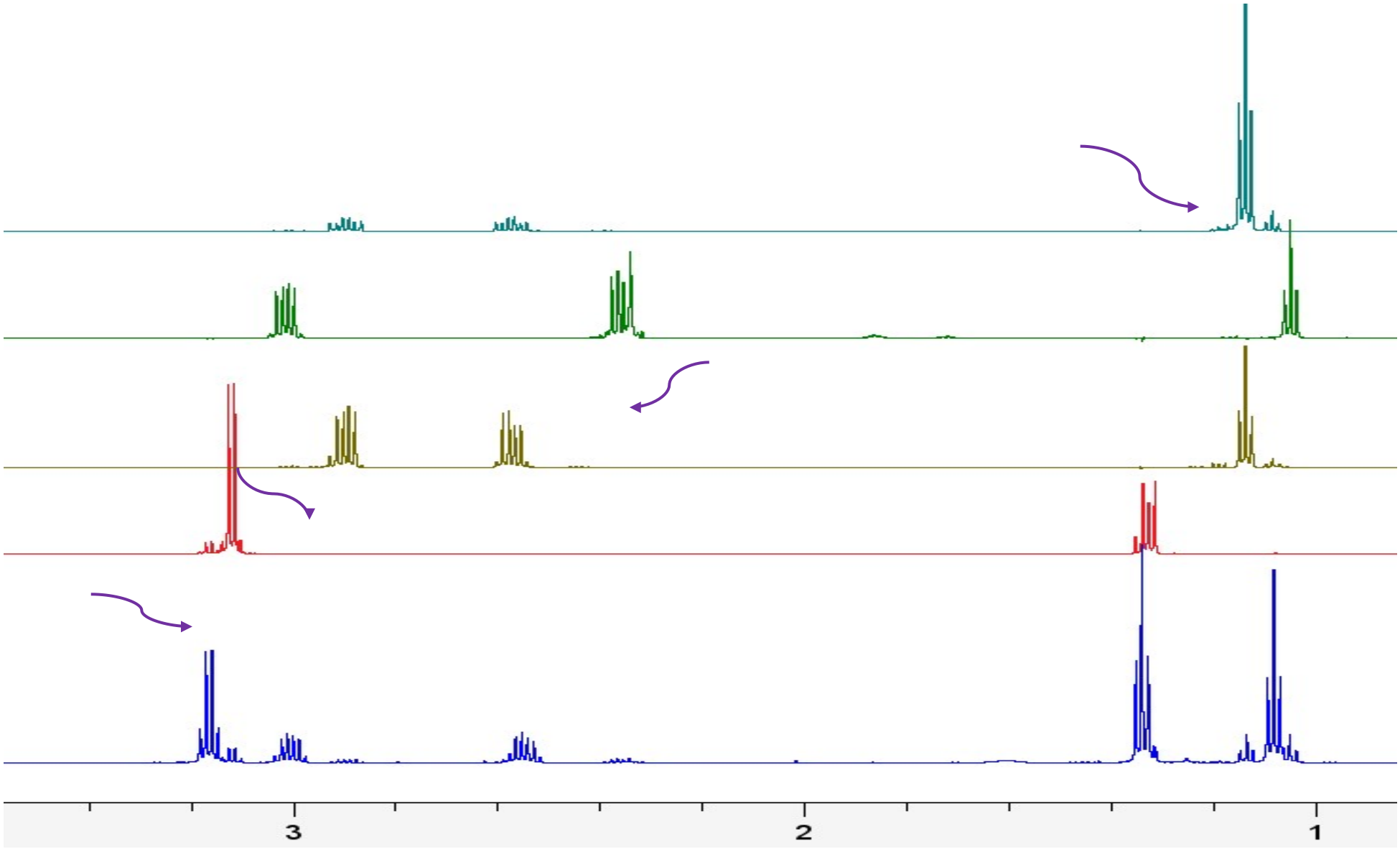
