

Supporting Information

A general and stereoselective approach to 14-membered cyclic bis-semicarbazones involving BF₃-catalyzed amidoalkylation of 2-(trimethylsilyloxy)propene

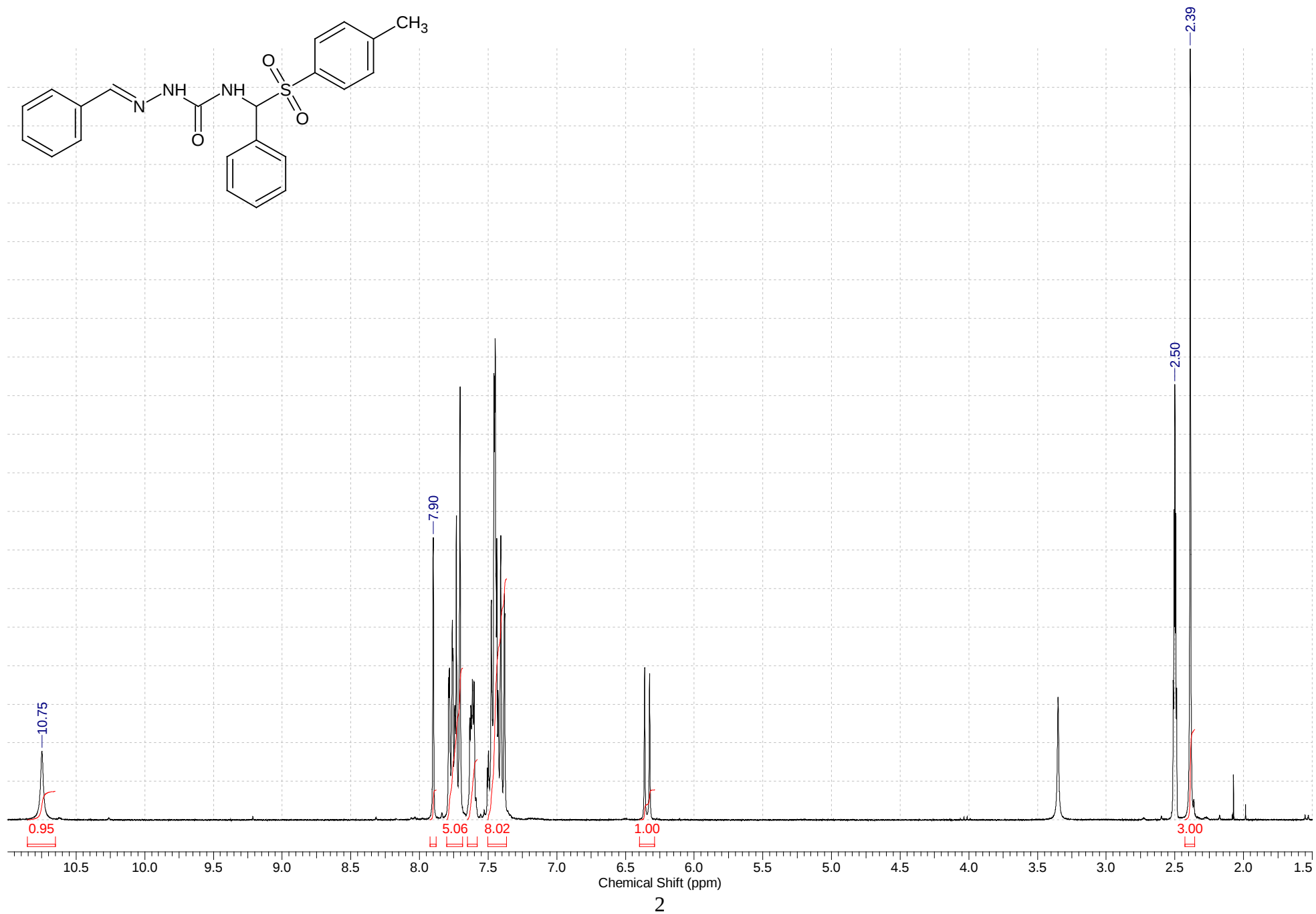
Anastasia A. Fesenko and Anatoly D. Shutalev*

*N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 47 Leninsky Ave.,
119991 Moscow, Russian Federation*

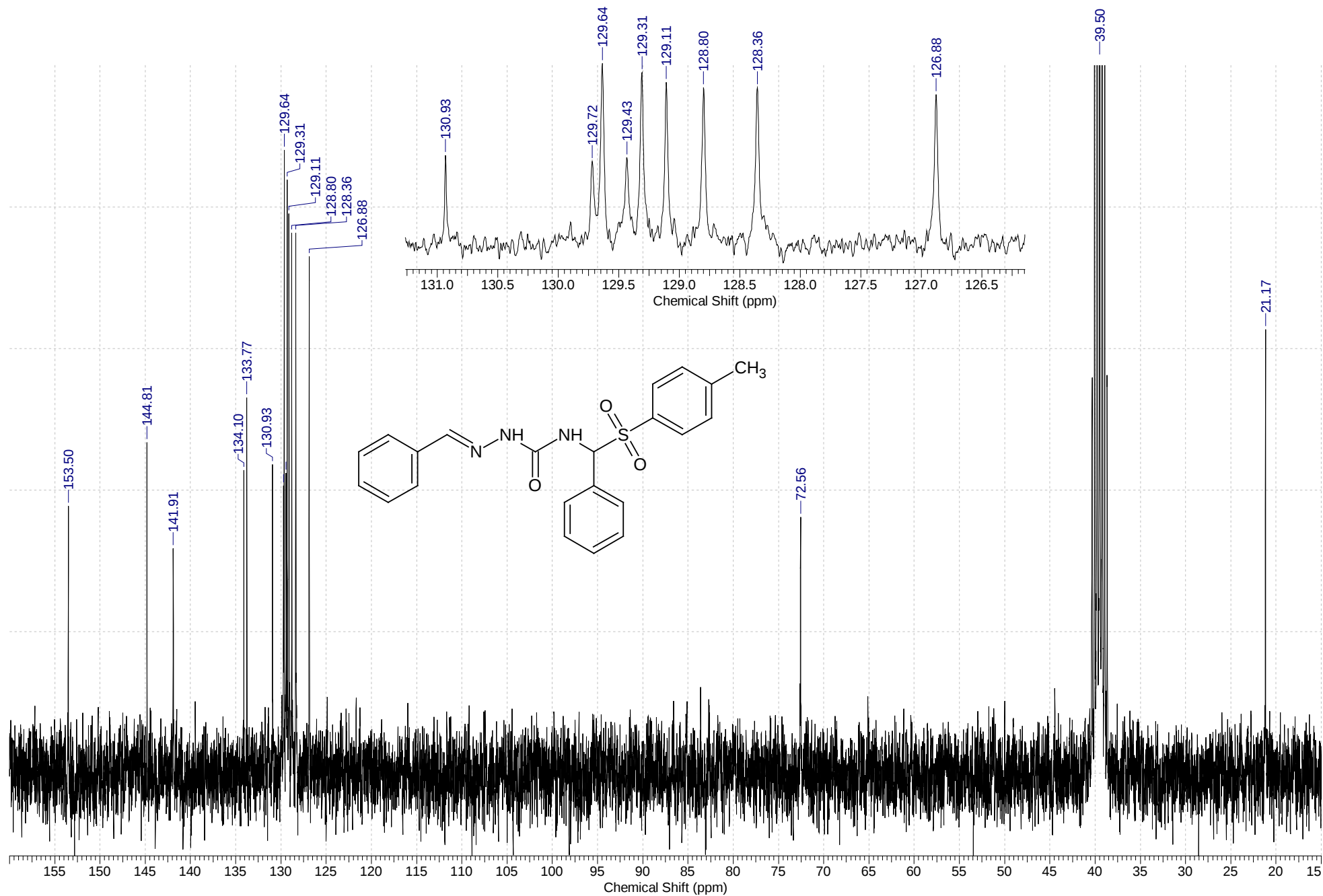
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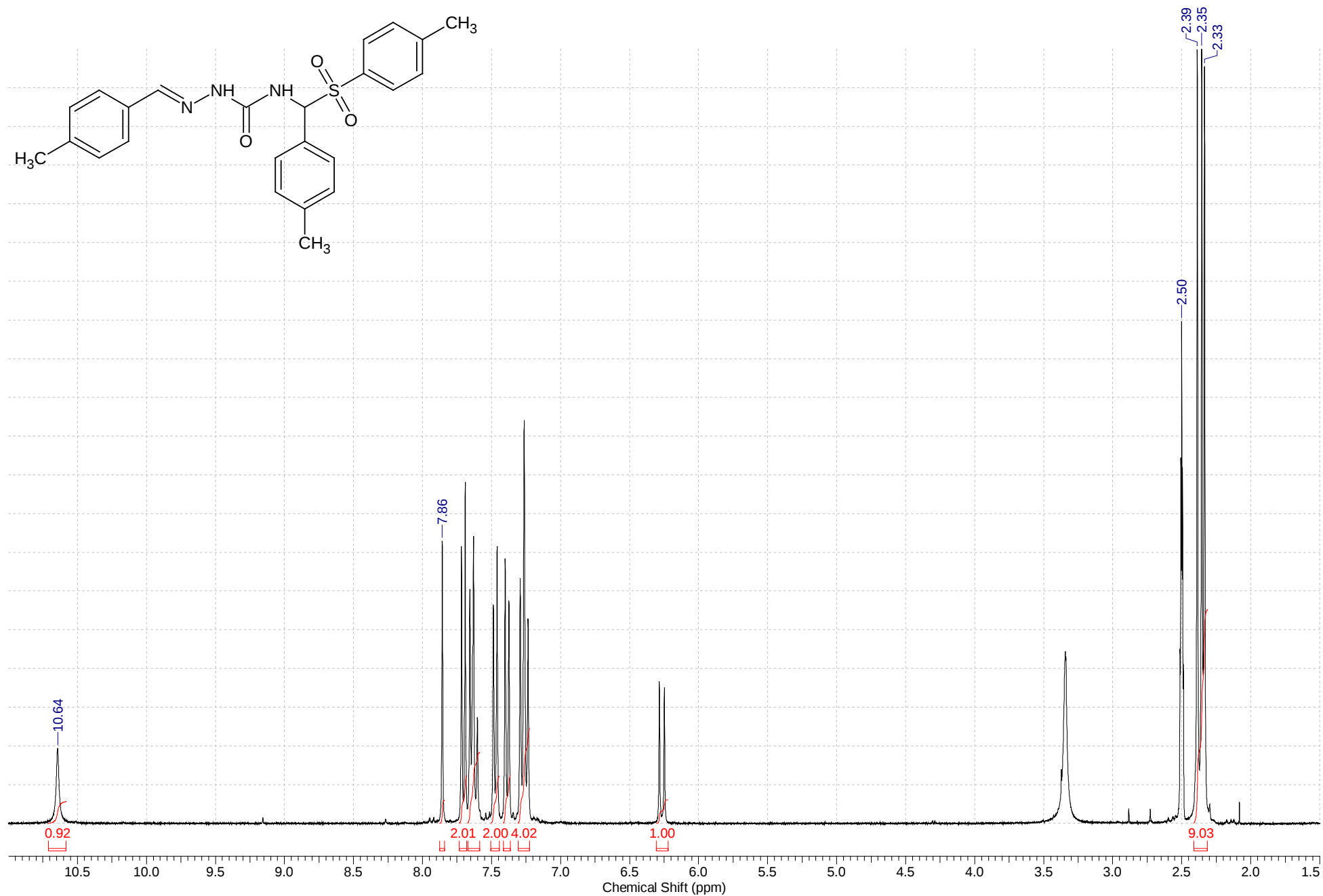
¹H NMR spectrum of compound **5a** (DMSO-*d*₆).



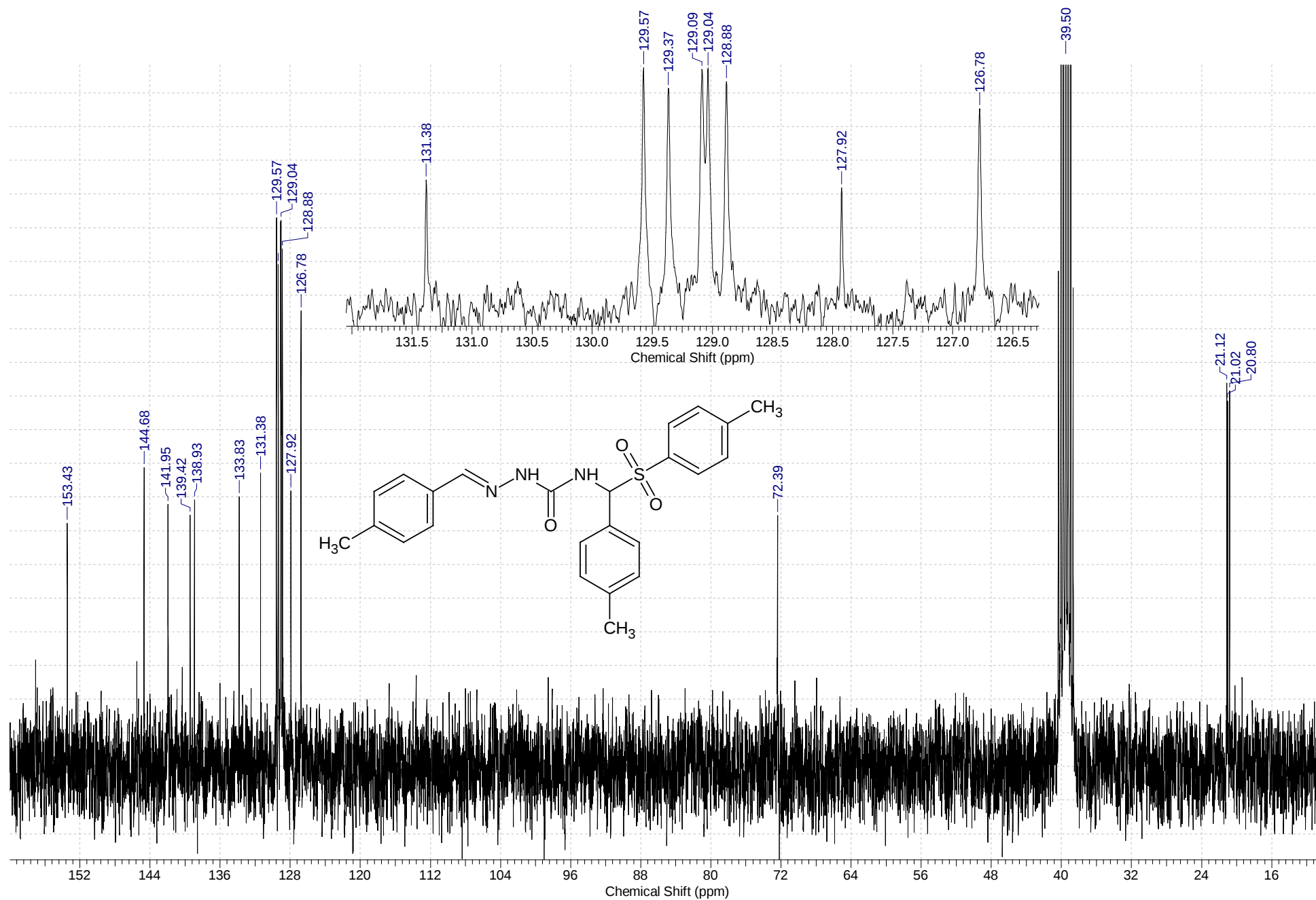
^{13}C NMR spectrum of compound **5a** (DMSO- d_6).



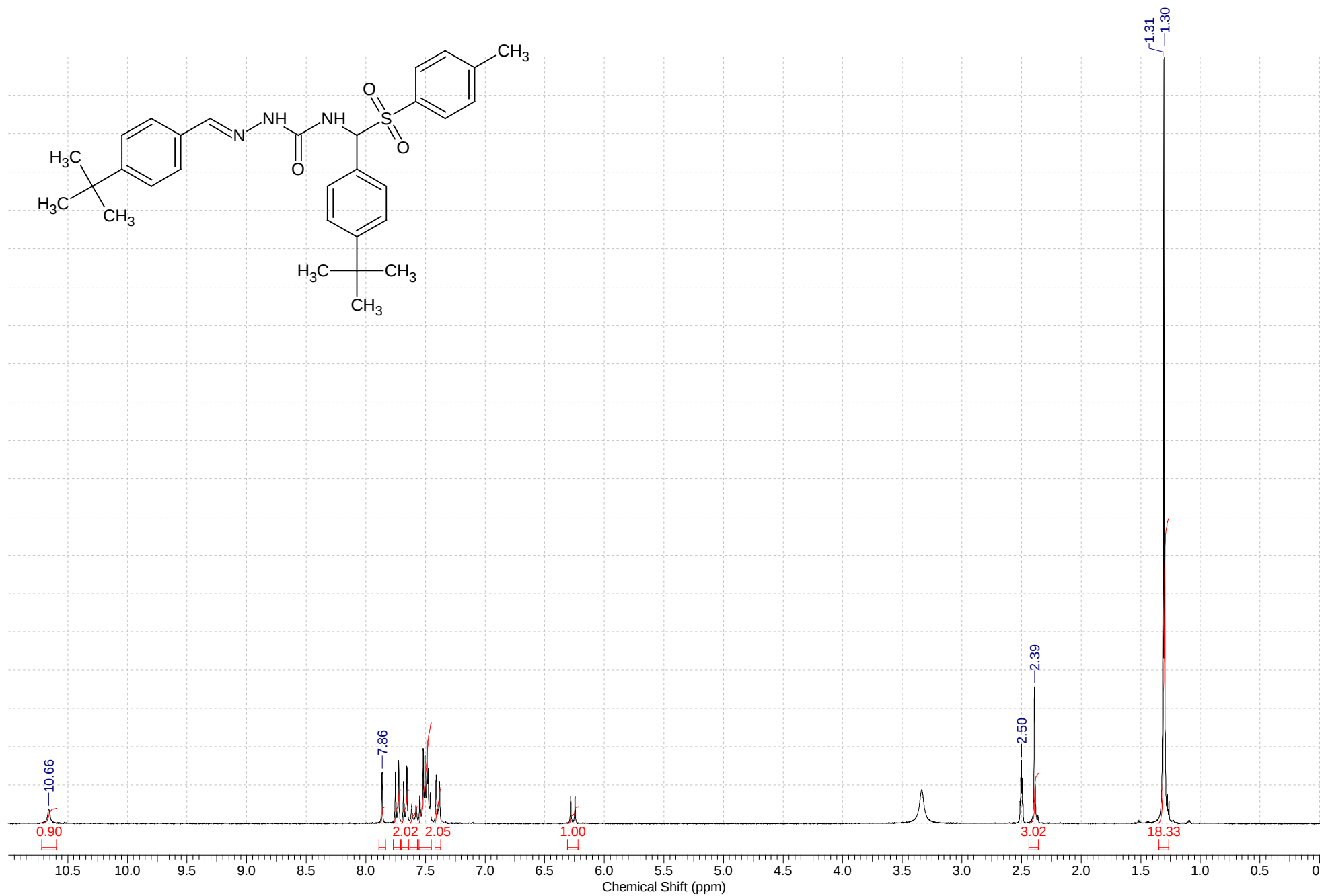
^1H NMR spectrum of compound **5b** ($\text{DMSO}-d_6$).



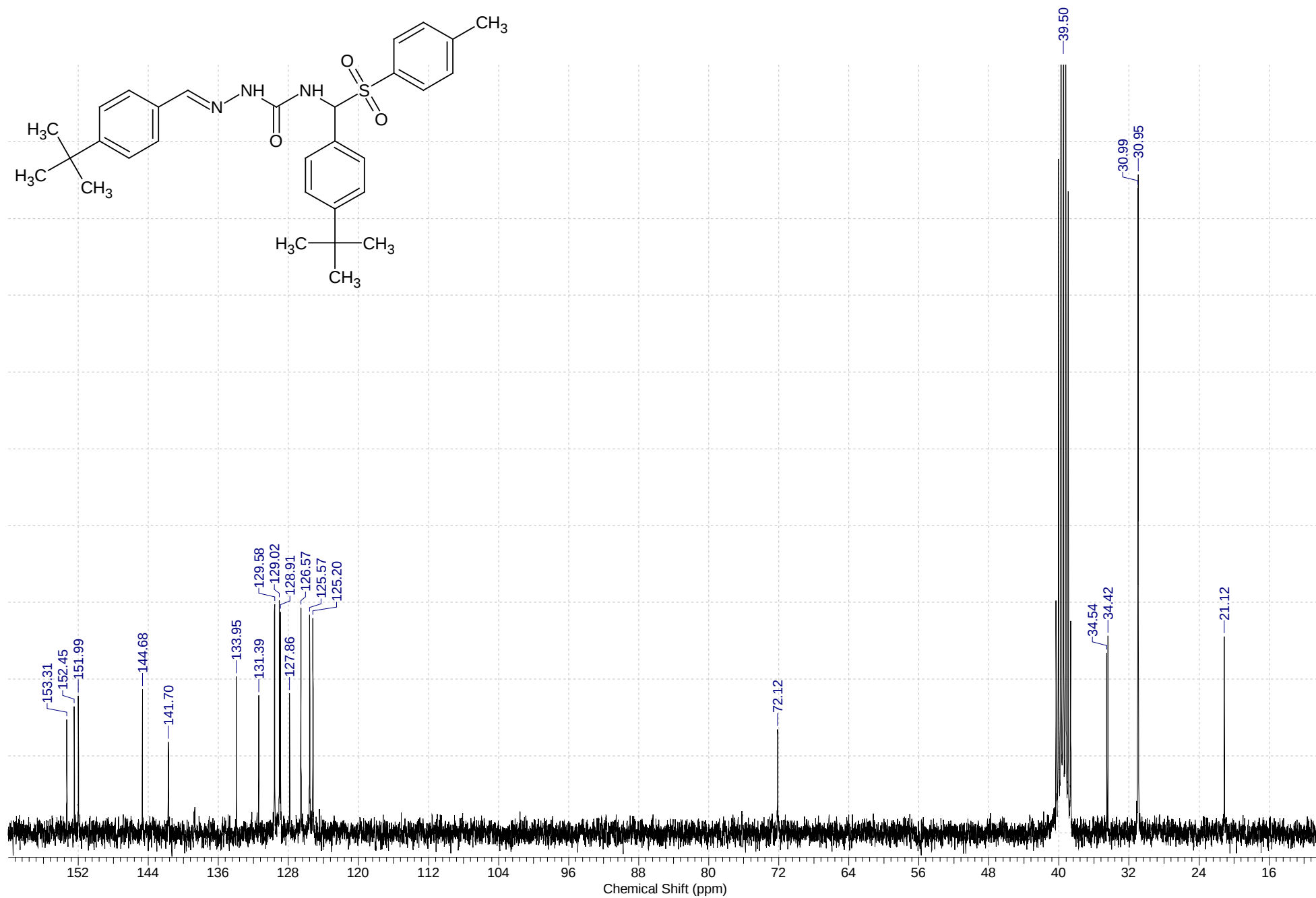
^{13}C NMR spectrum of compound **5b** (DMSO- d_6).



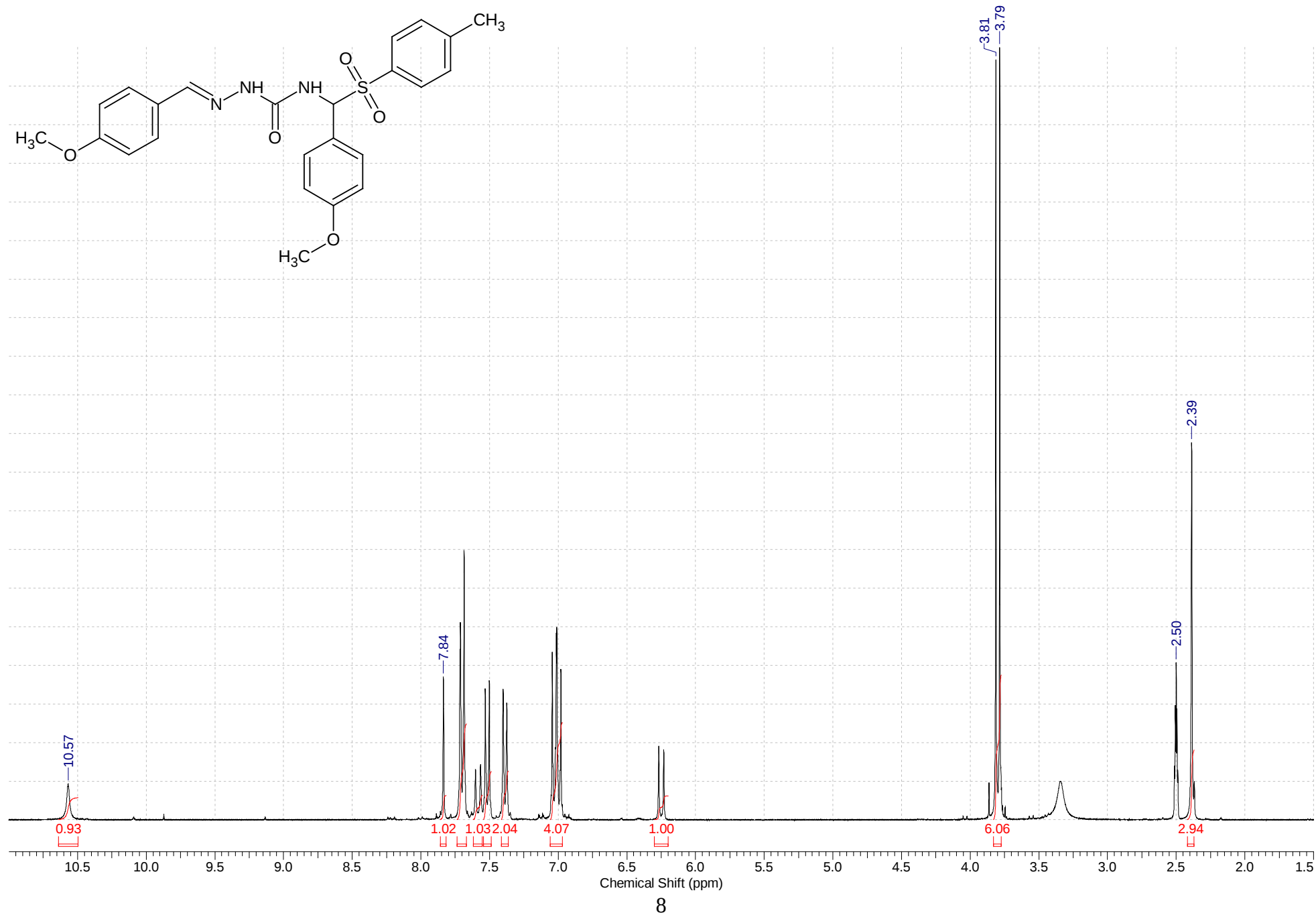
¹H NMR spectrum of compound 5c (DMSO-*d*₆).



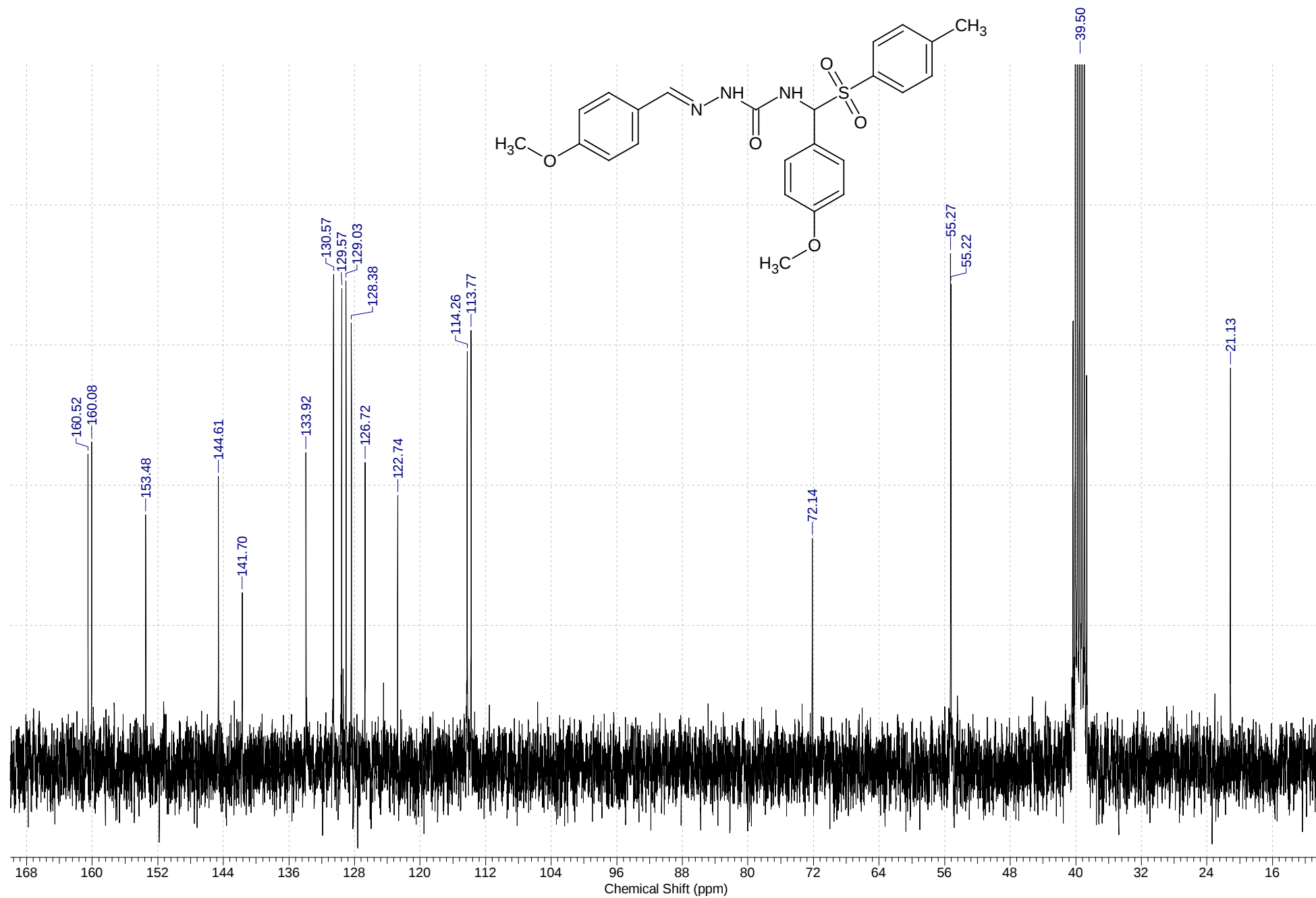
^{13}C NMR spectrum of compound **5c** ($\text{DMSO}-d_6$).



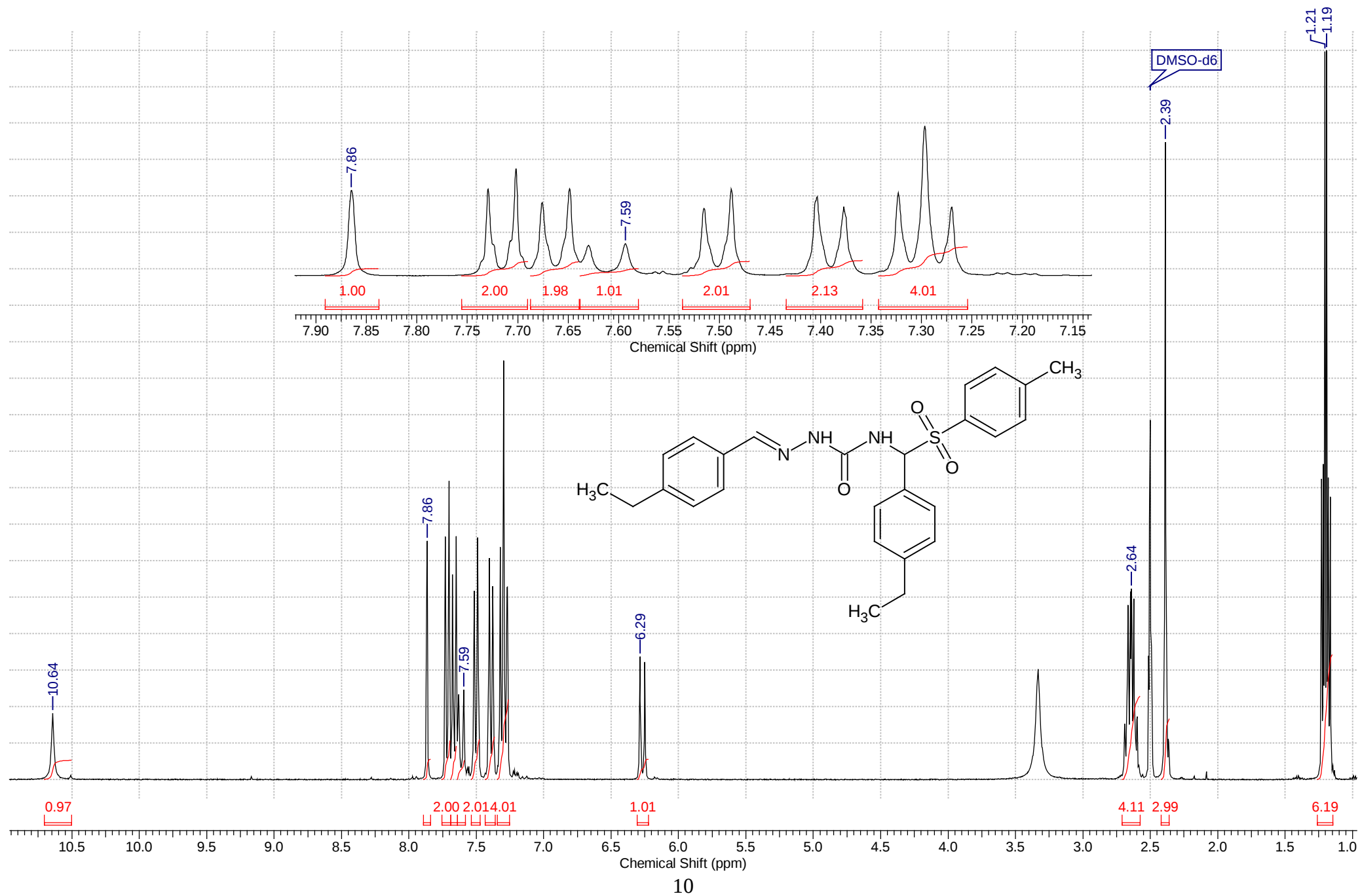
¹H NMR spectrum of compound **5d** (DMSO-*d*₆).



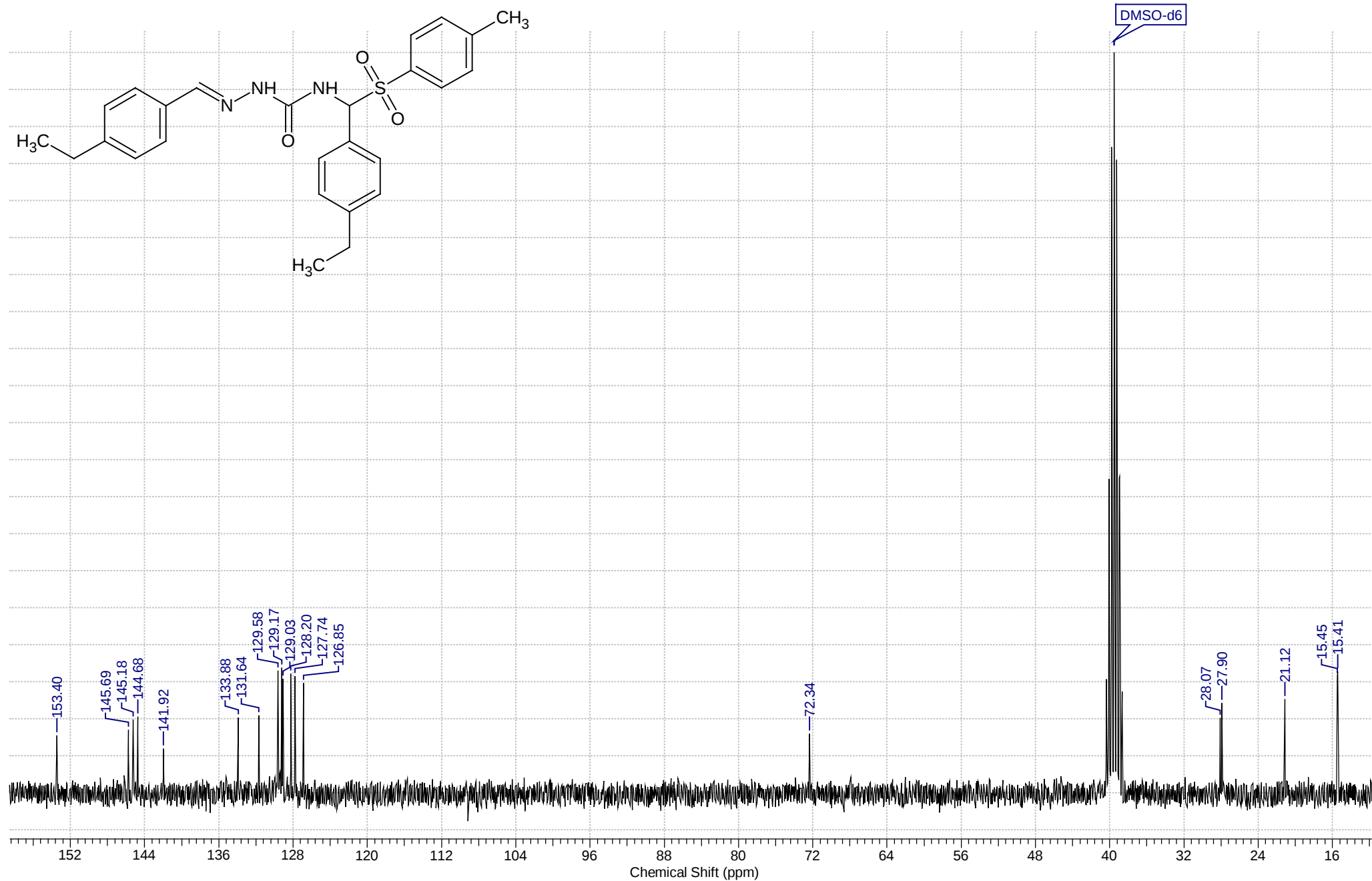
^{13}C NMR spectrum of compound **5d** (DMSO- d_6).



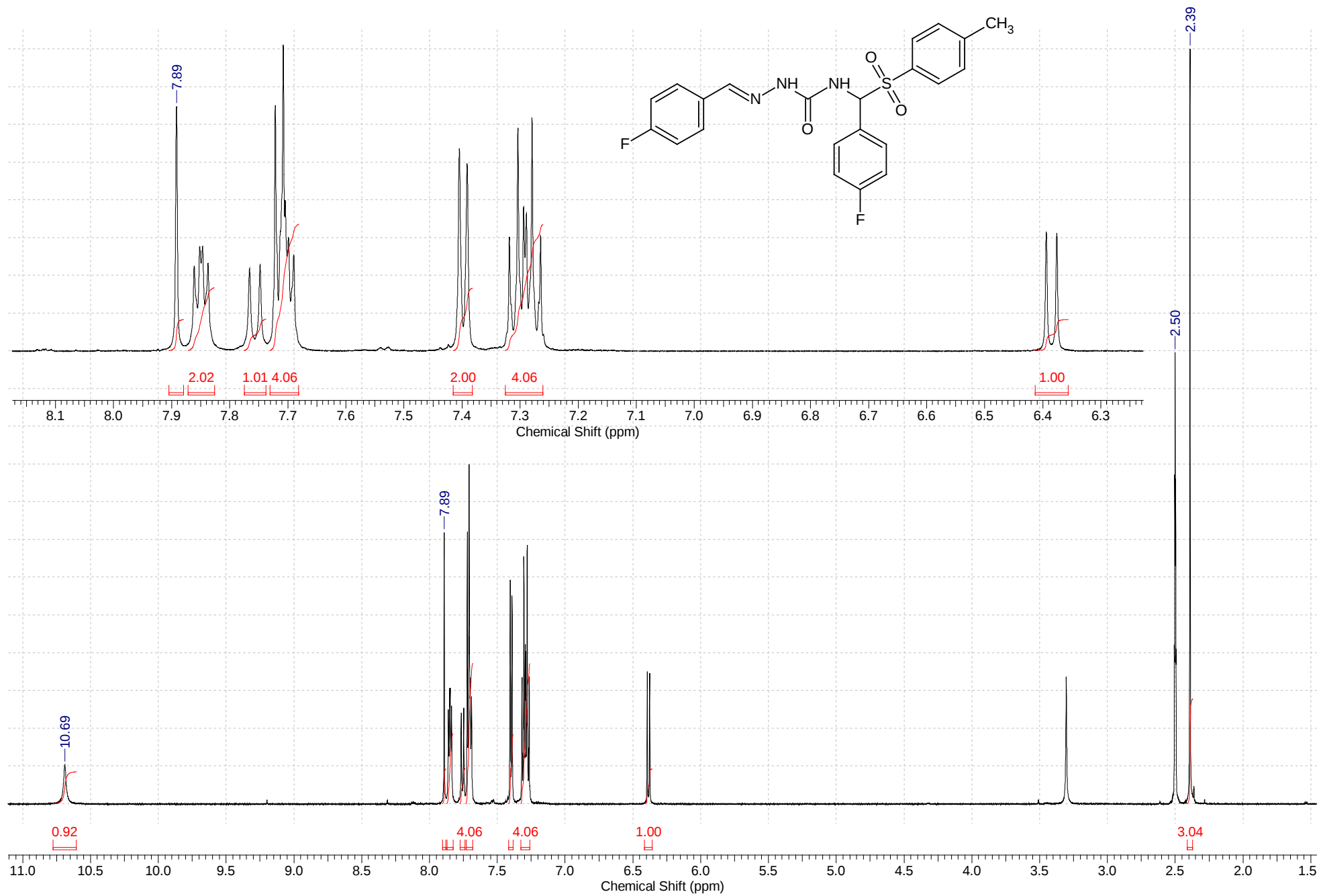
^1H NMR spectrum of compound **5e** ($\text{DMSO}-d_6$).



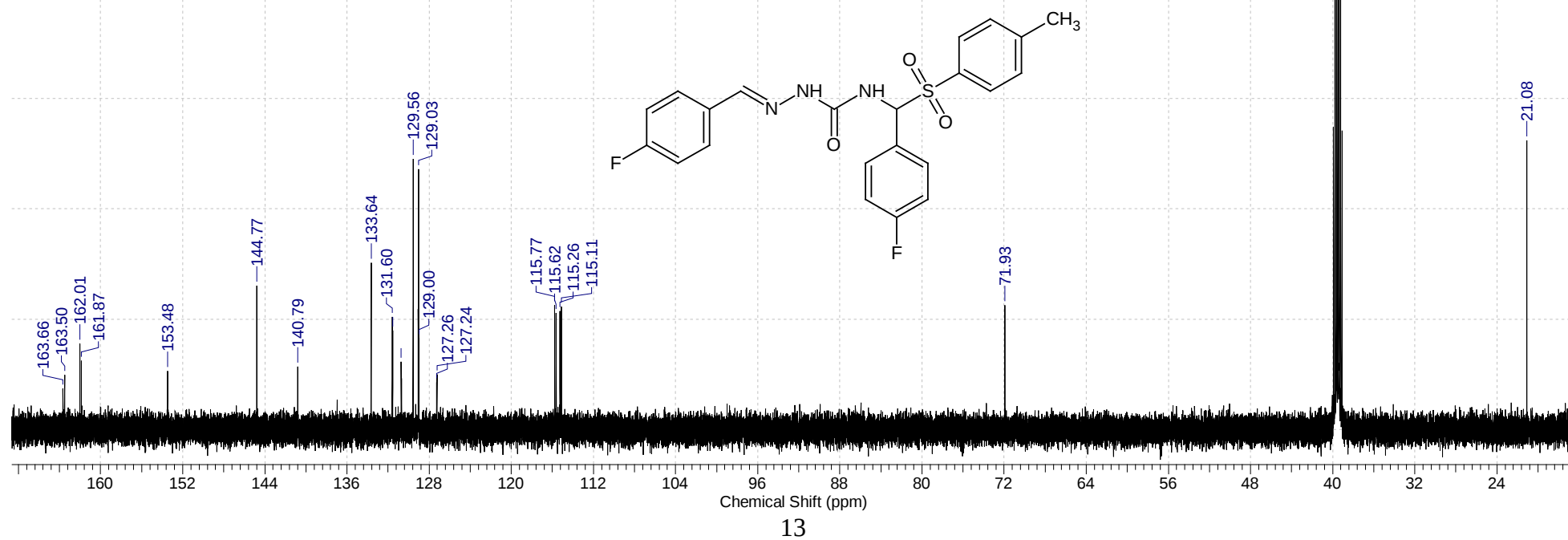
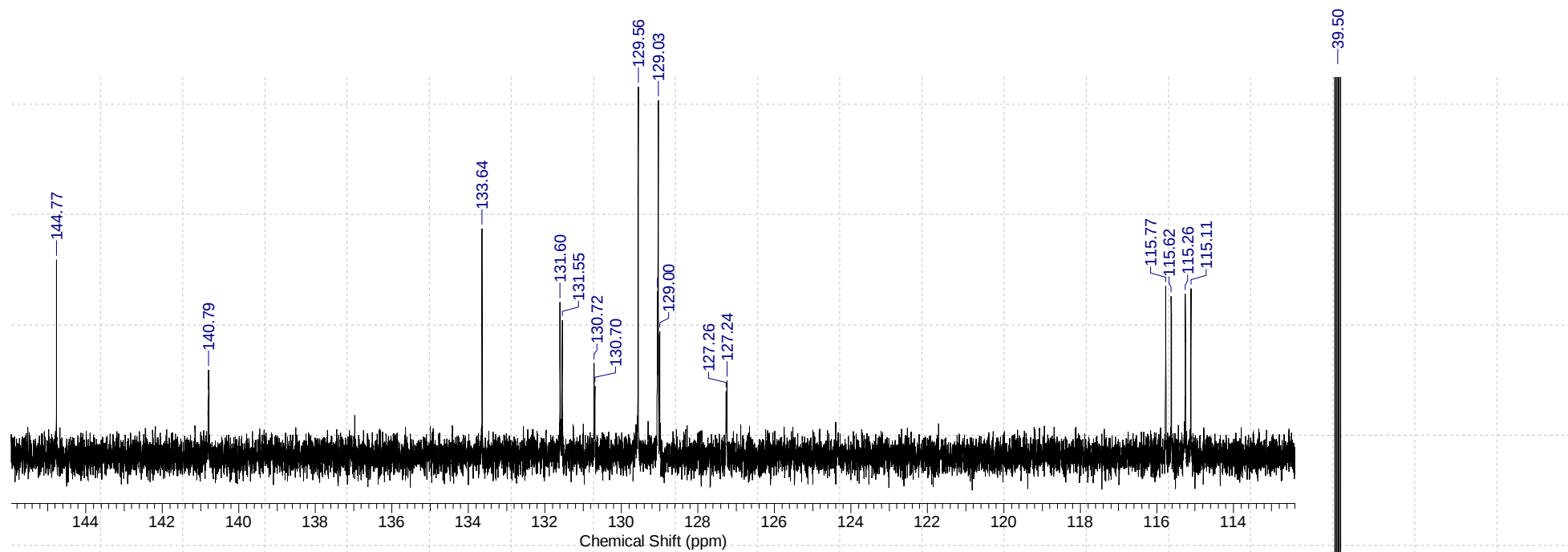
^{13}C NMR spectrum of compound **5e** ($\text{DMSO-}d_6$).