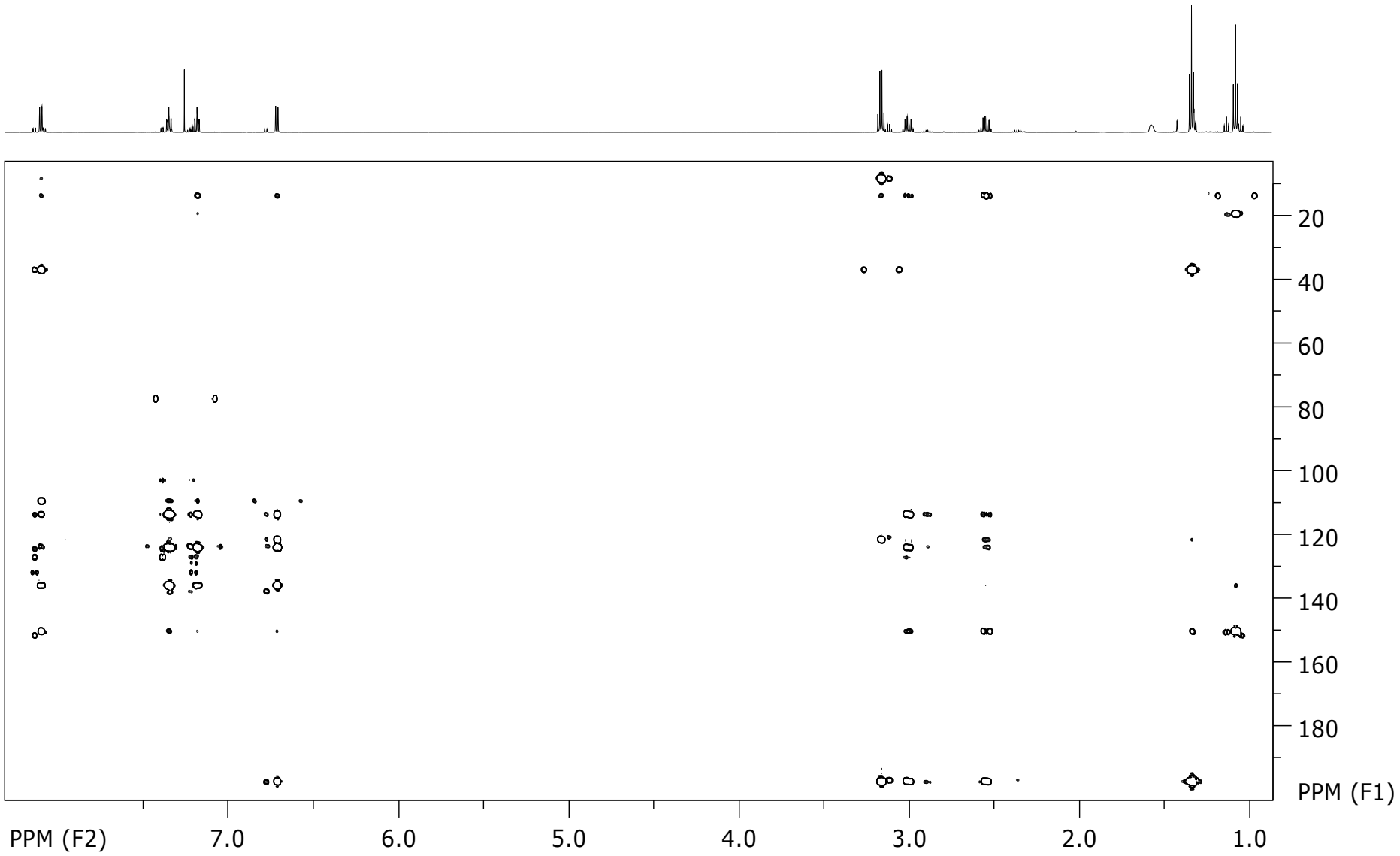