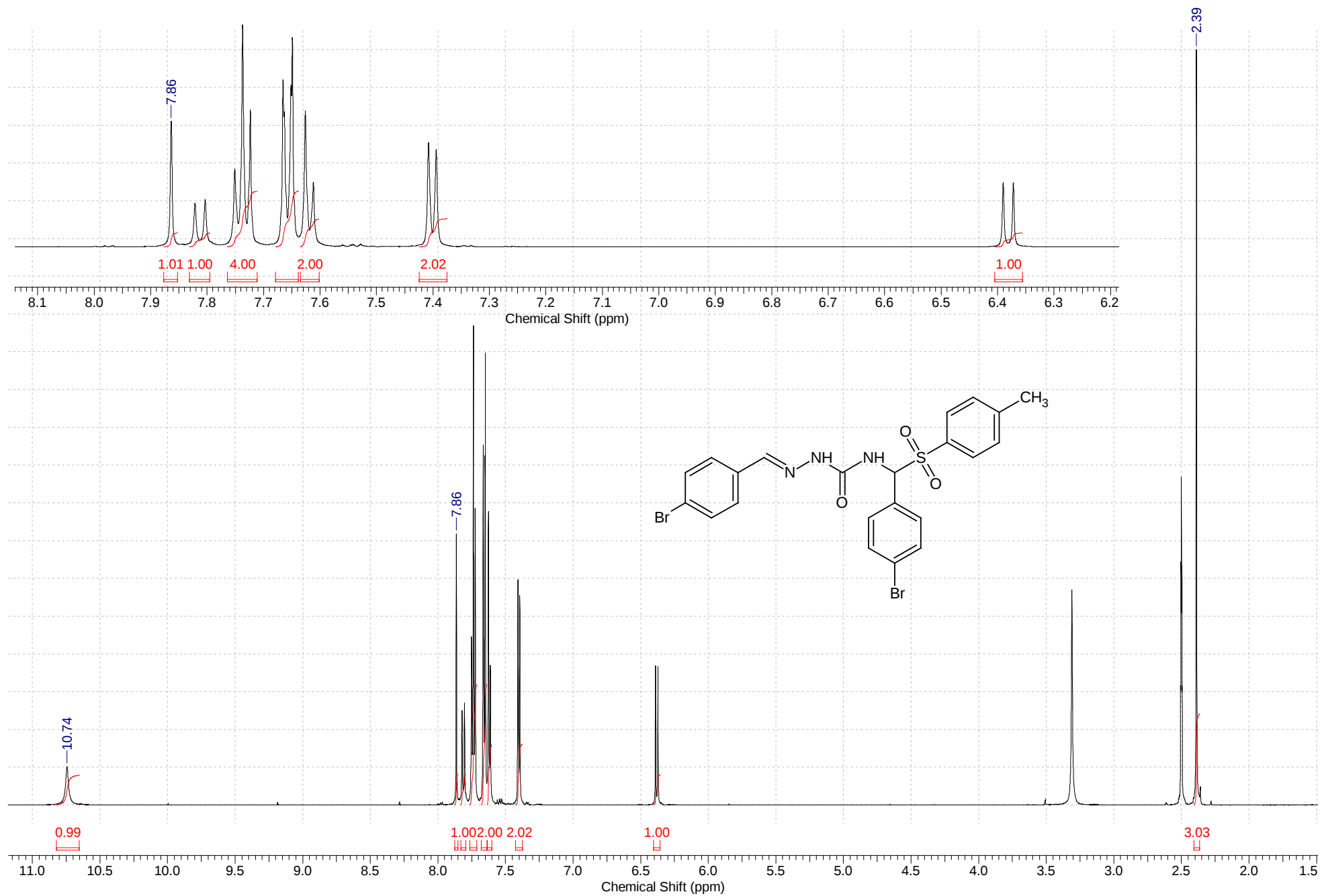
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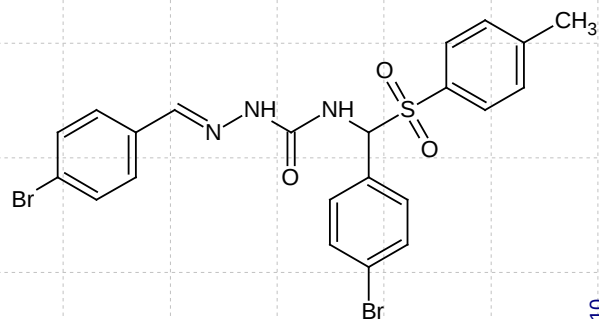
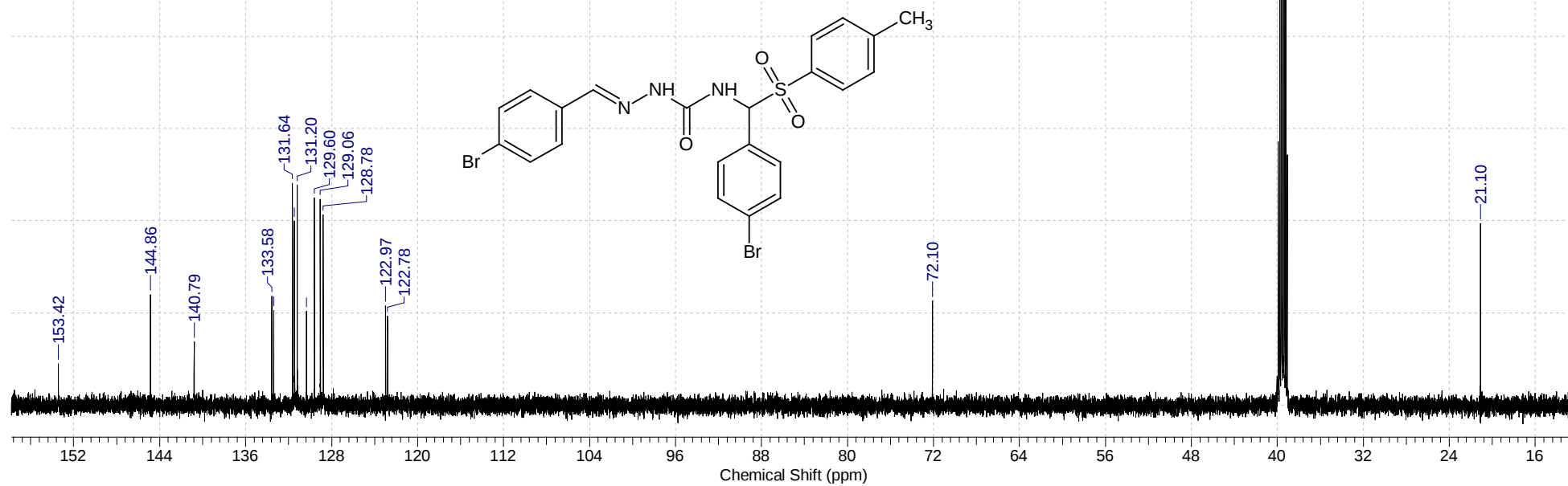
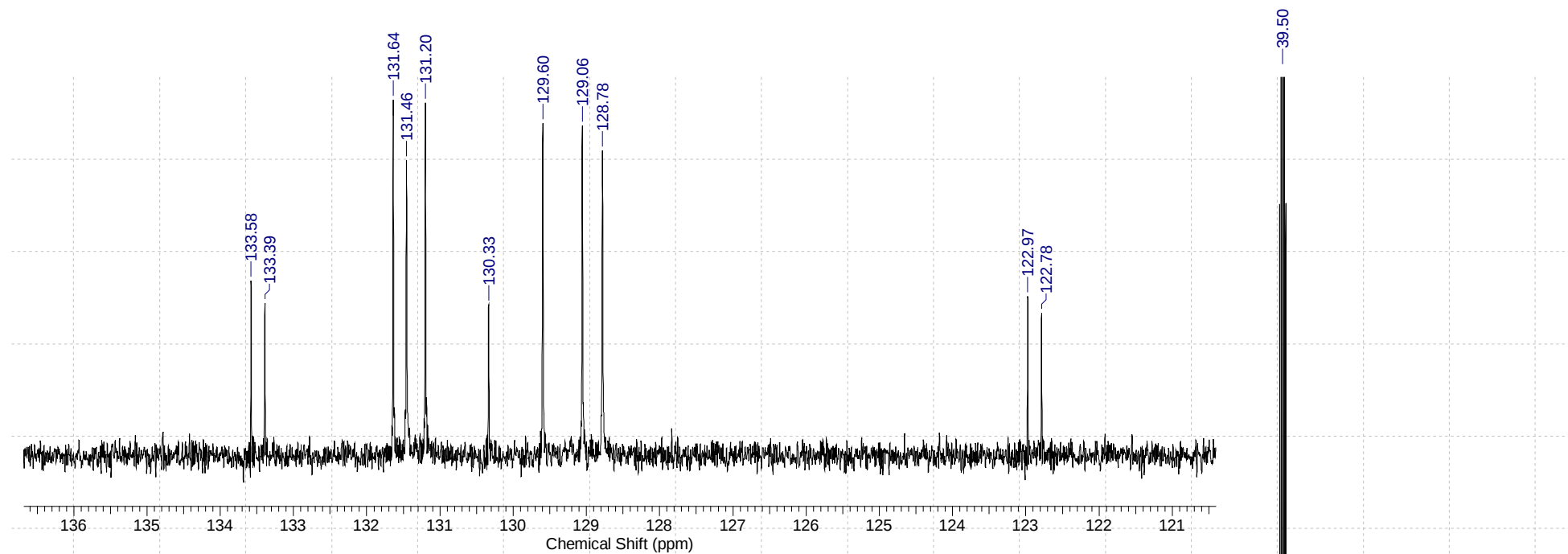
^{13}C NMR spectrum of compound 5f (DMSO- d_6).



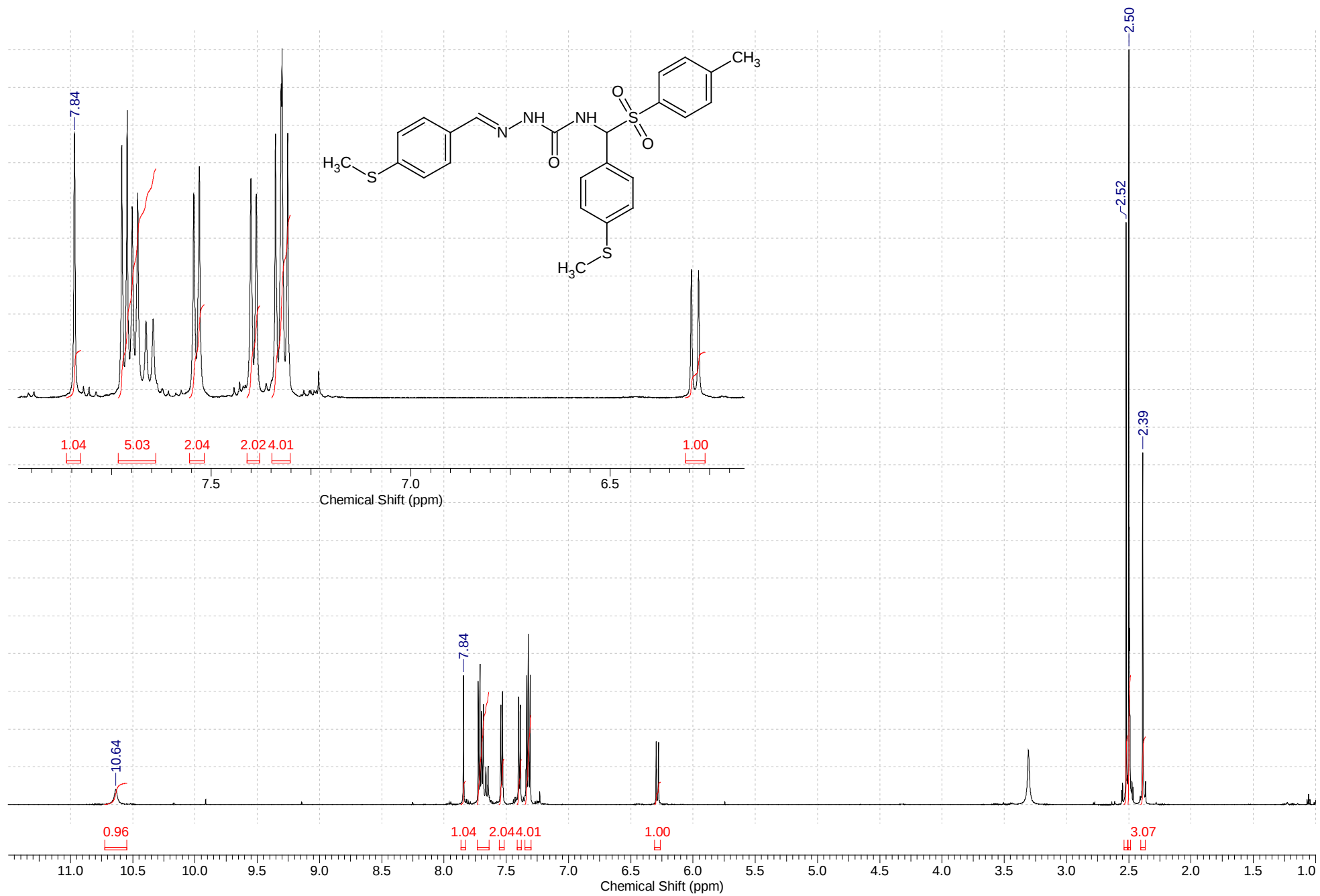
^1H NMR spectrum of compound **5g** ($\text{DMSO}-d_6$).



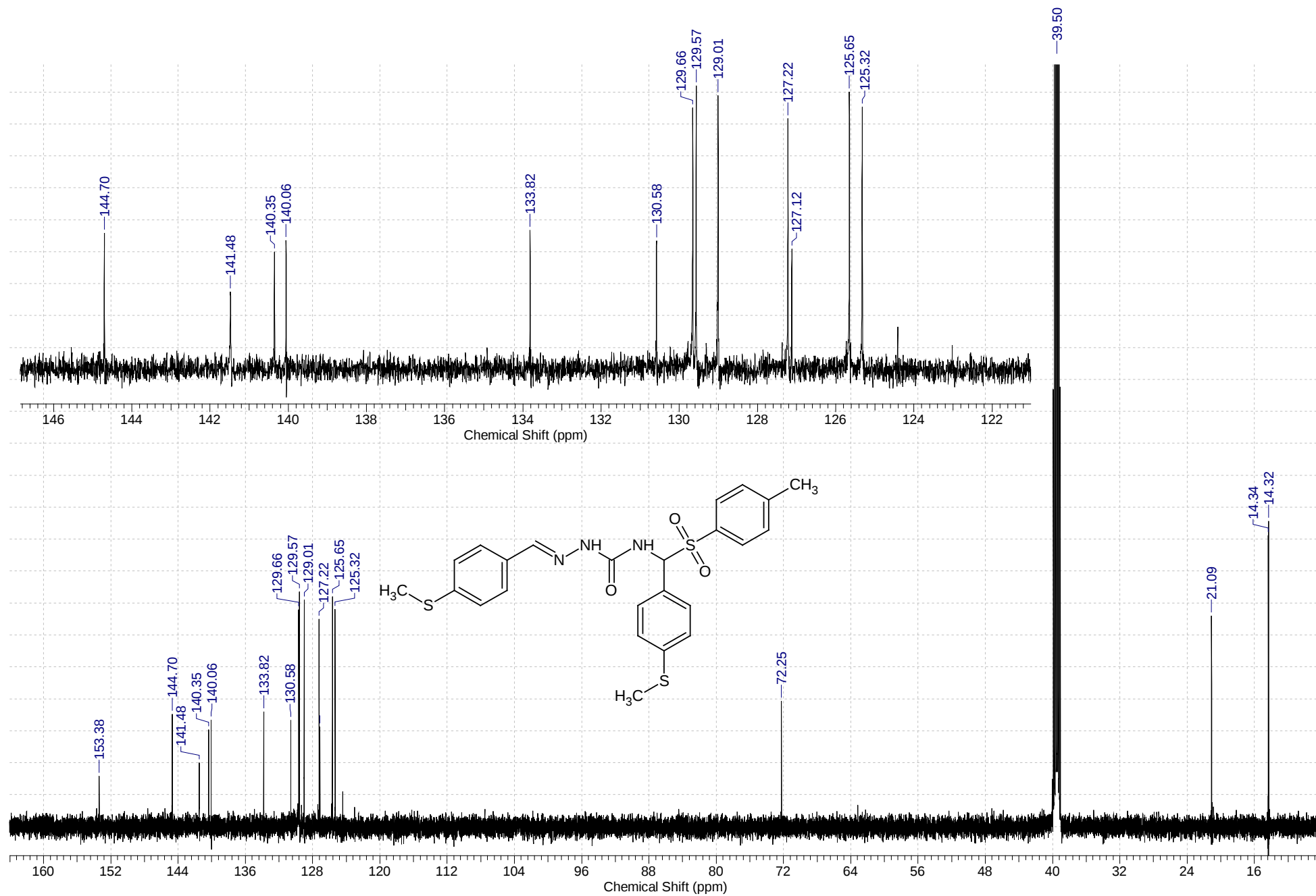
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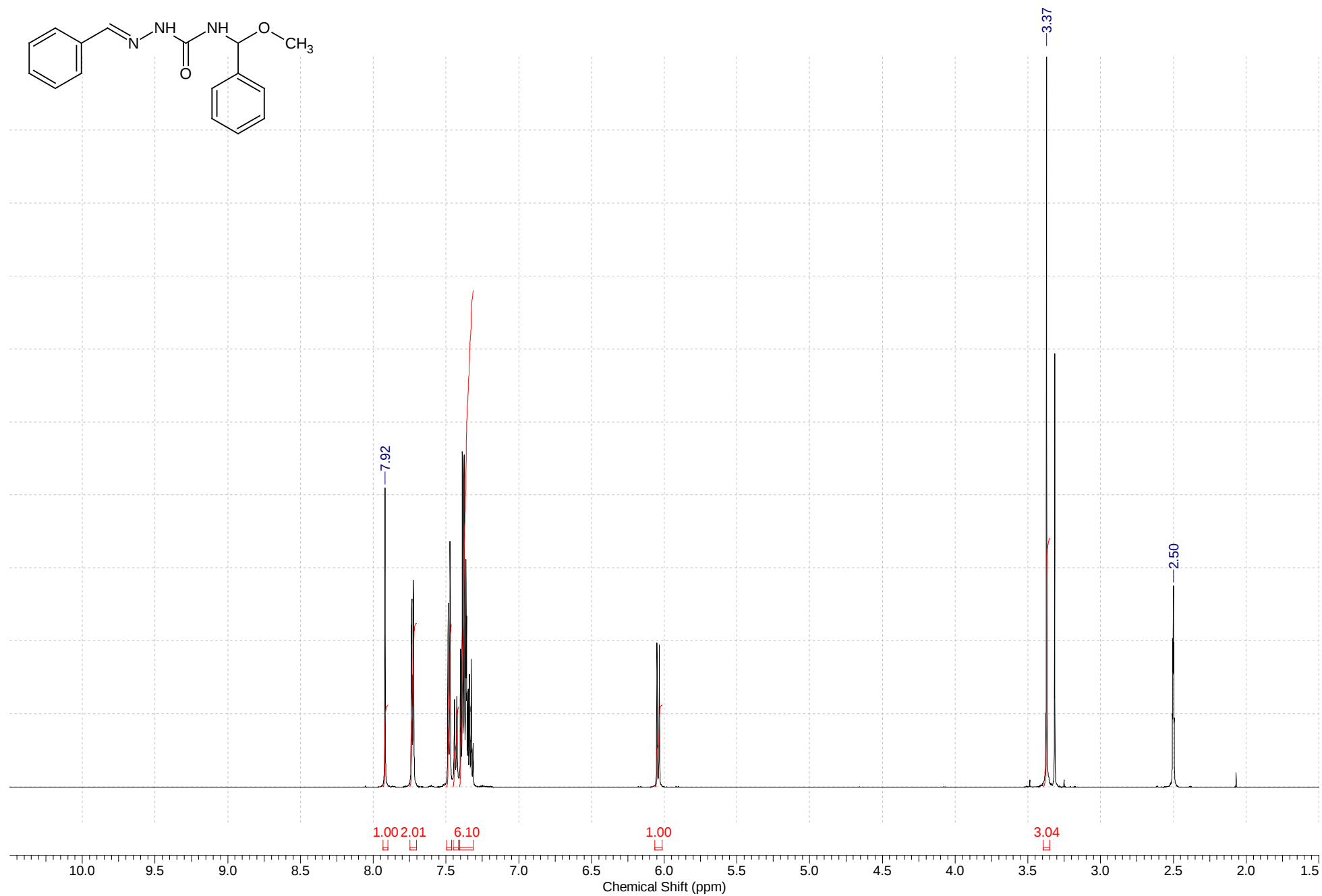
^1H NMR spectrum of compound **5h** ($\text{DMSO}-d_6$).



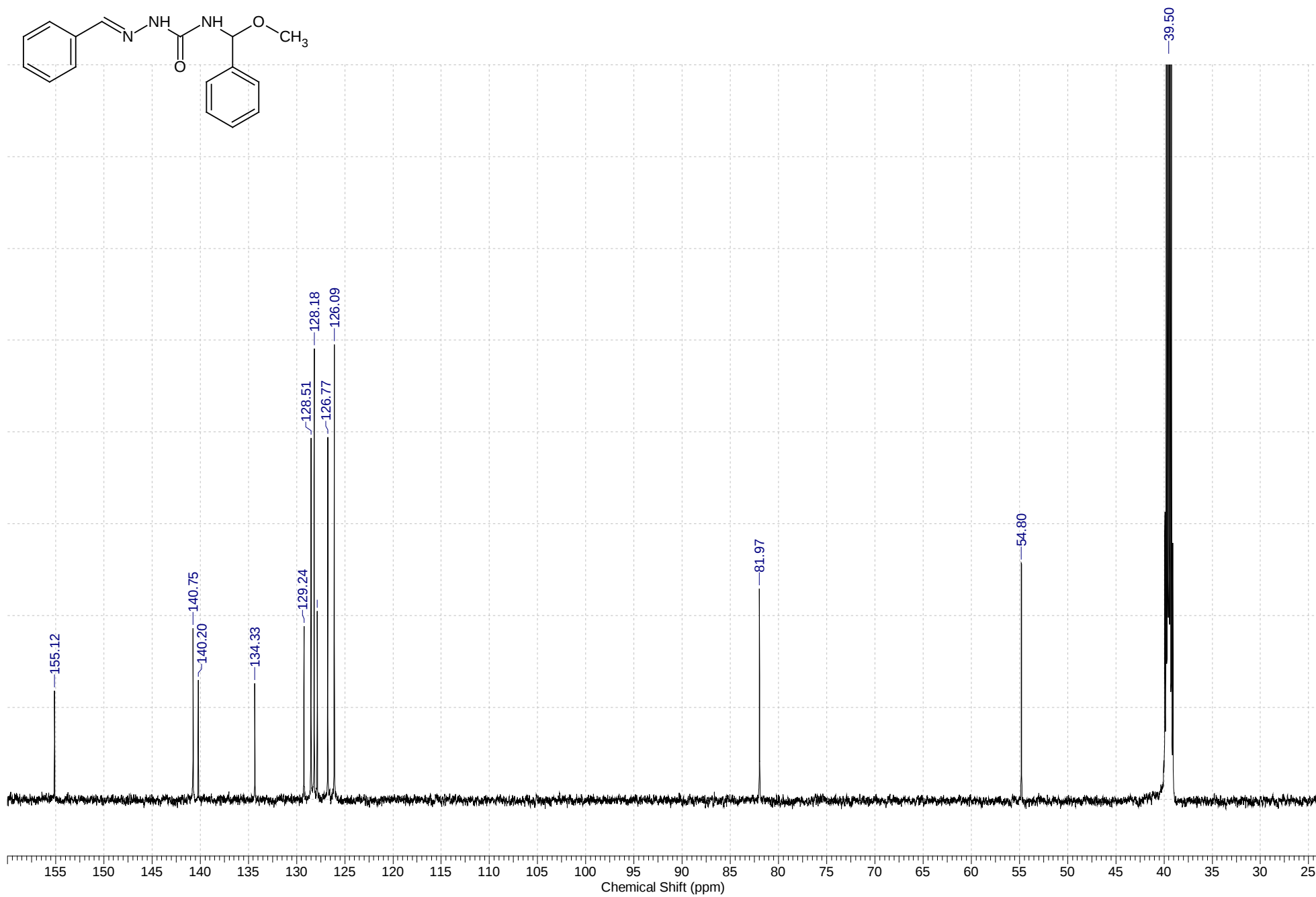
^{13}C NMR spectrum of compound **5h** (DMSO- d_6).



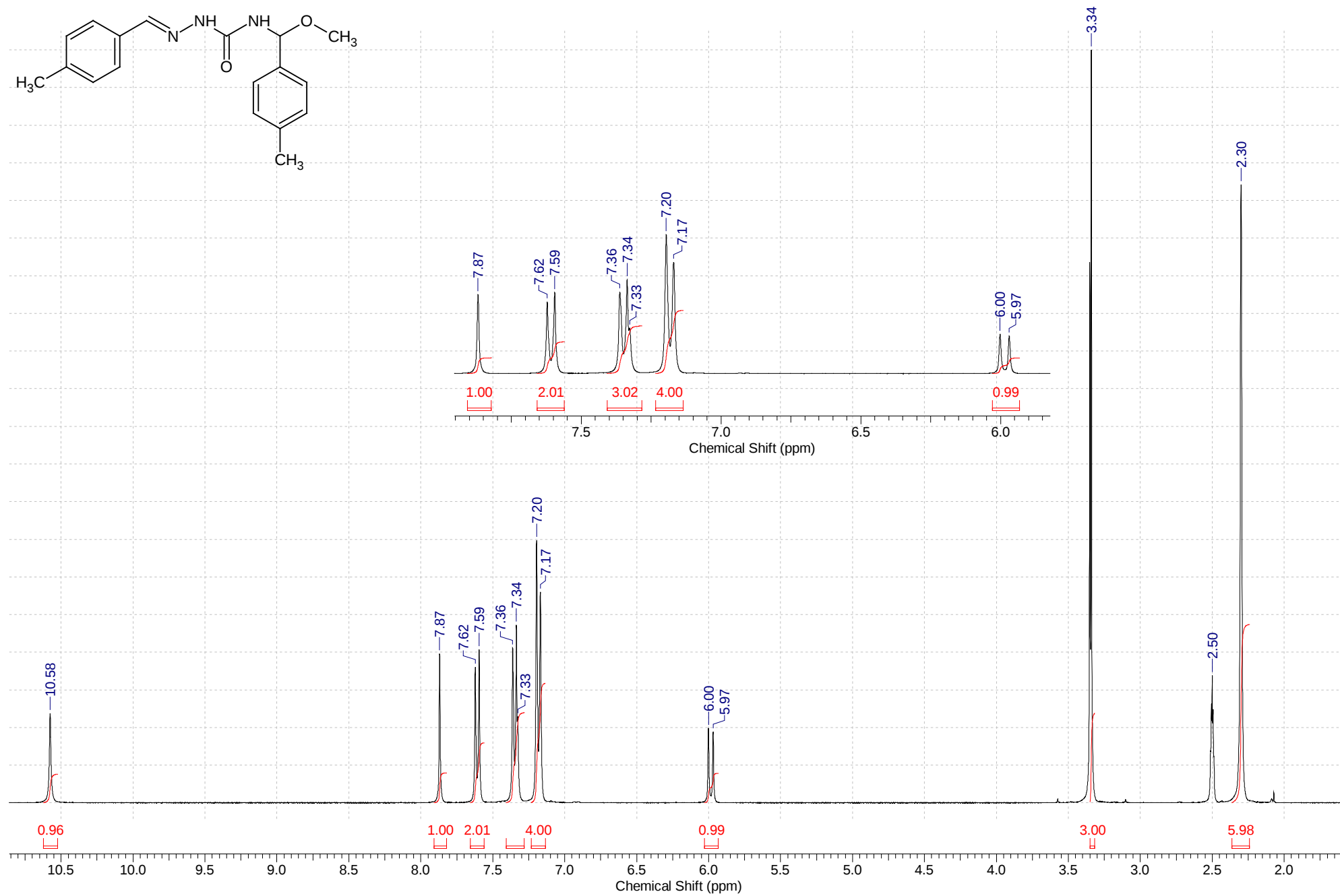
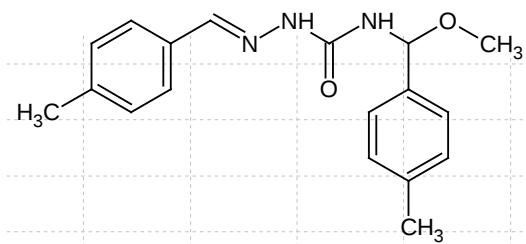
¹H NMR spectrum of compound **9a** (DMSO-*d*₆).



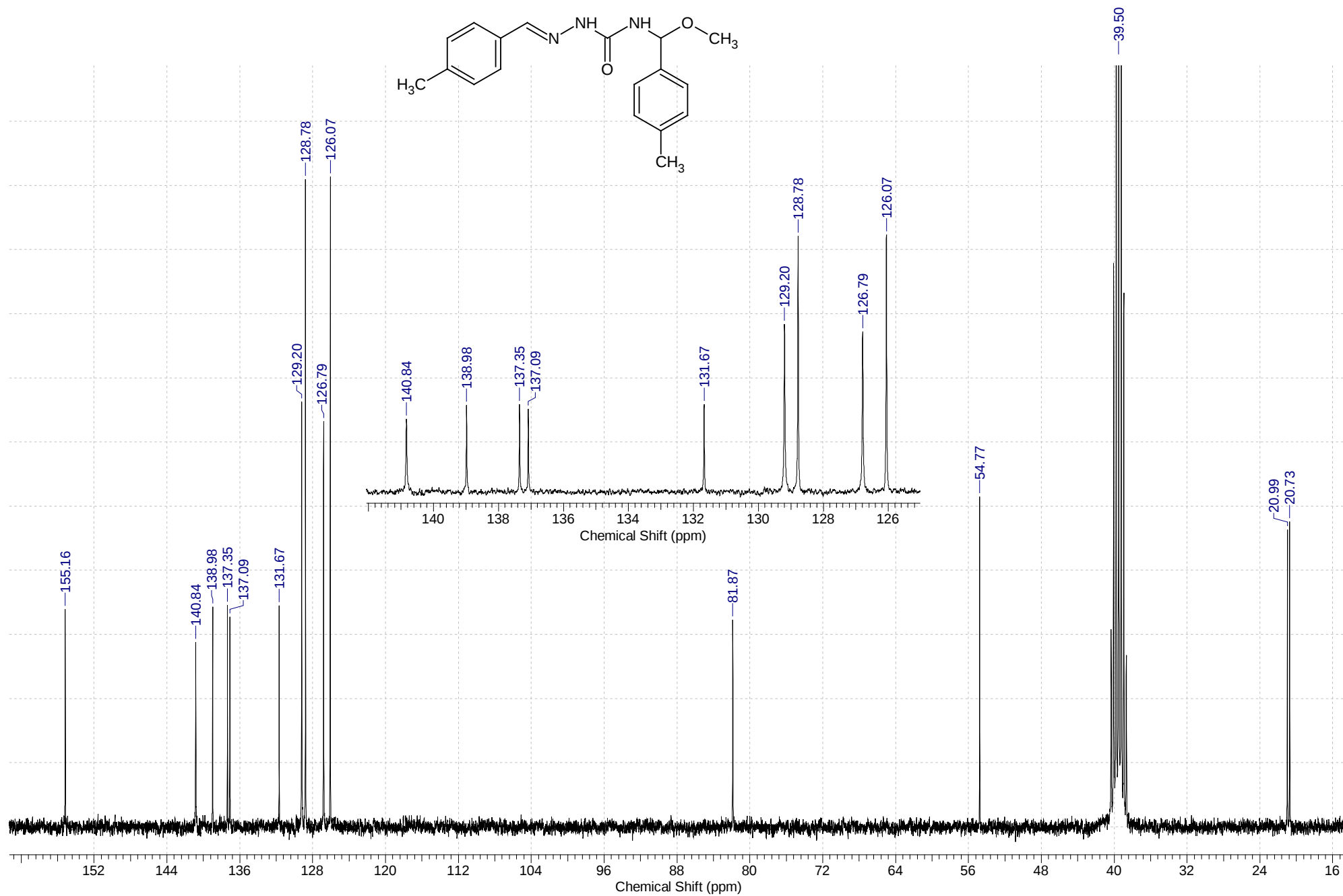
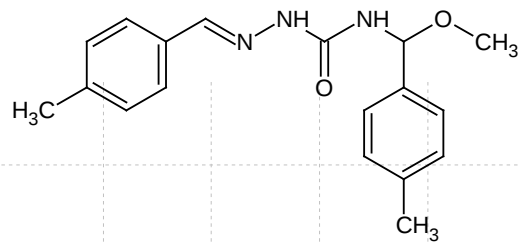
^{13}C NMR spectrum of compound **9a** ($\text{DMSO-}d_6$).



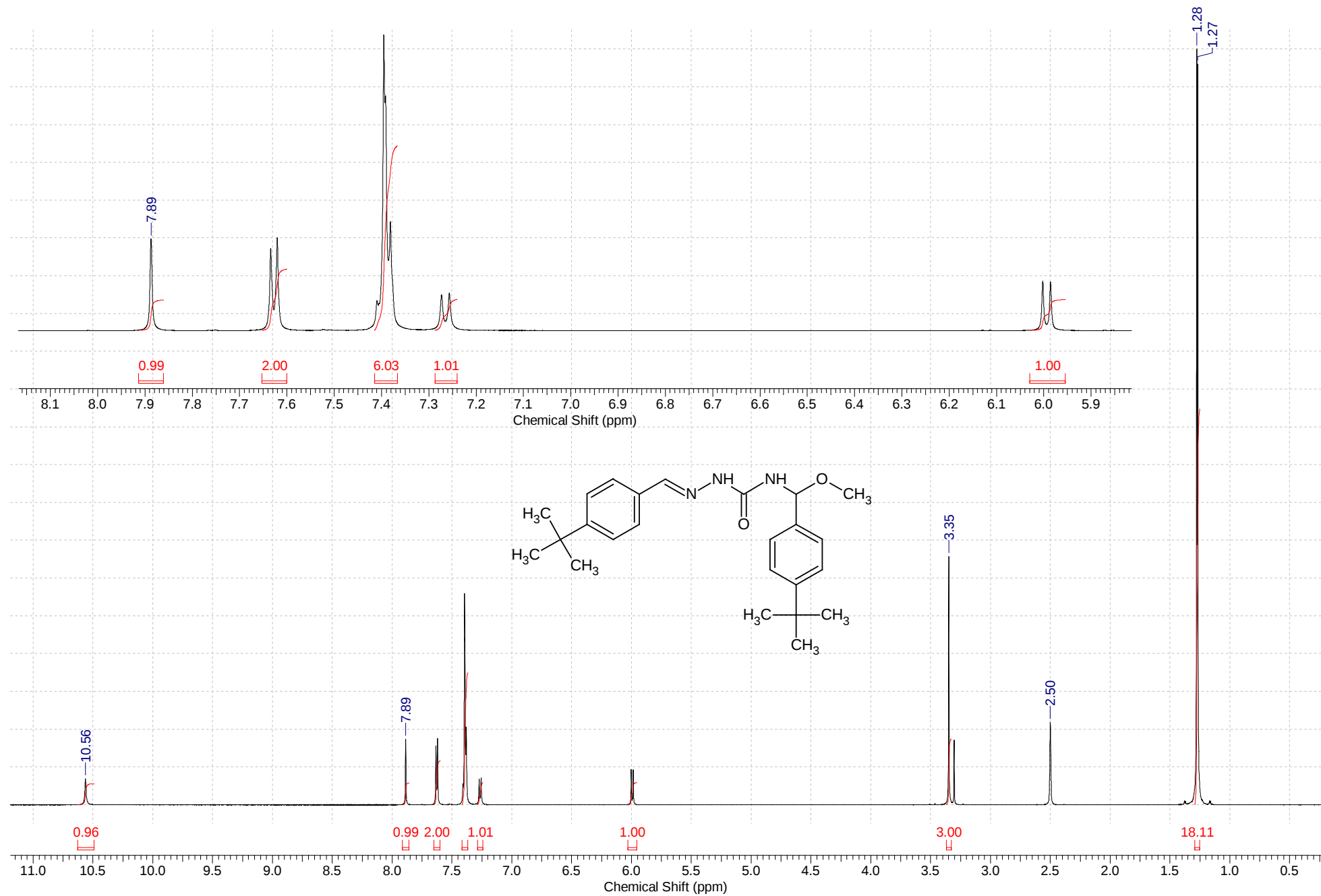
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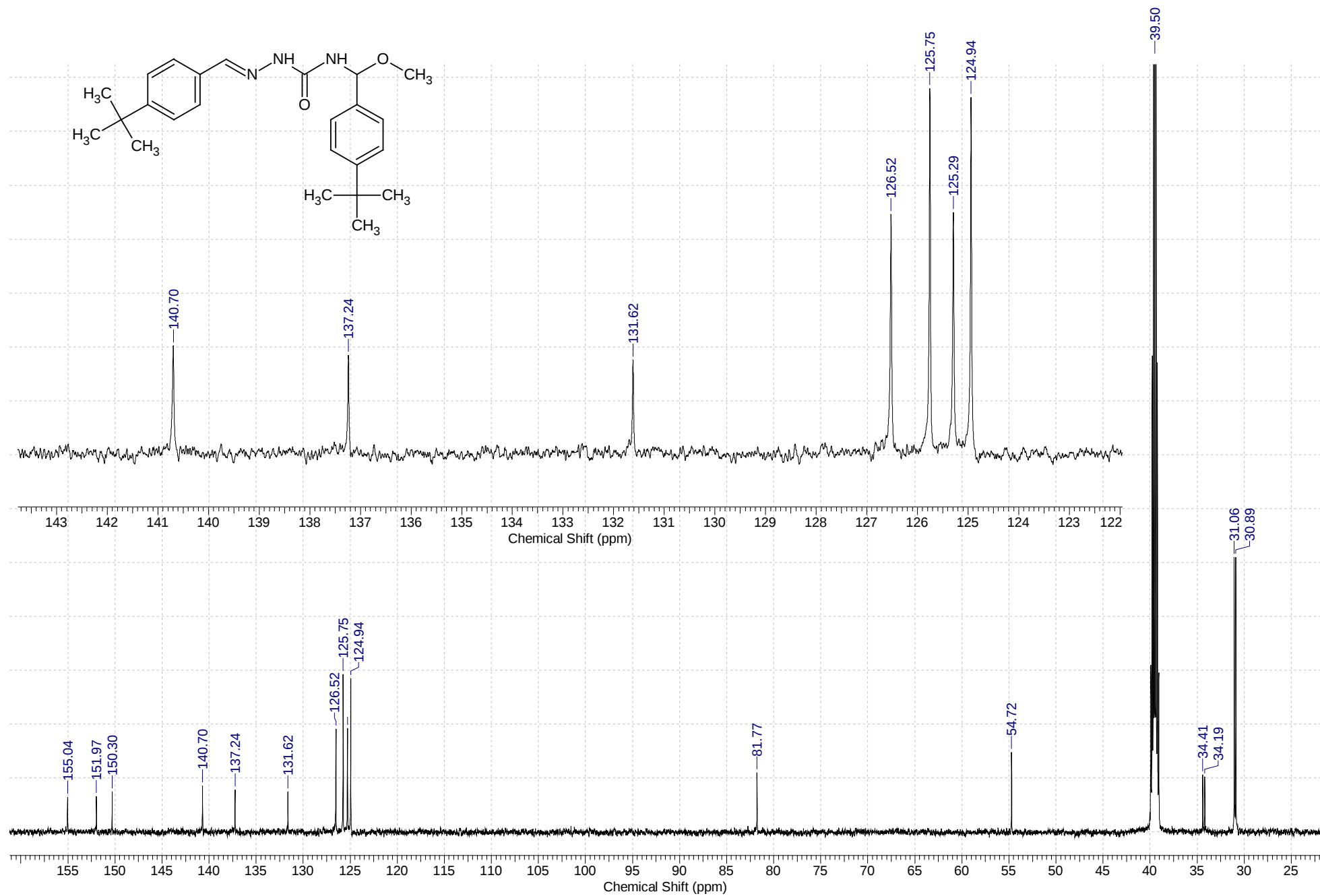
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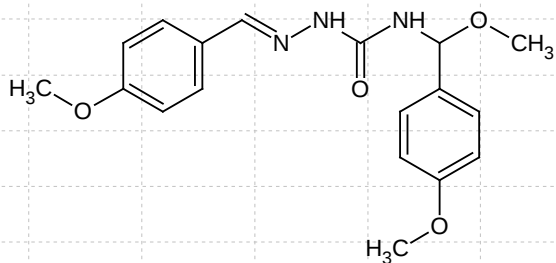
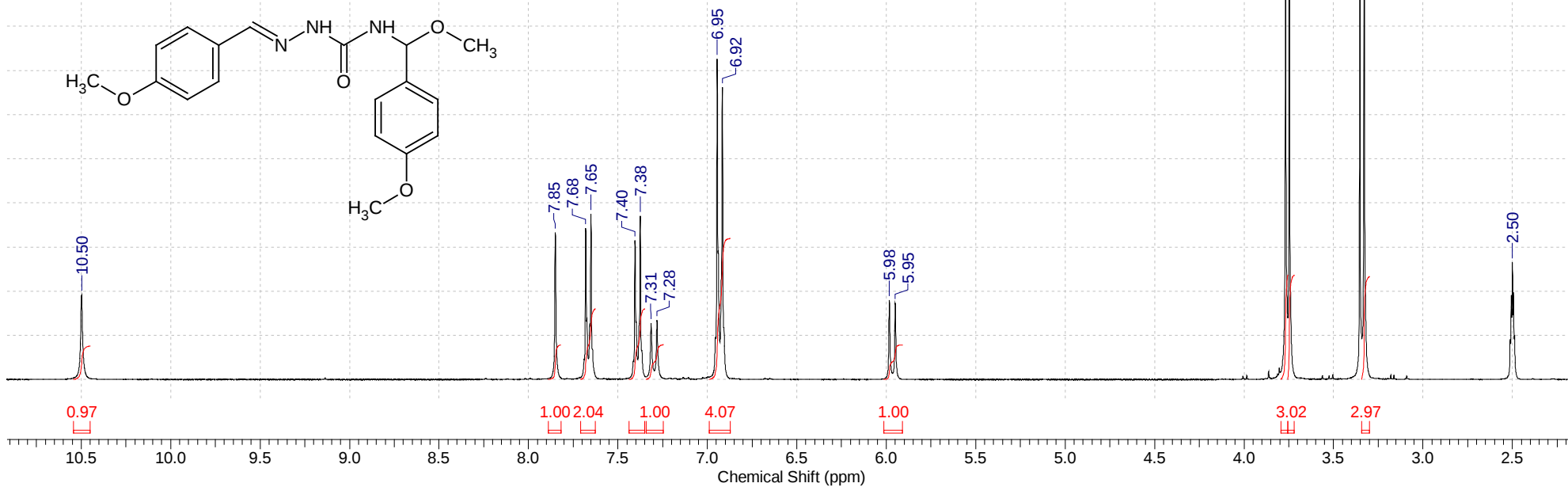
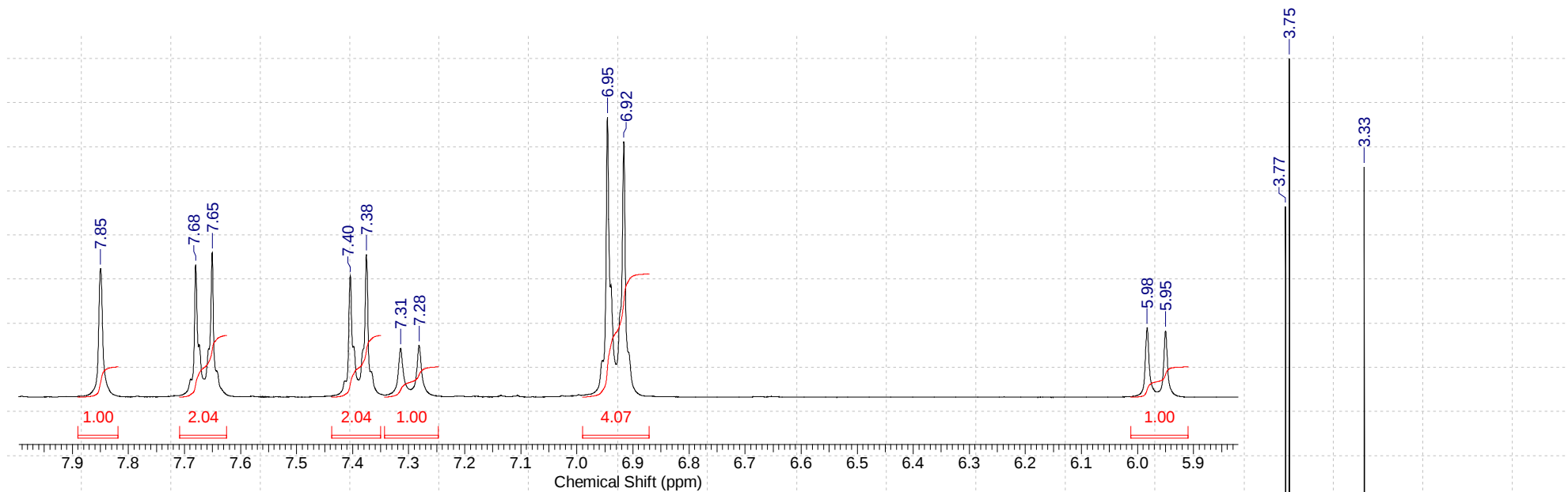
^1H NMR spectrum of compound **9c** ($\text{DMSO}-d_6$).



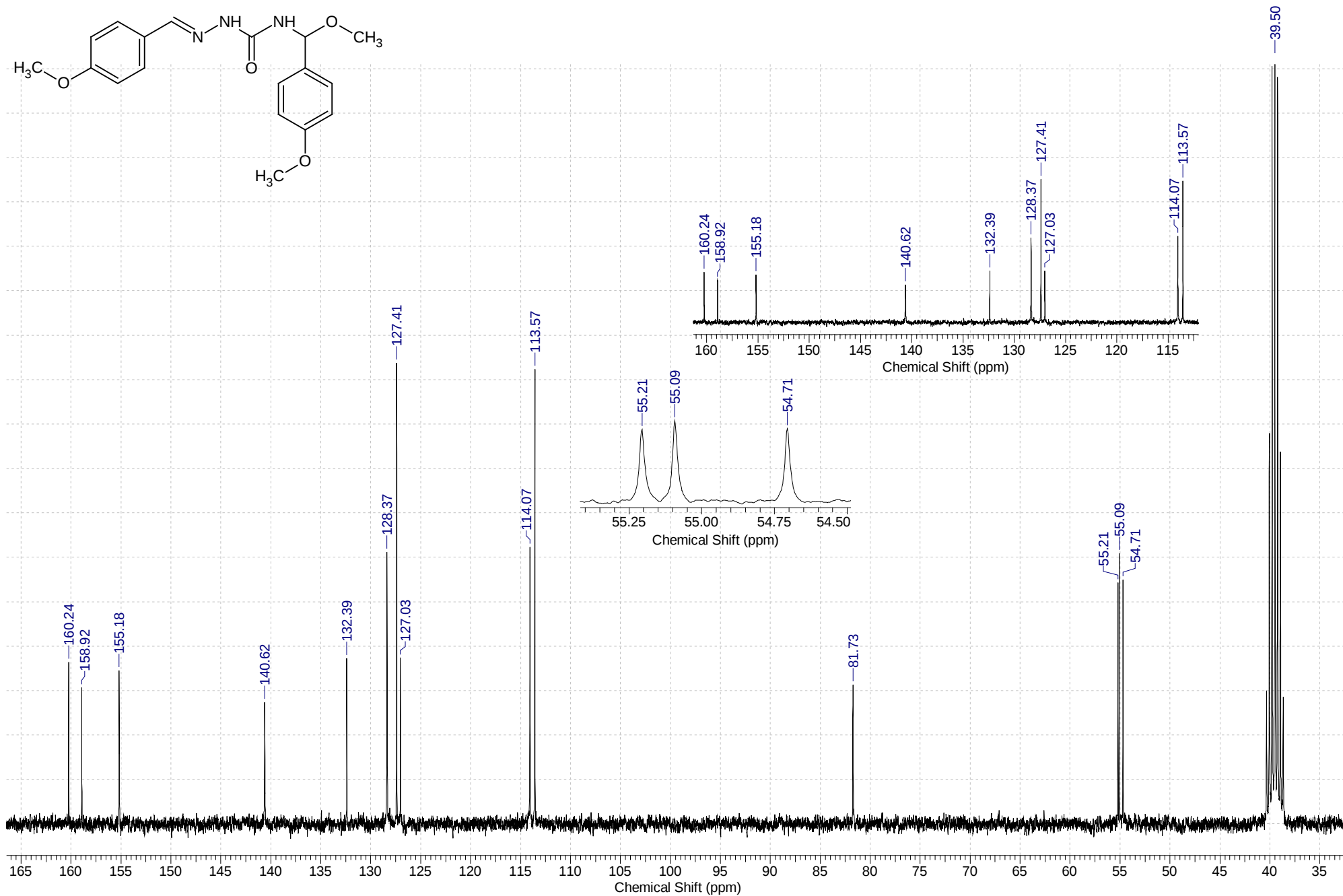
^{13}C NMR spectrum of compound **9c** ($\text{DMSO-}d_6$).