Fig. 53 Expansion of ^{1}H NMR of **4h/8h** (bottom) and selective 1D TOCSYs. Sites of irradiation are indicated by purple arrows

A



B

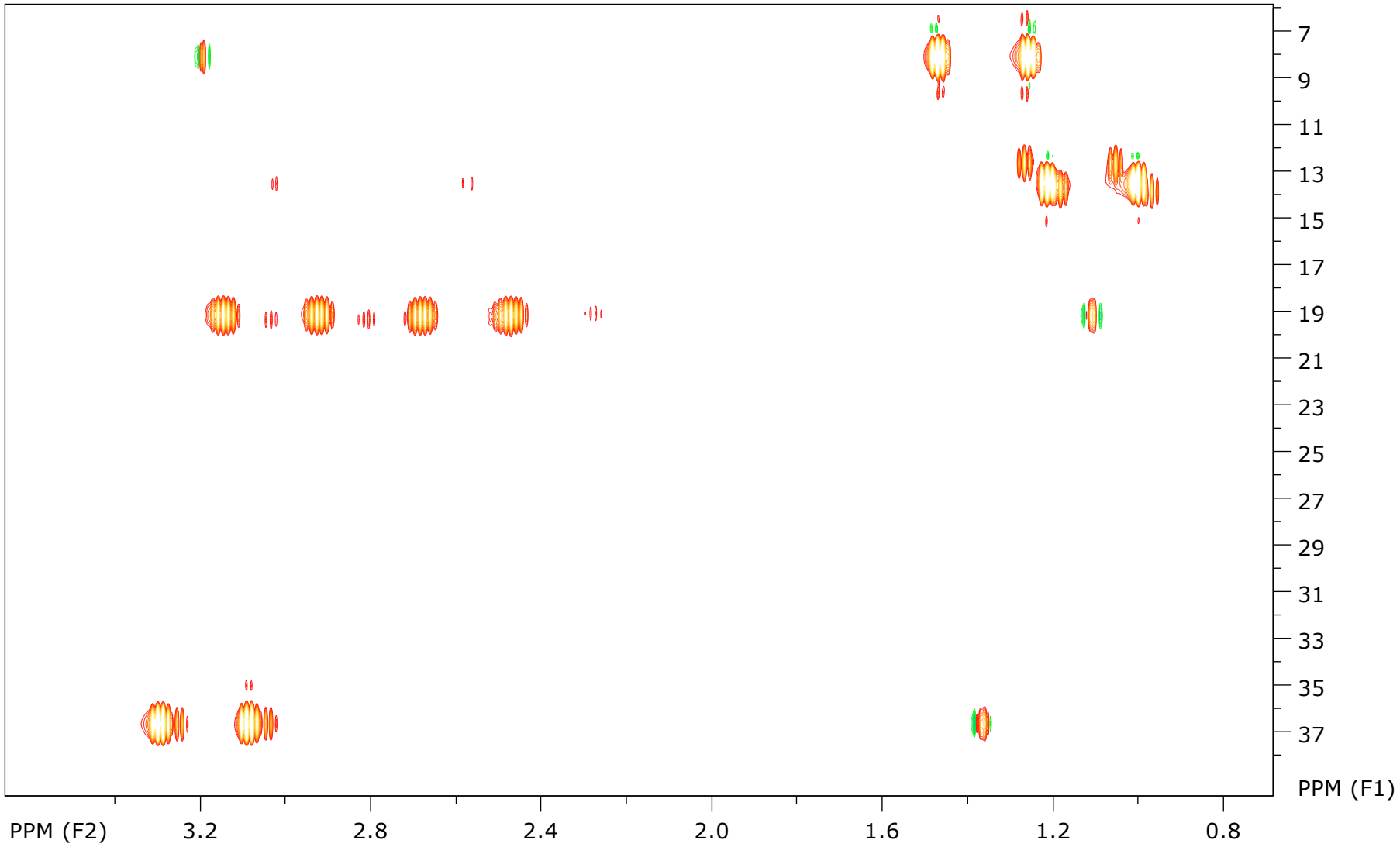


Fig. 54 Super long range $^{1}H,^{13}C$ HMBC (A) and bandselective nondecoupled $^{1}H,^{13}C$ HSQC (B) of **4h/8h** in $CDCl_3$ at 600 MHz