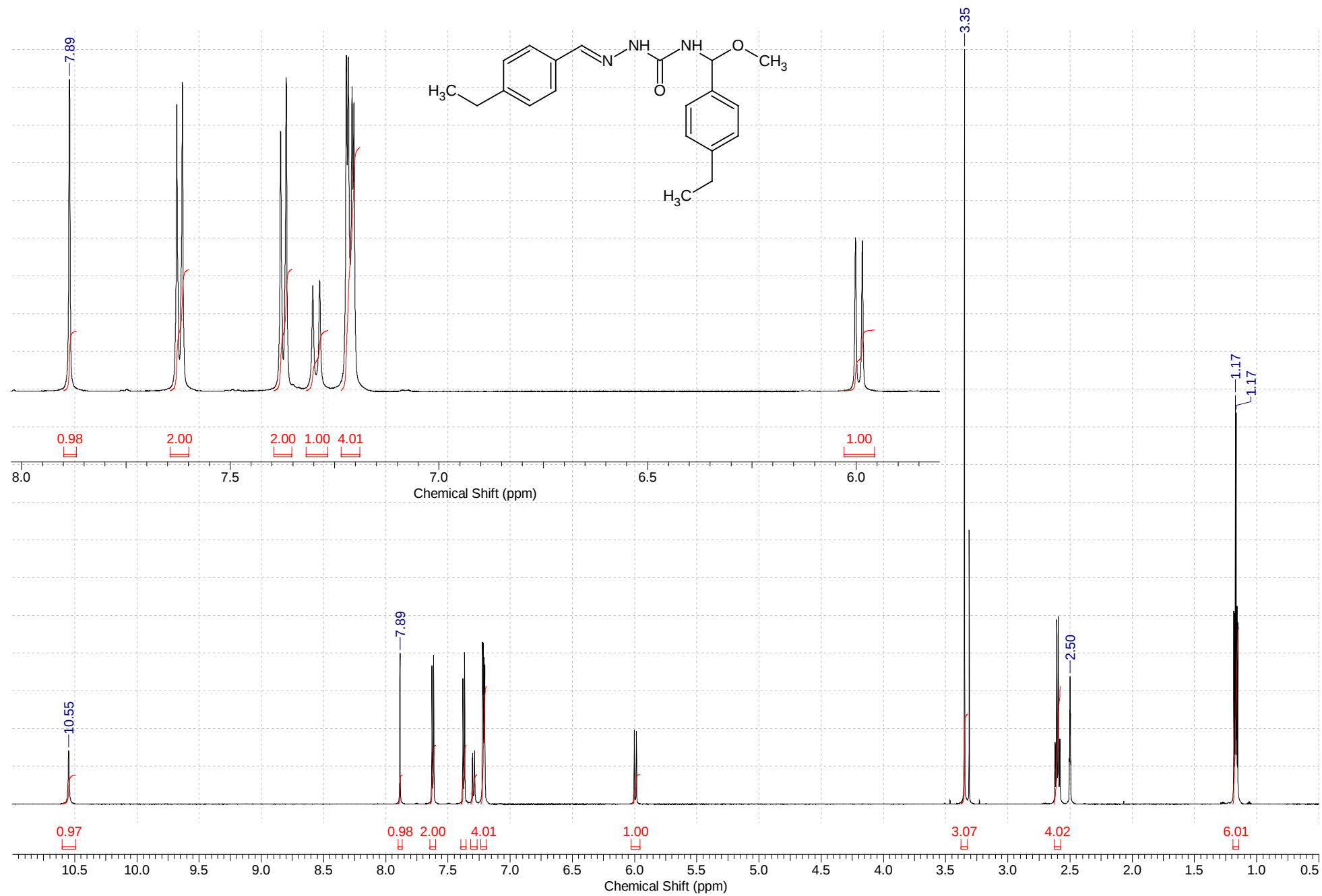
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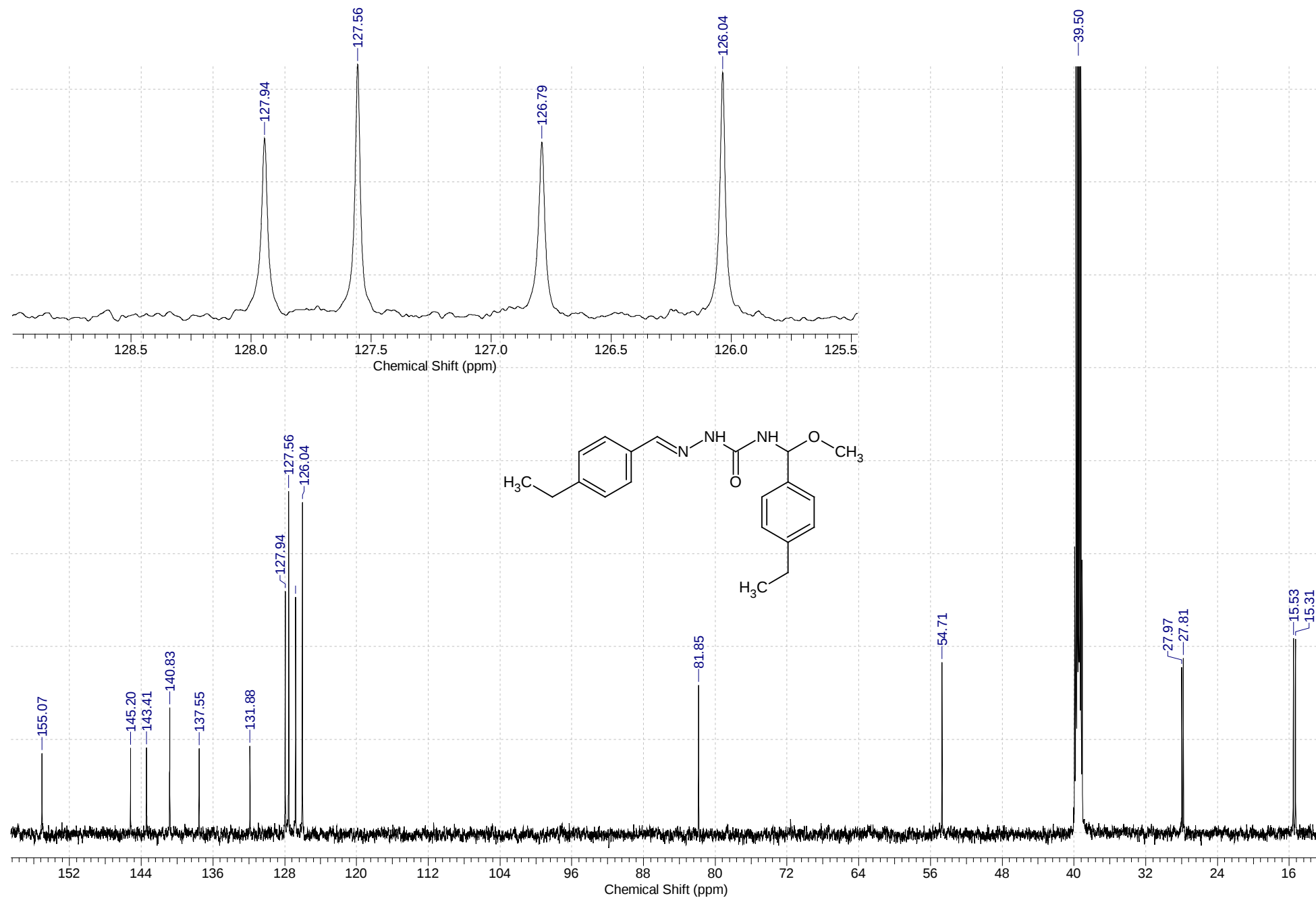
^{13}C NMR spectrum of compound **9d** ($\text{DMSO-}d_6$).



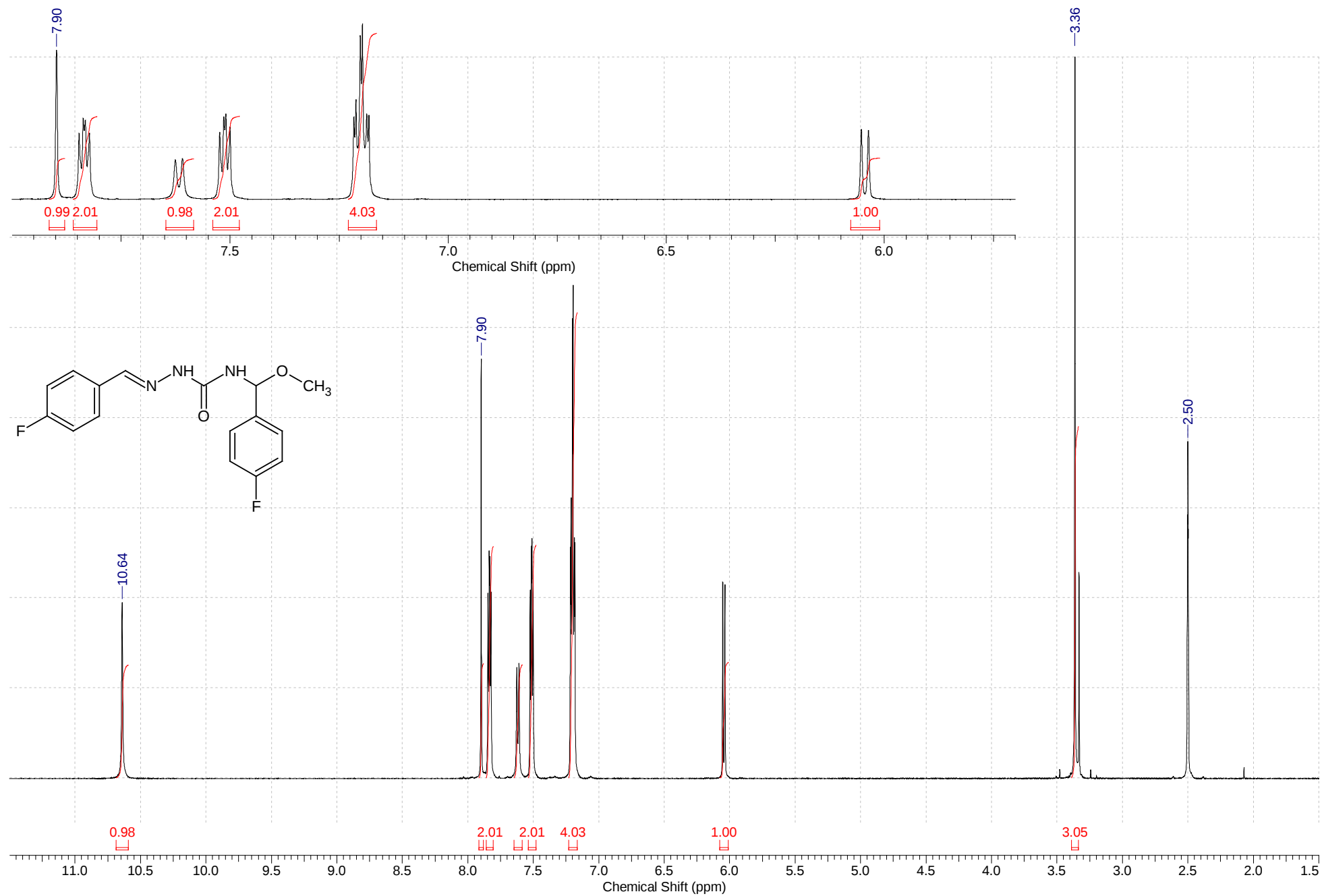
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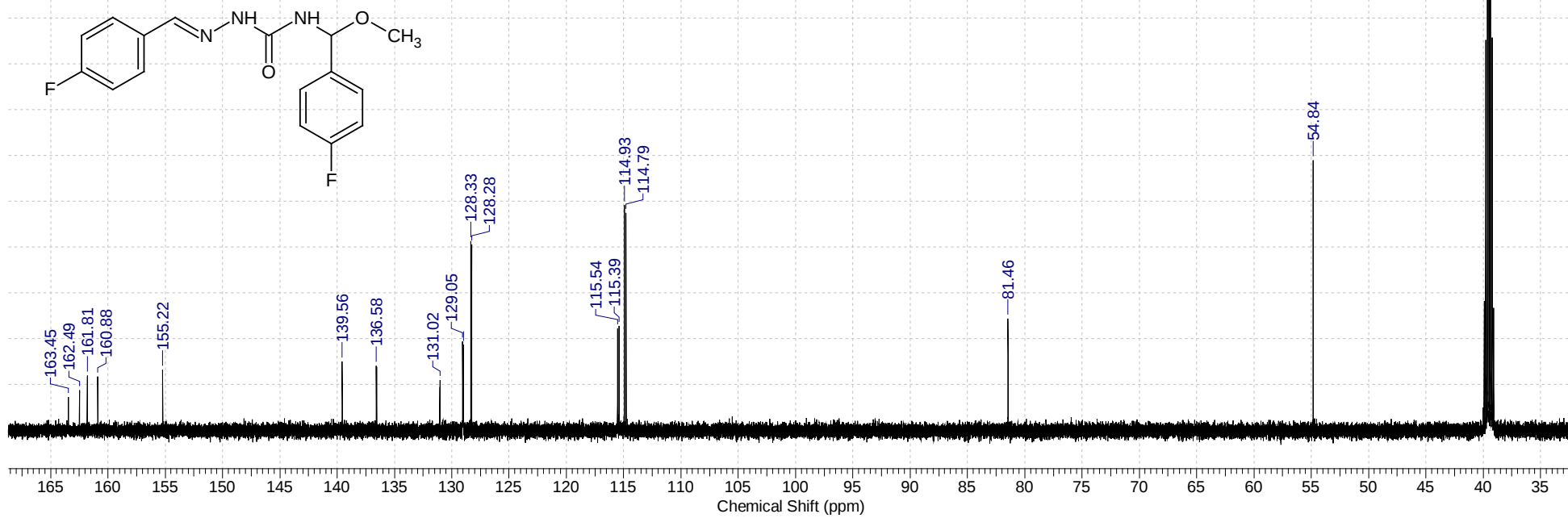
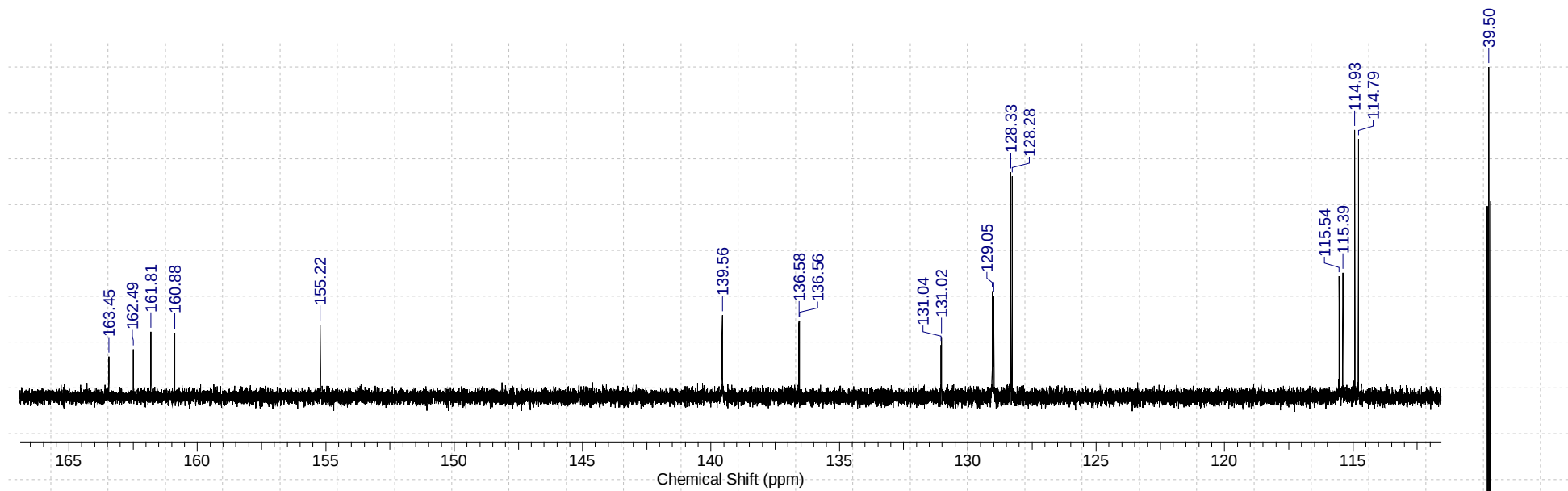
^{13}C NMR spectrum of compound **9e** (DMSO- d_6).



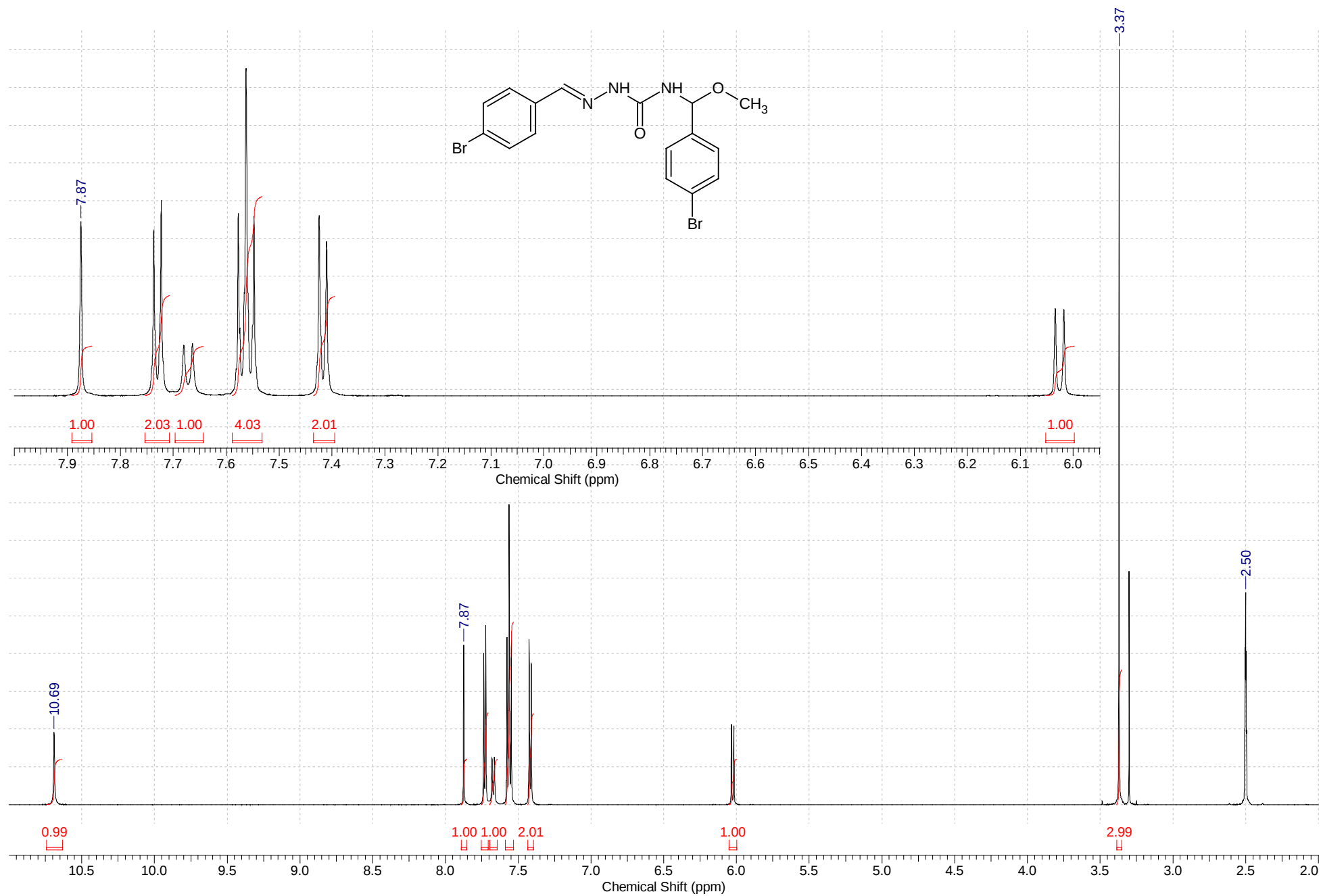
^1H NMR spectrum of compound **9f** ($\text{DMSO}-d_6$).



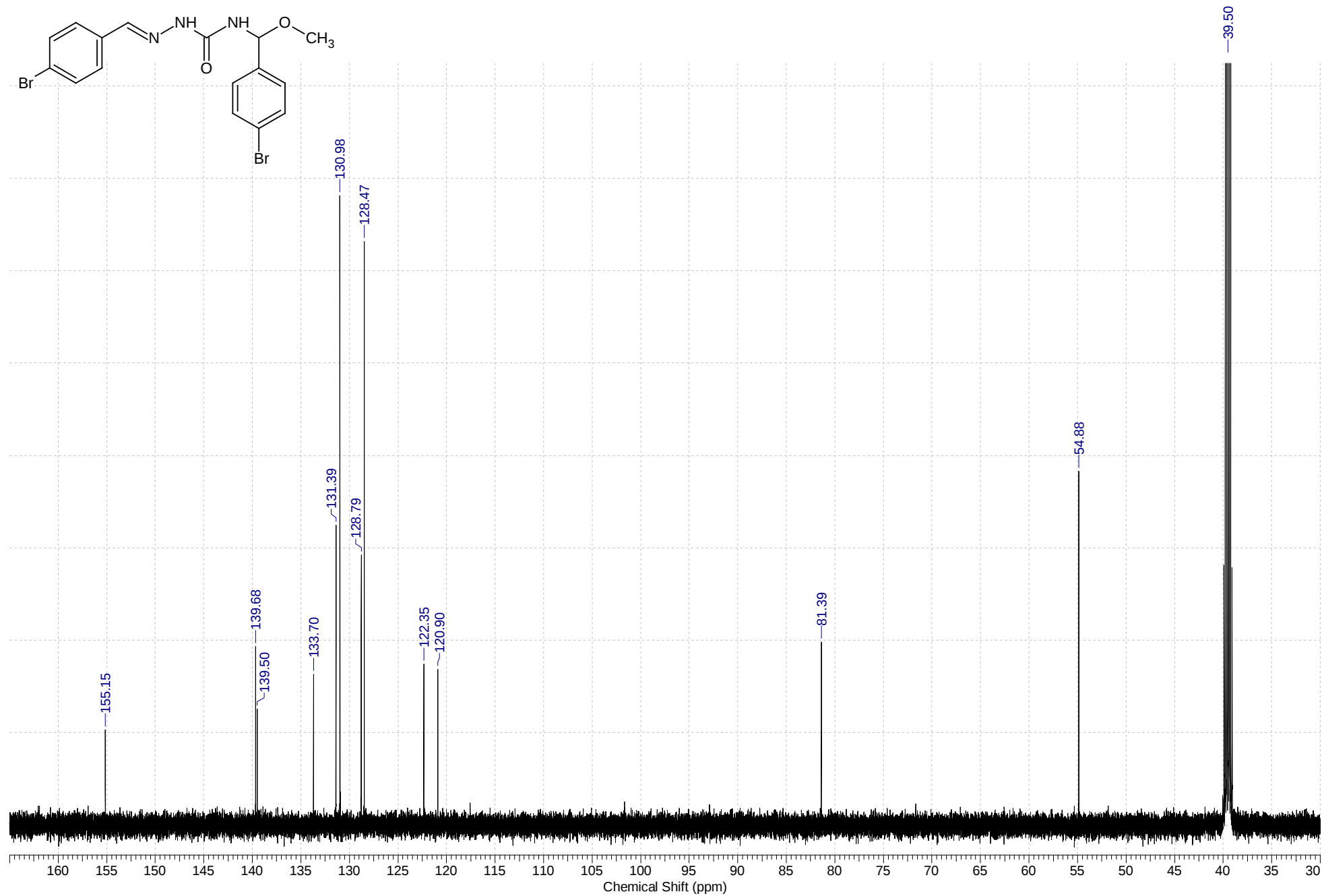
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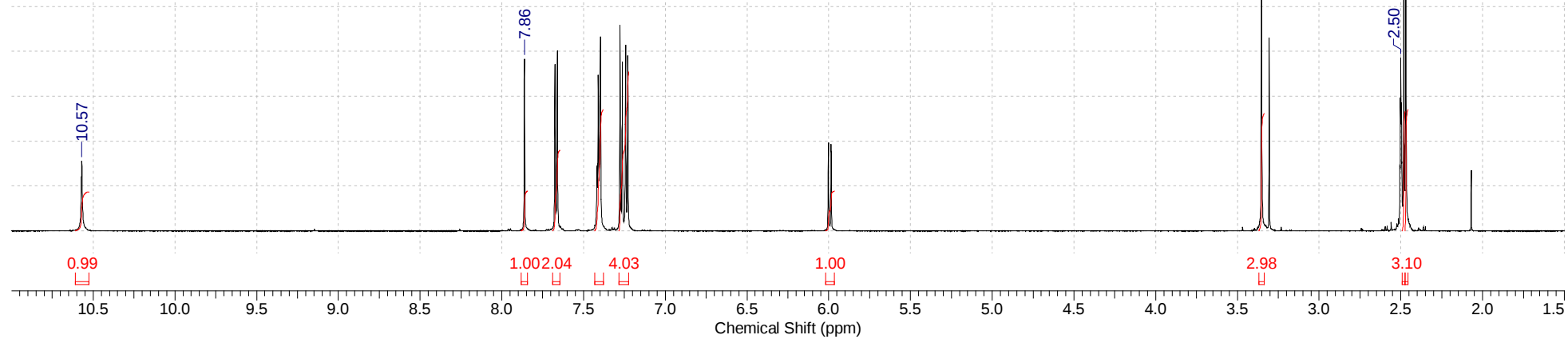
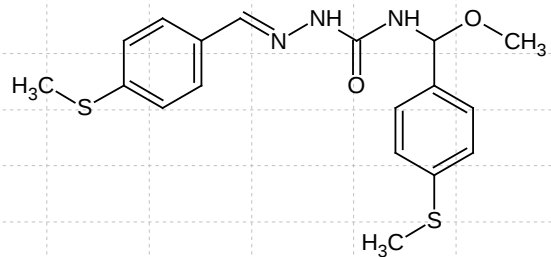
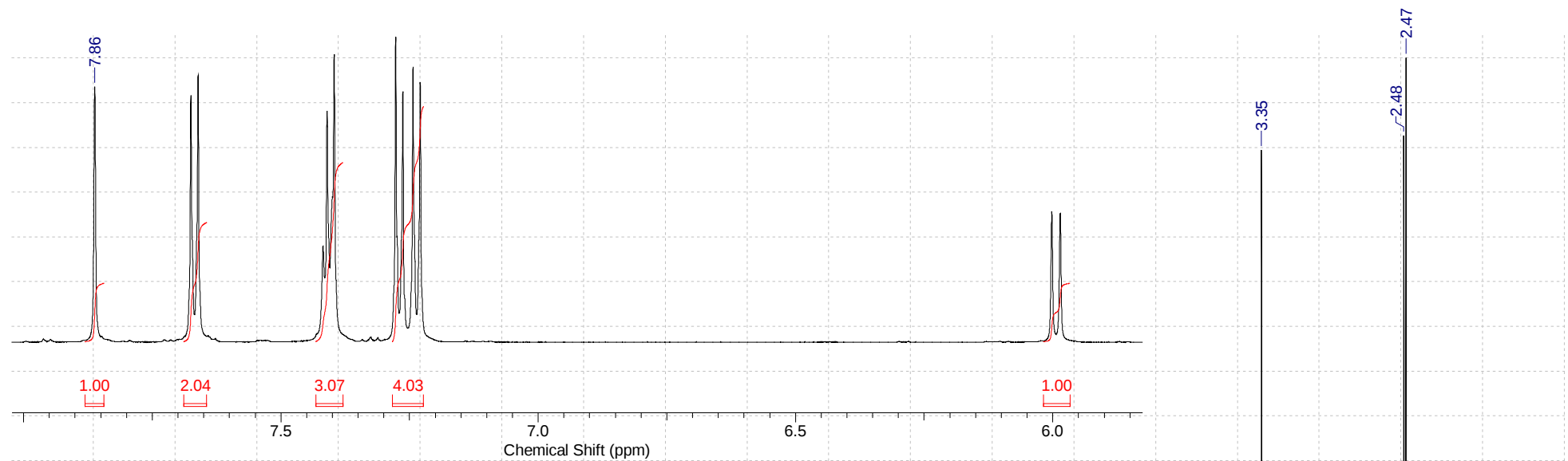
^1H NMR spectrum of compound **9g** ($\text{DMSO}-d_6$).



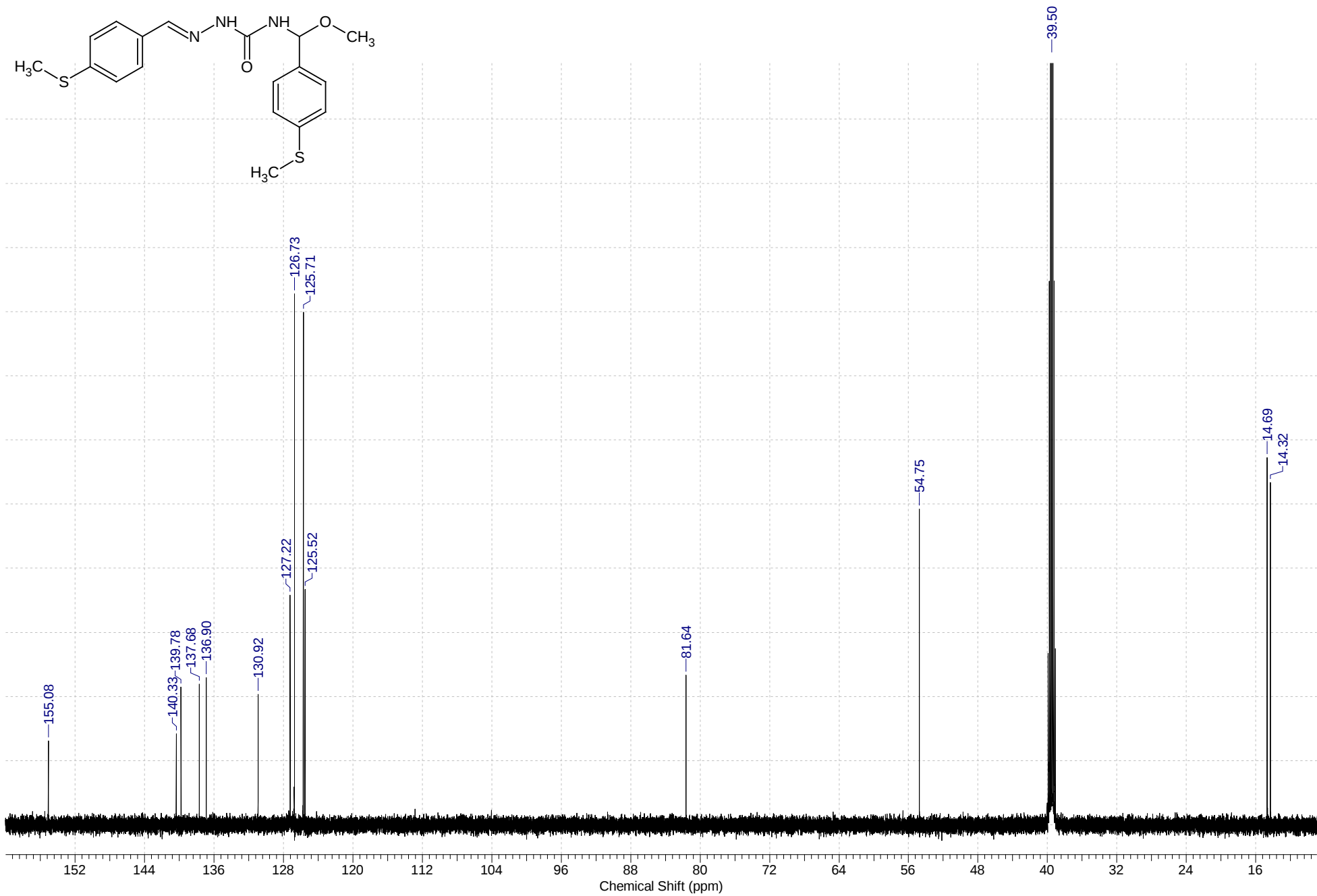
^{13}C NMR spectrum of compound **9g** ($\text{DMSO-}d_6$).



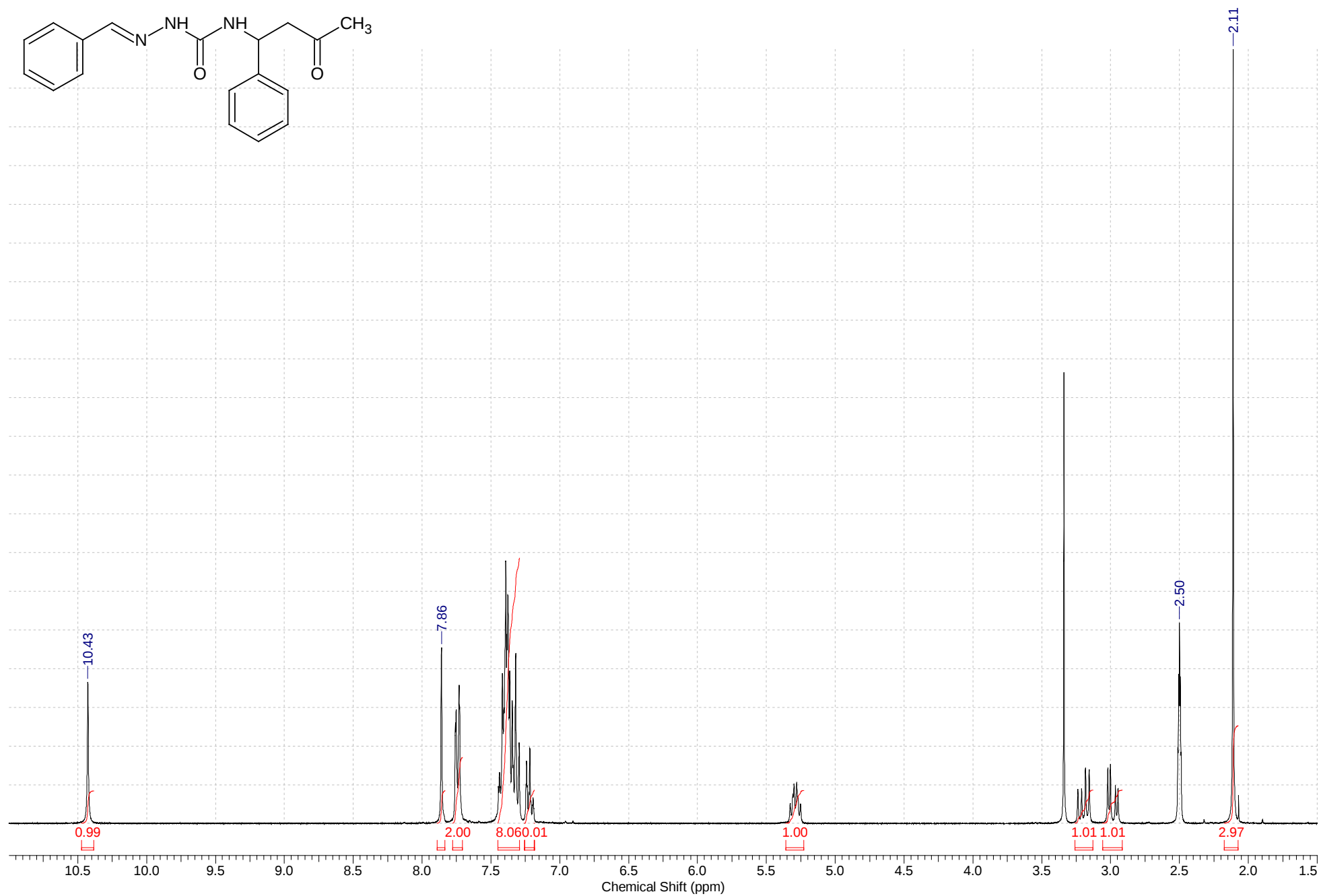
¹H NMR spectrum of compound **9h** (DMSO-*d*₆).



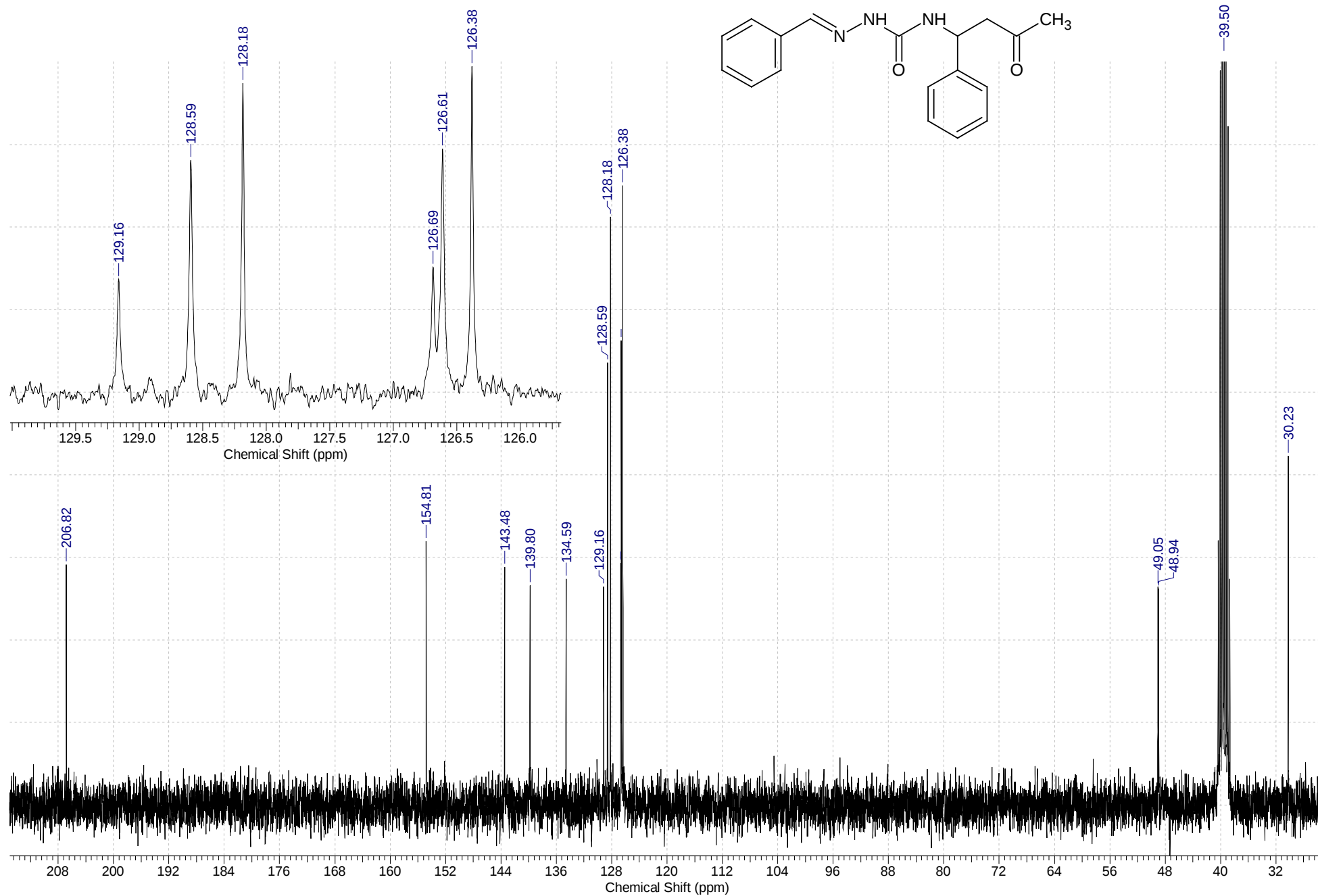
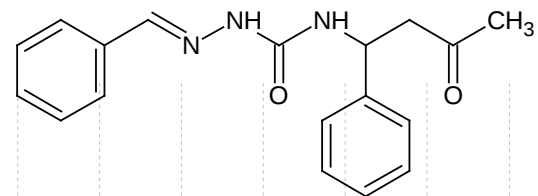
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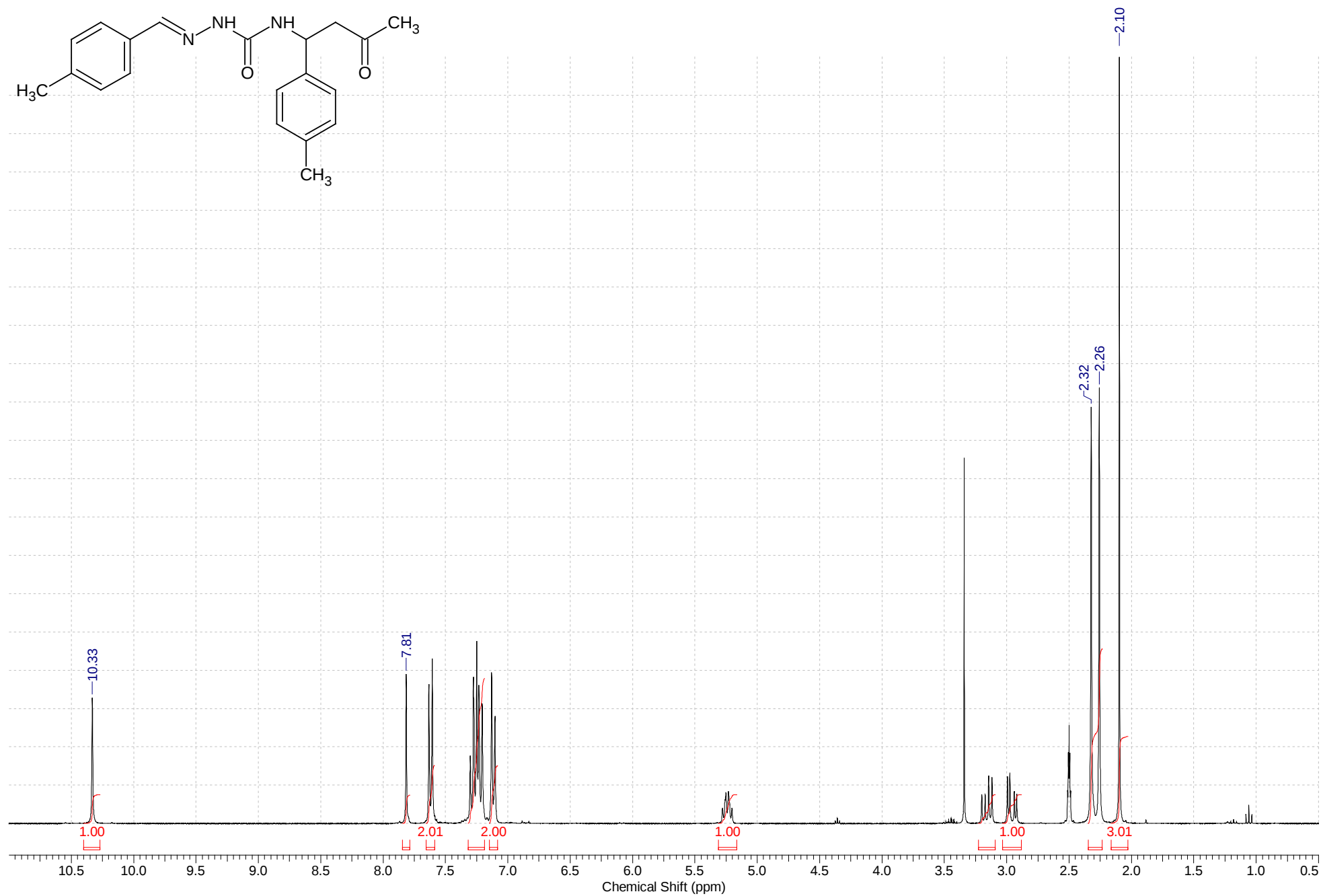
¹H NMR spectrum of compound **10a** (DMSO-*d*₆).



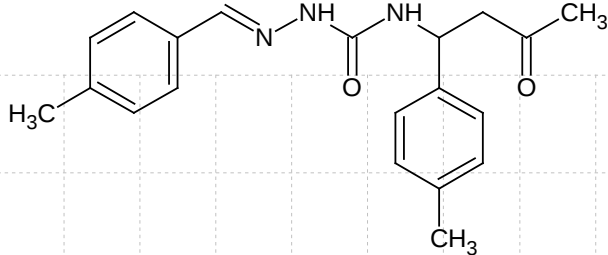
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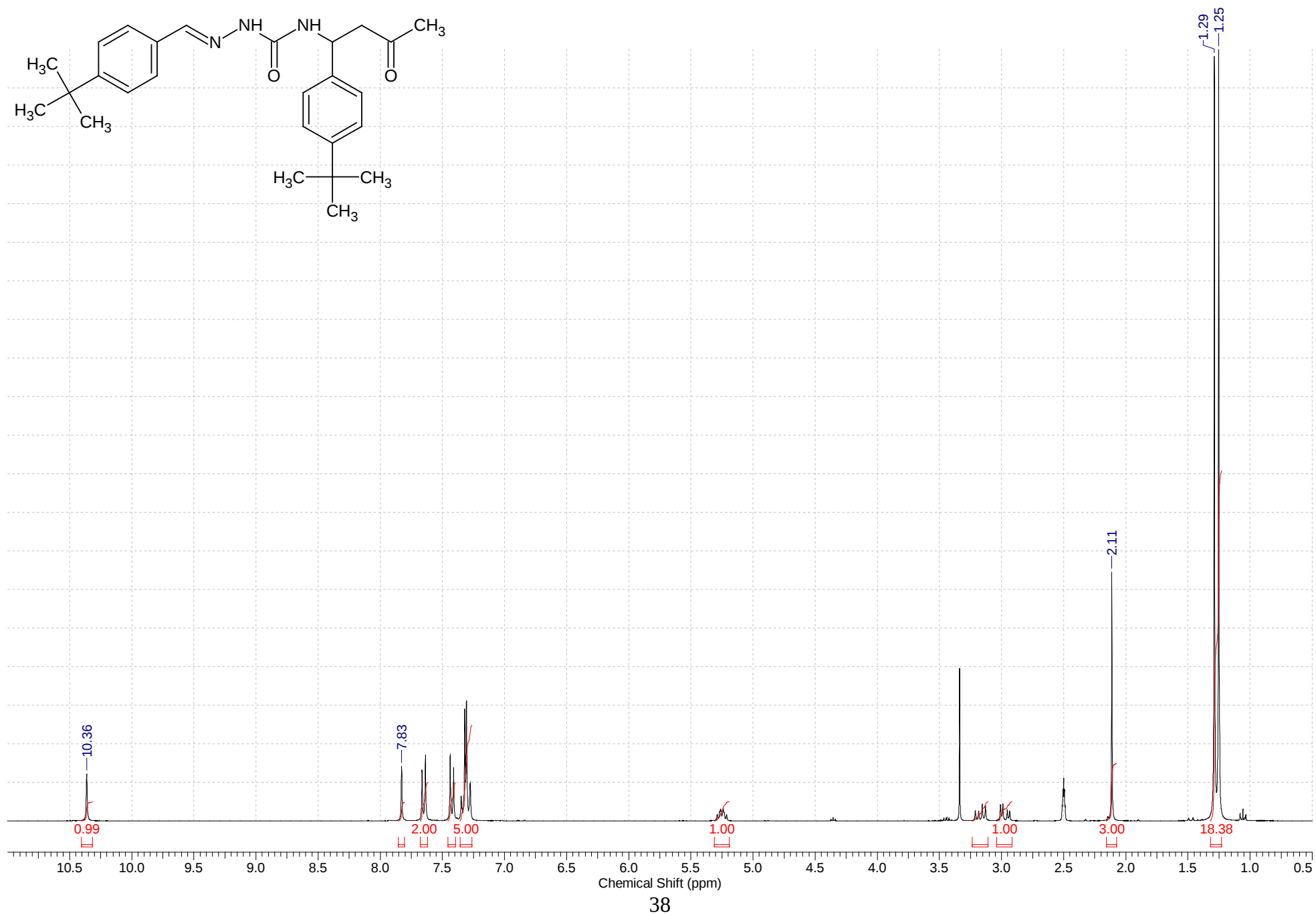
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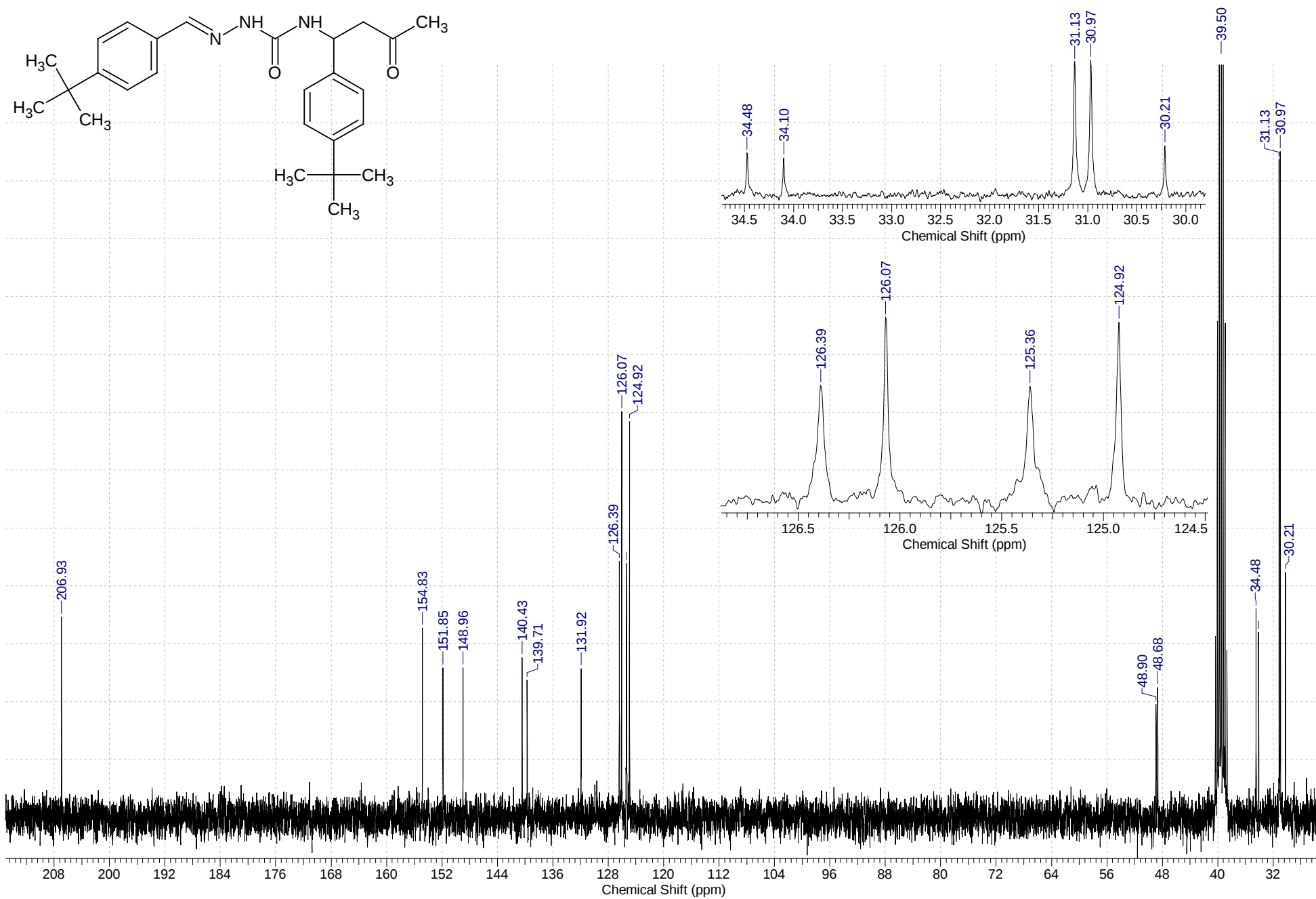
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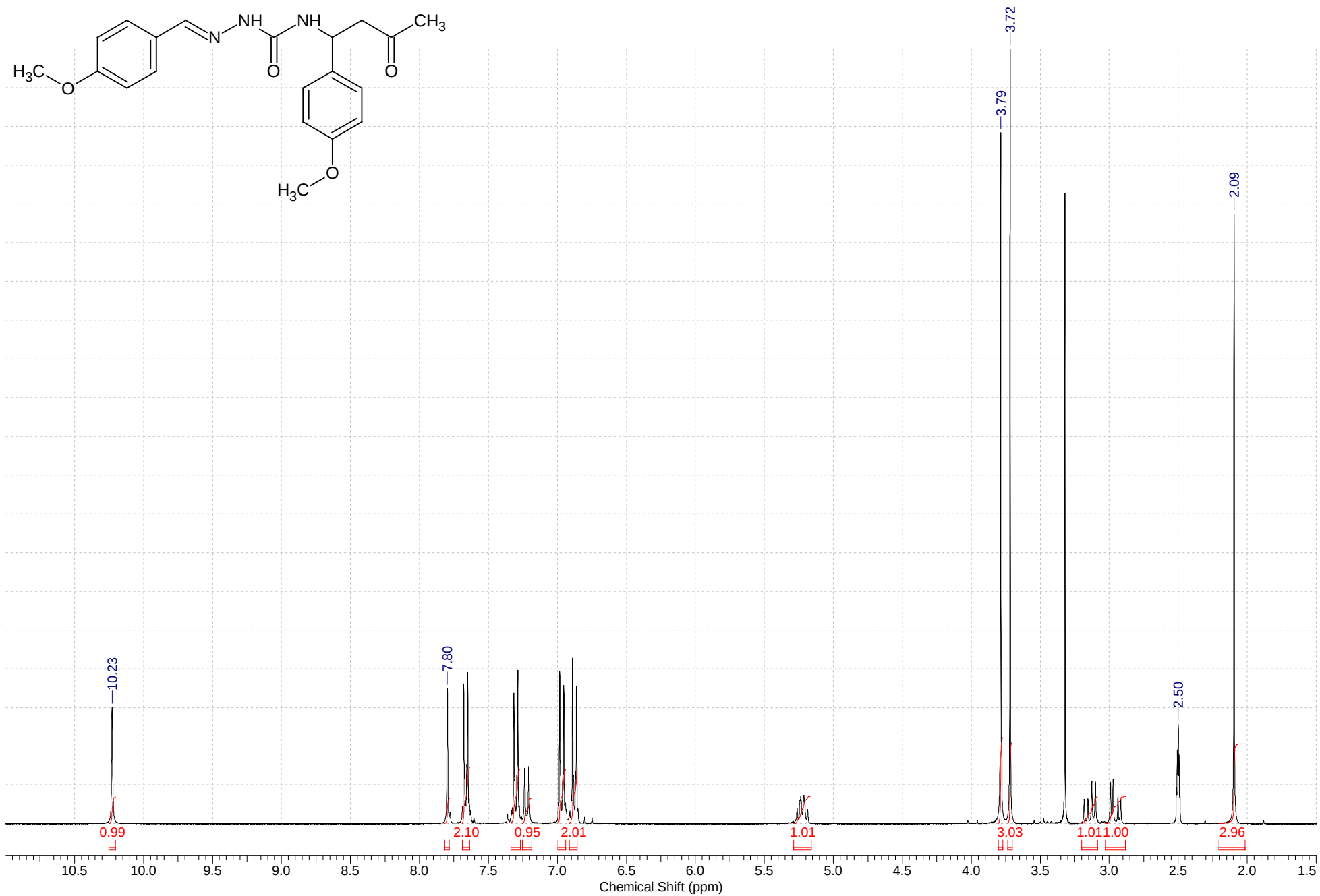
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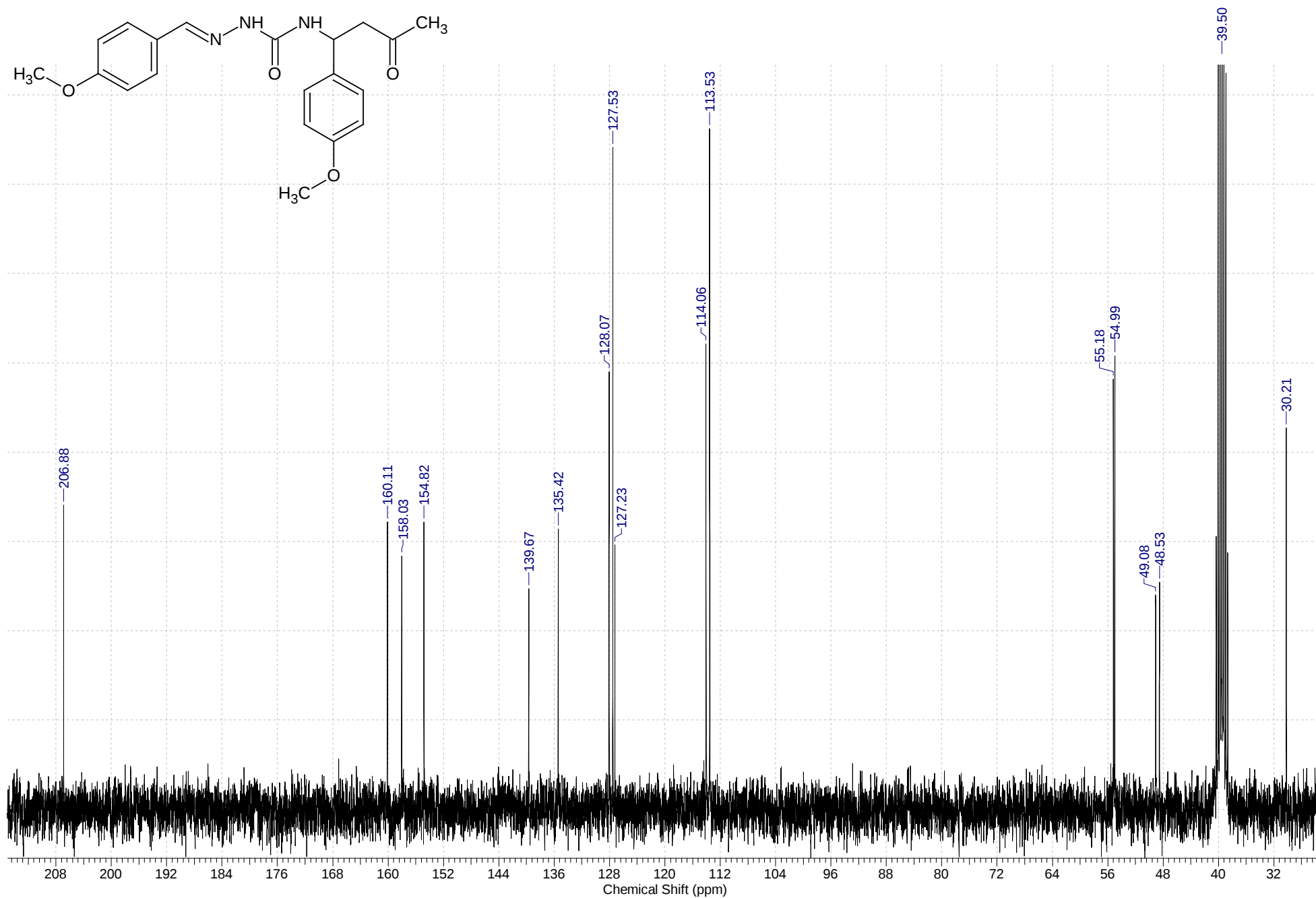
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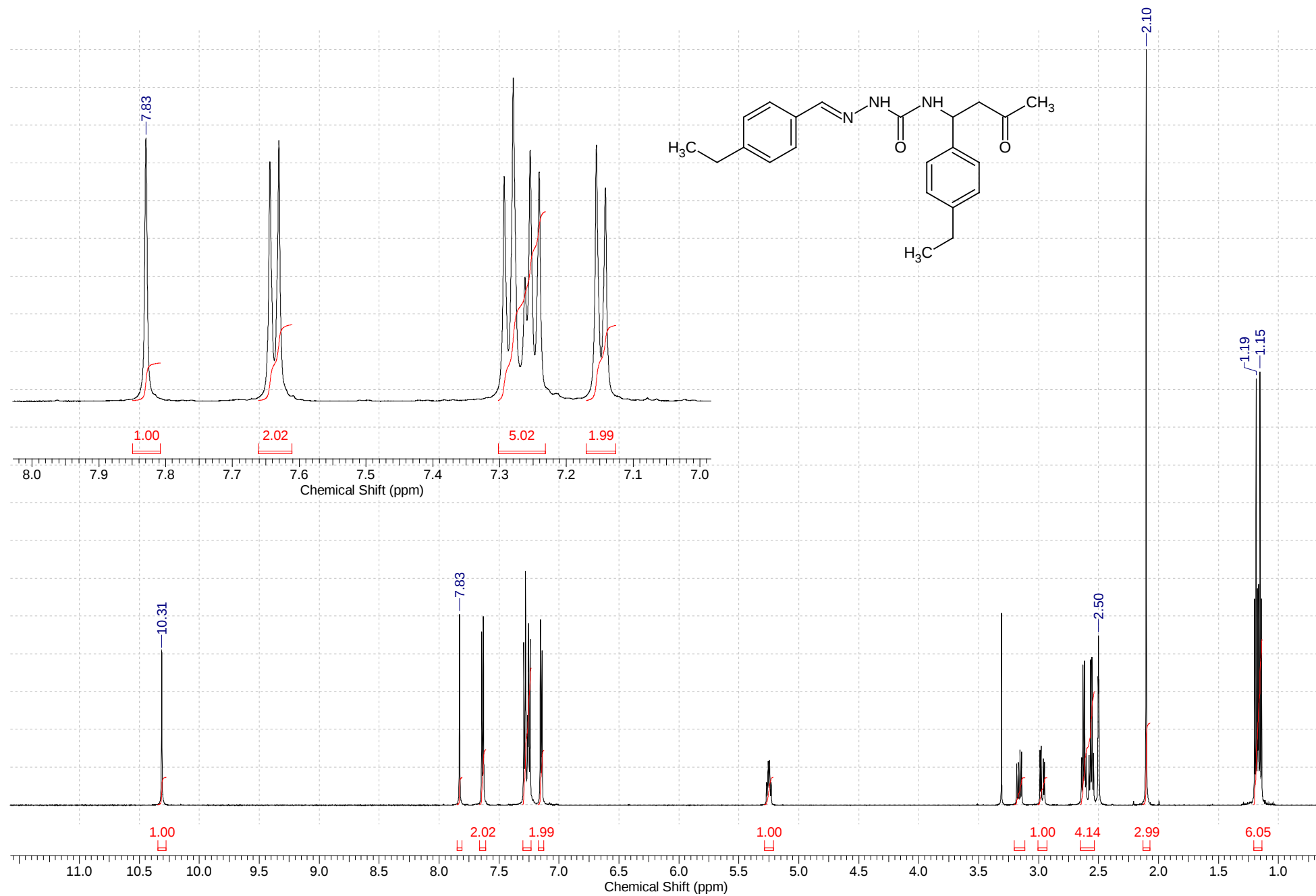
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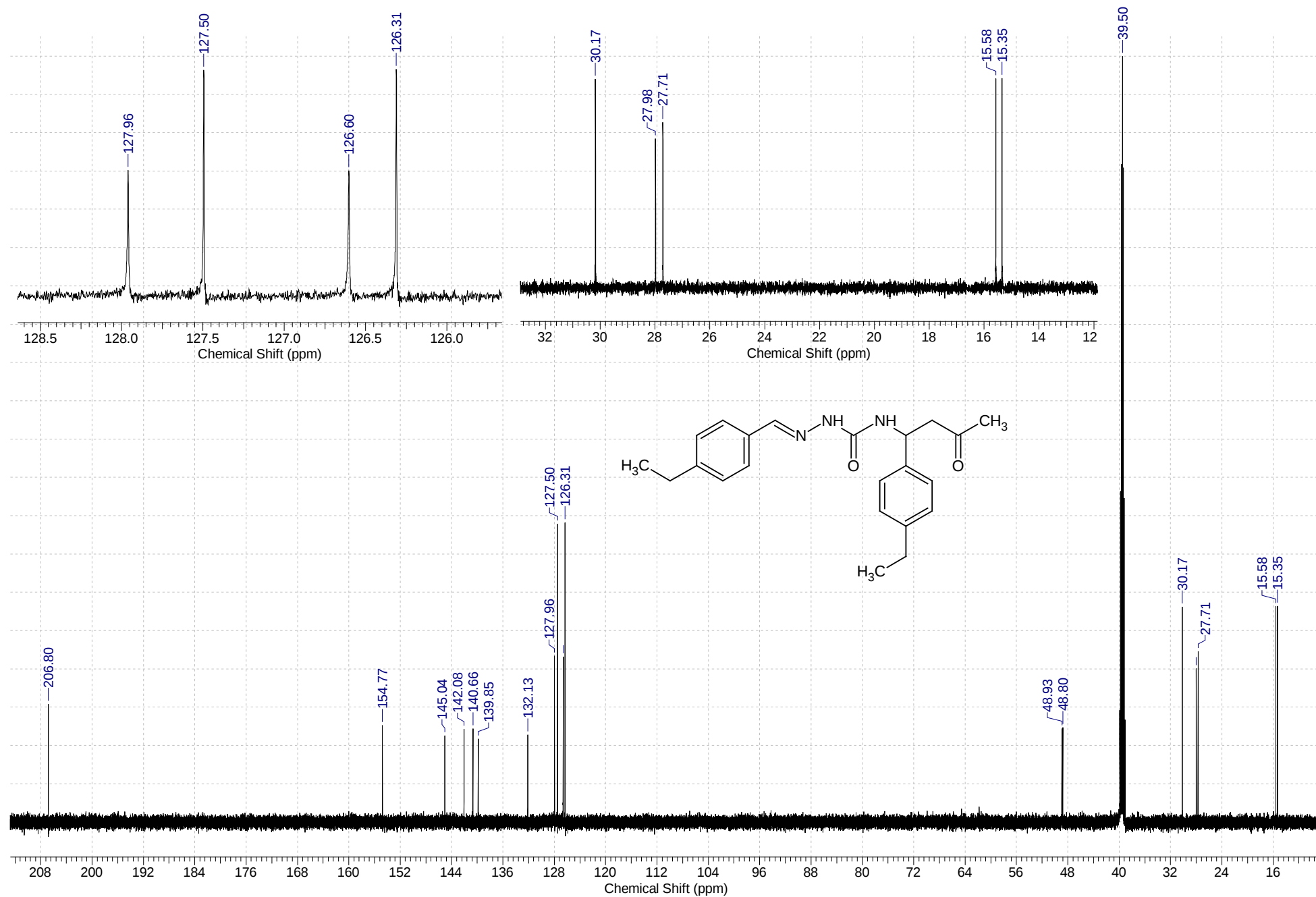
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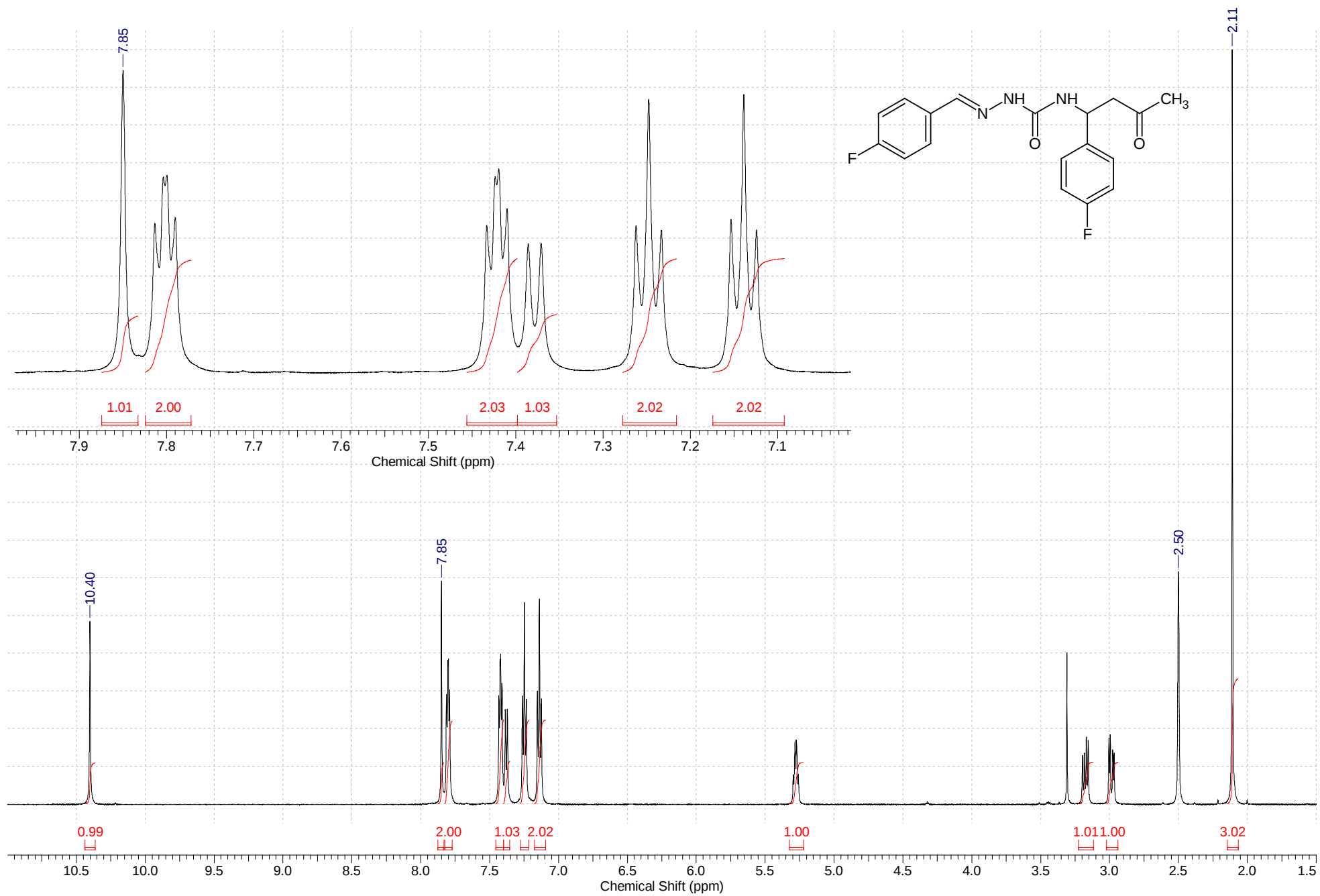
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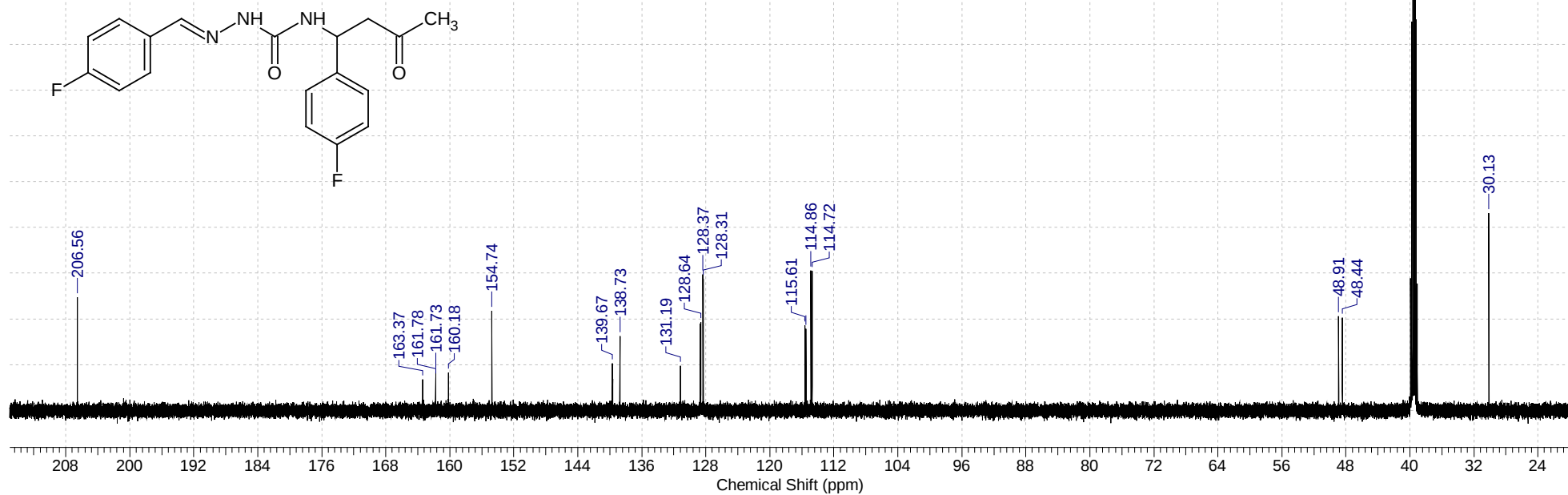
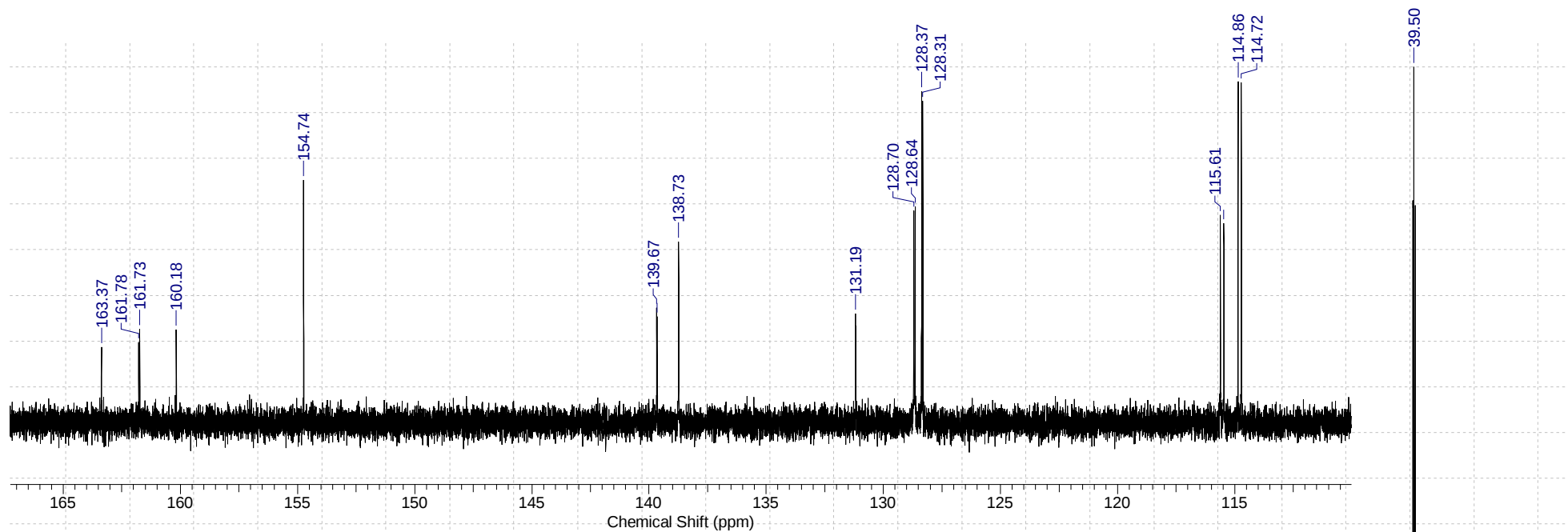
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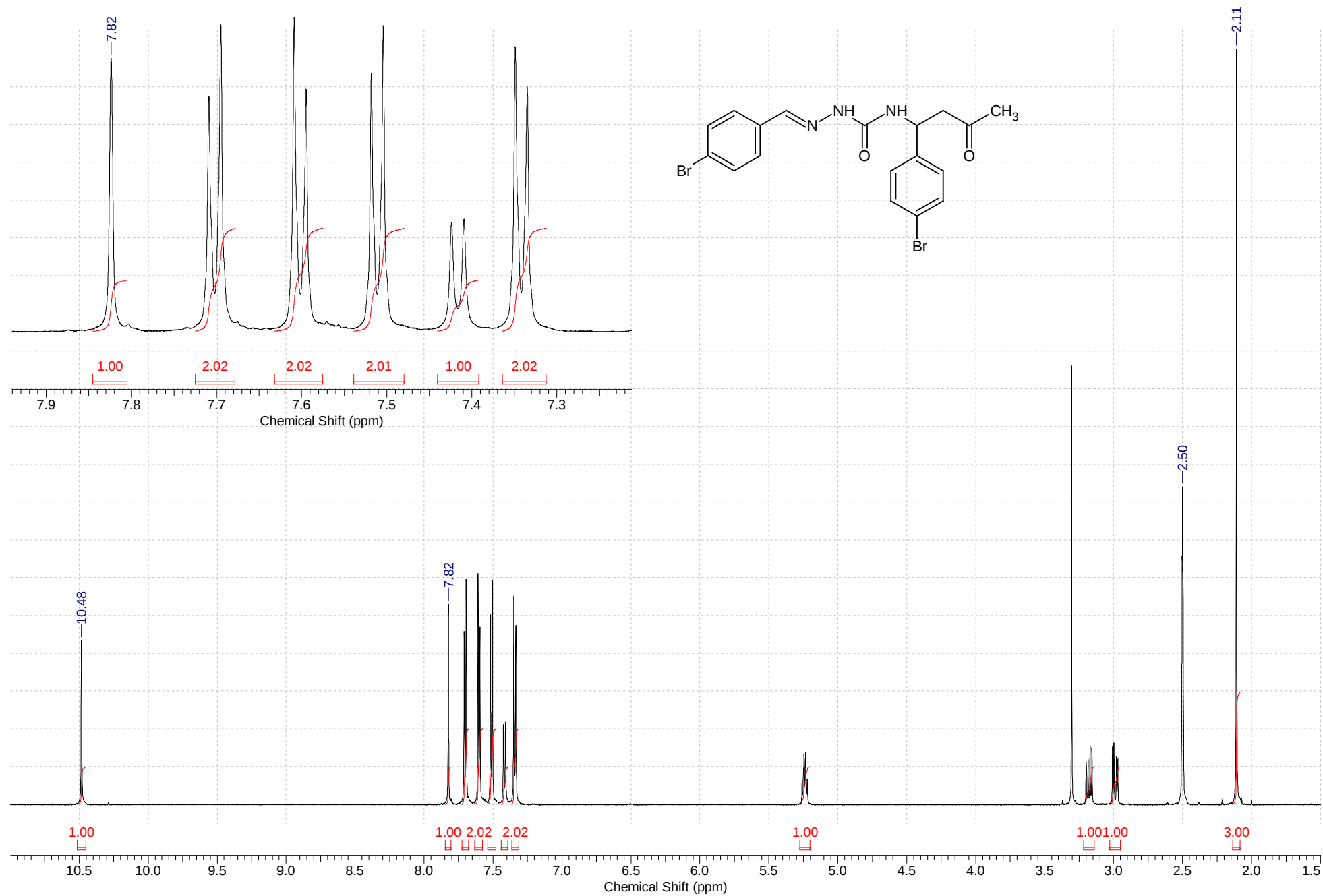
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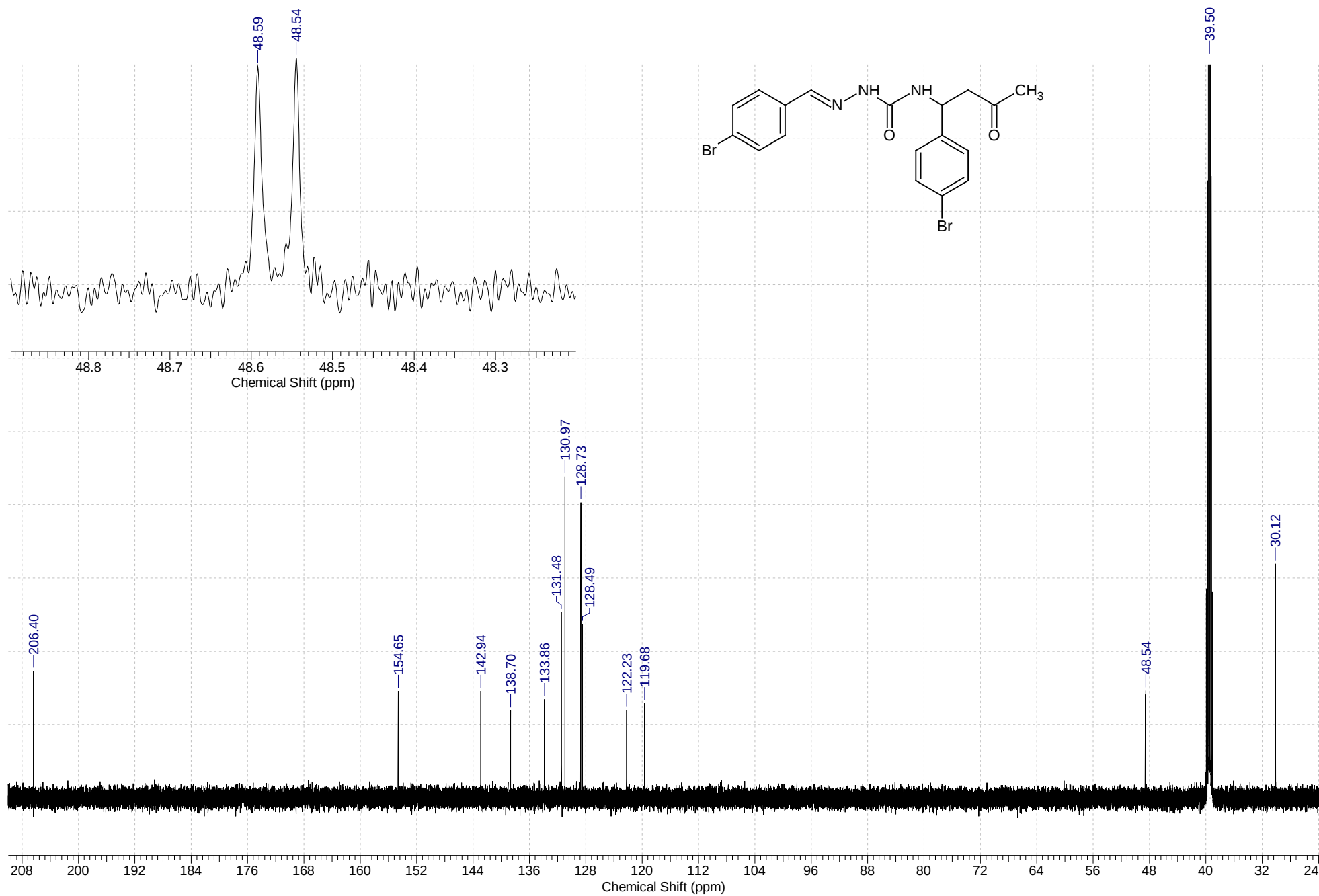
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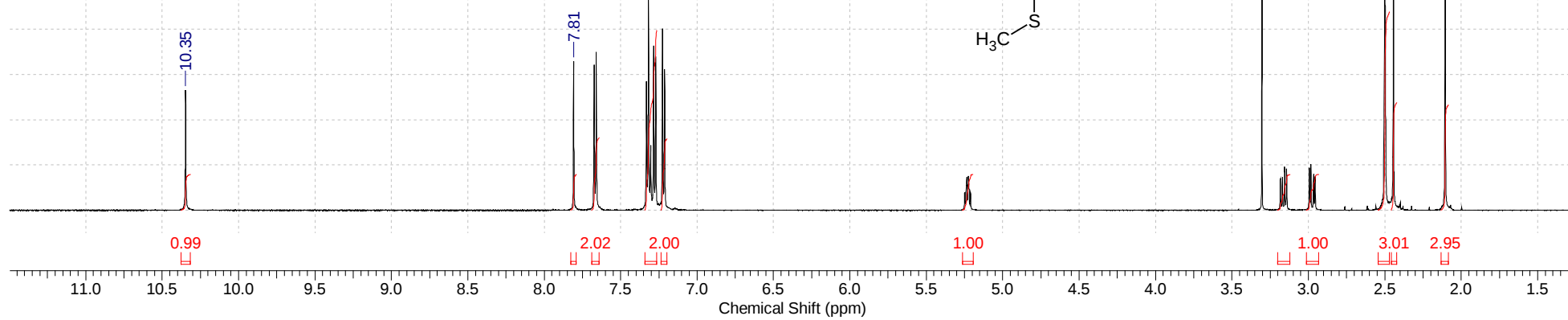
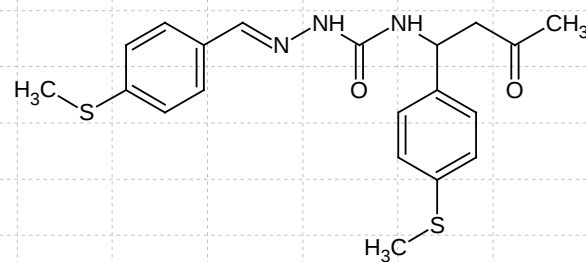
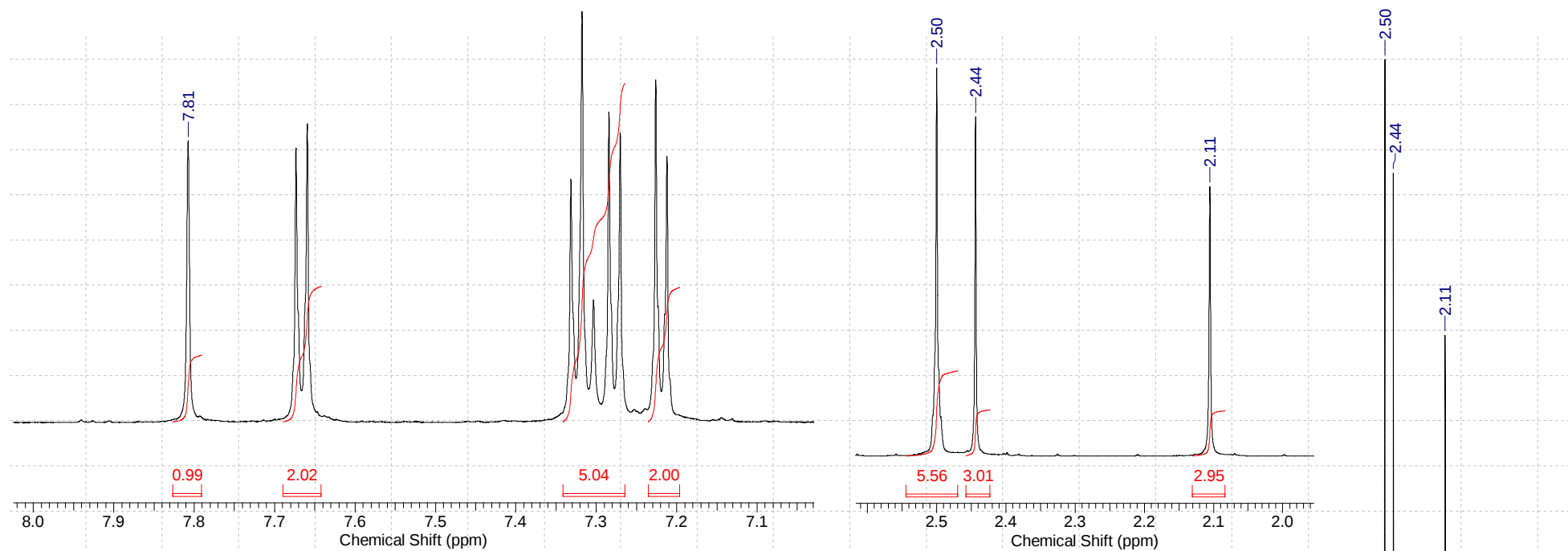
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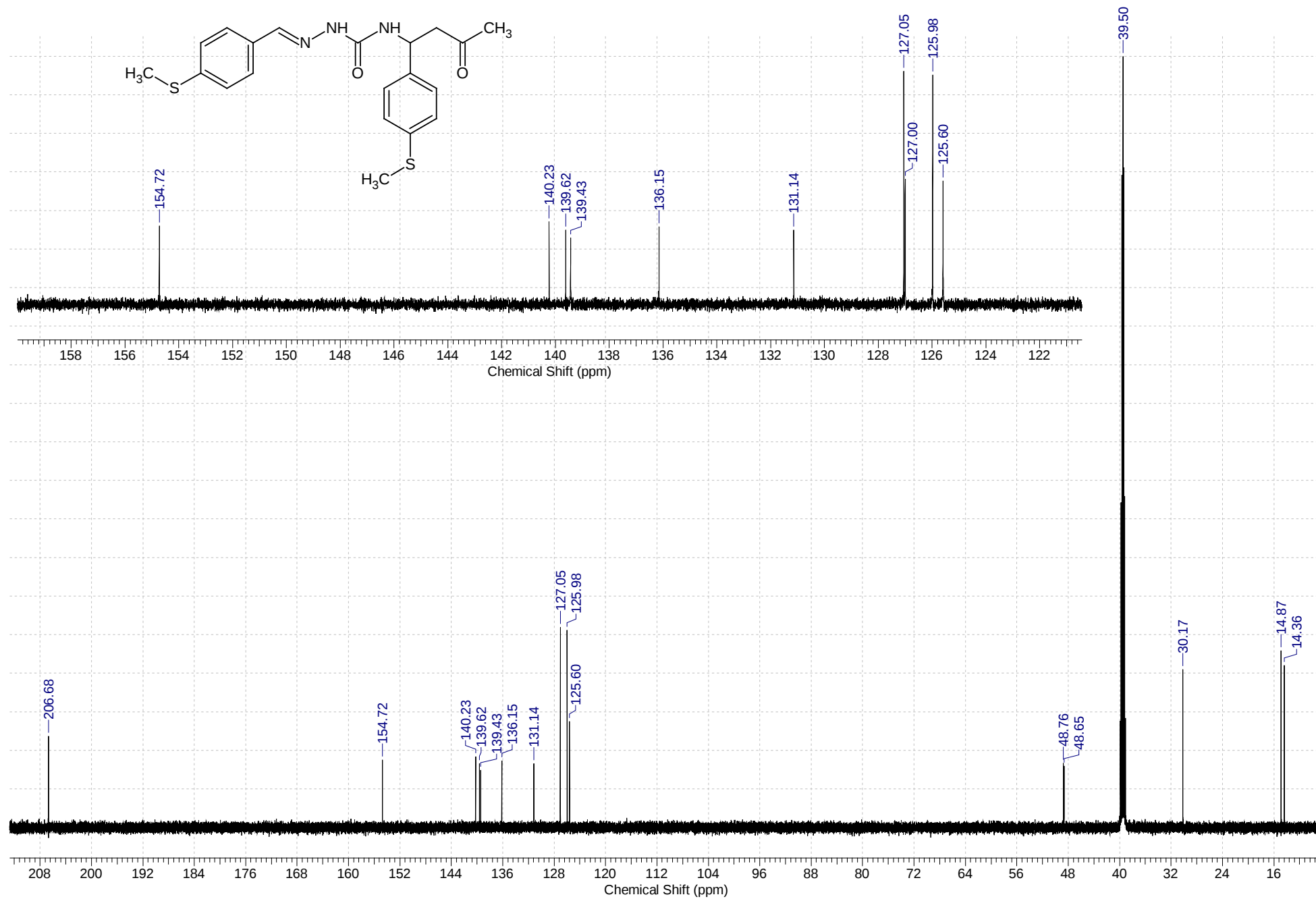
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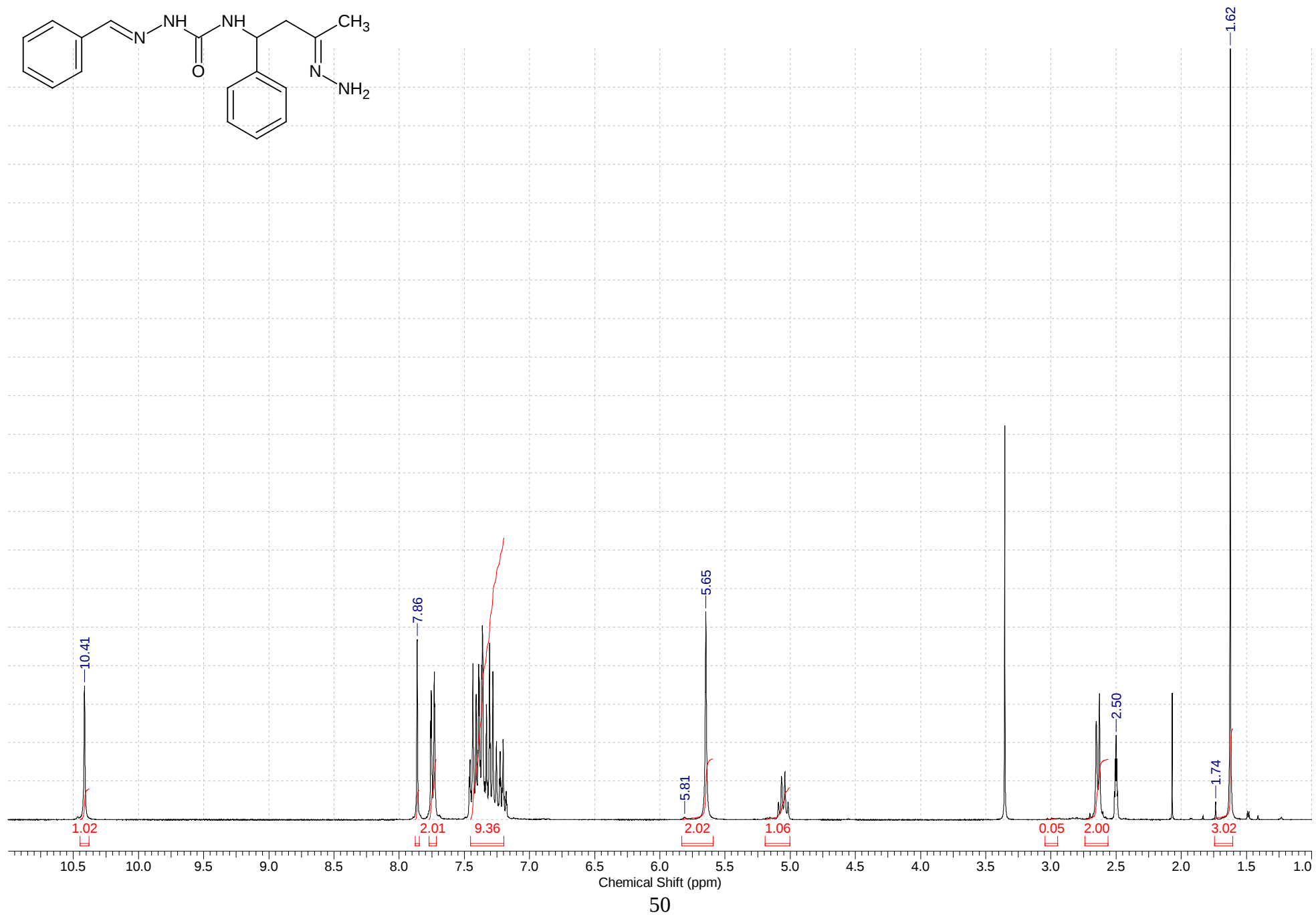
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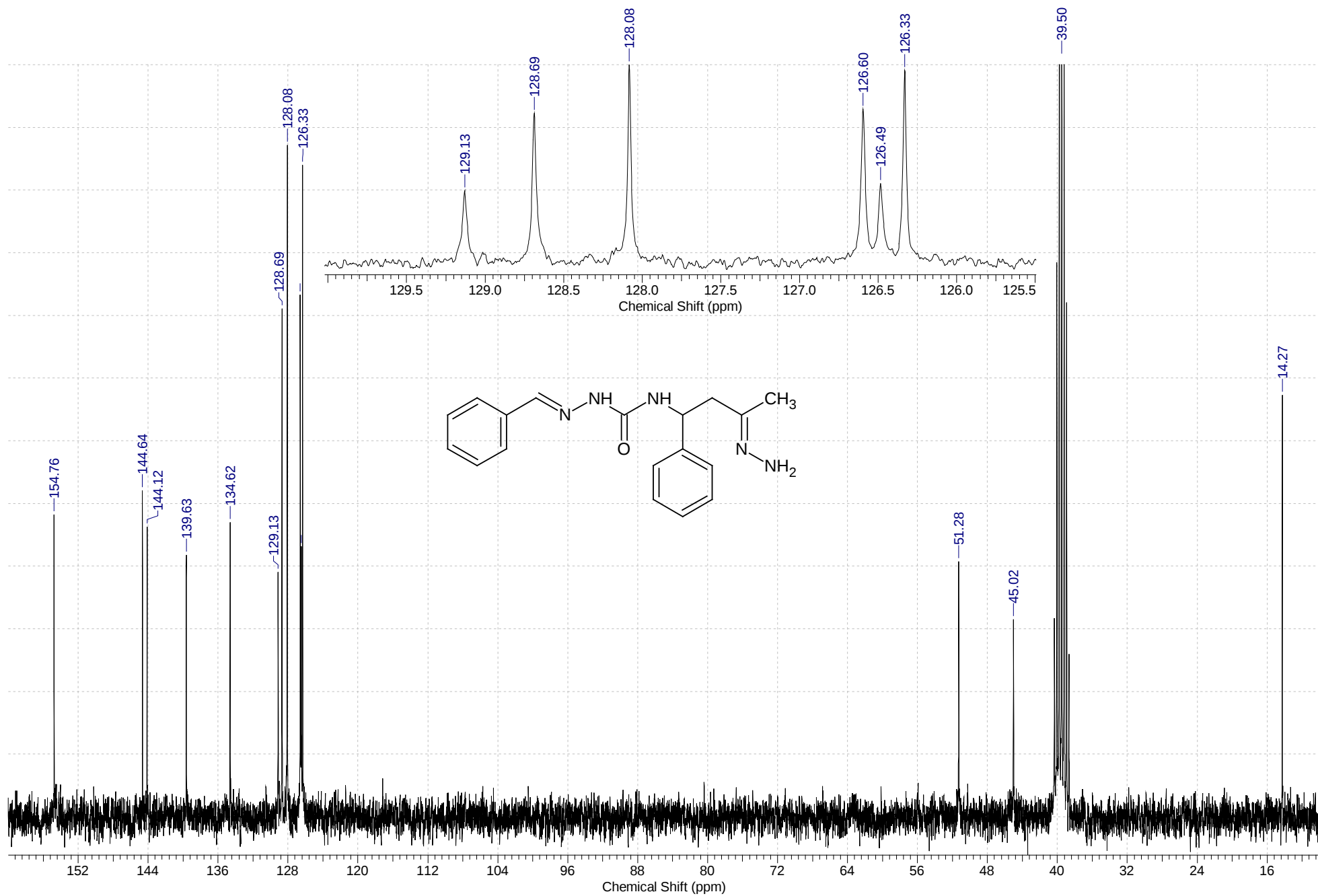
¹³C NMR spectrum of compound **10h** (DMSO-*d*₆).



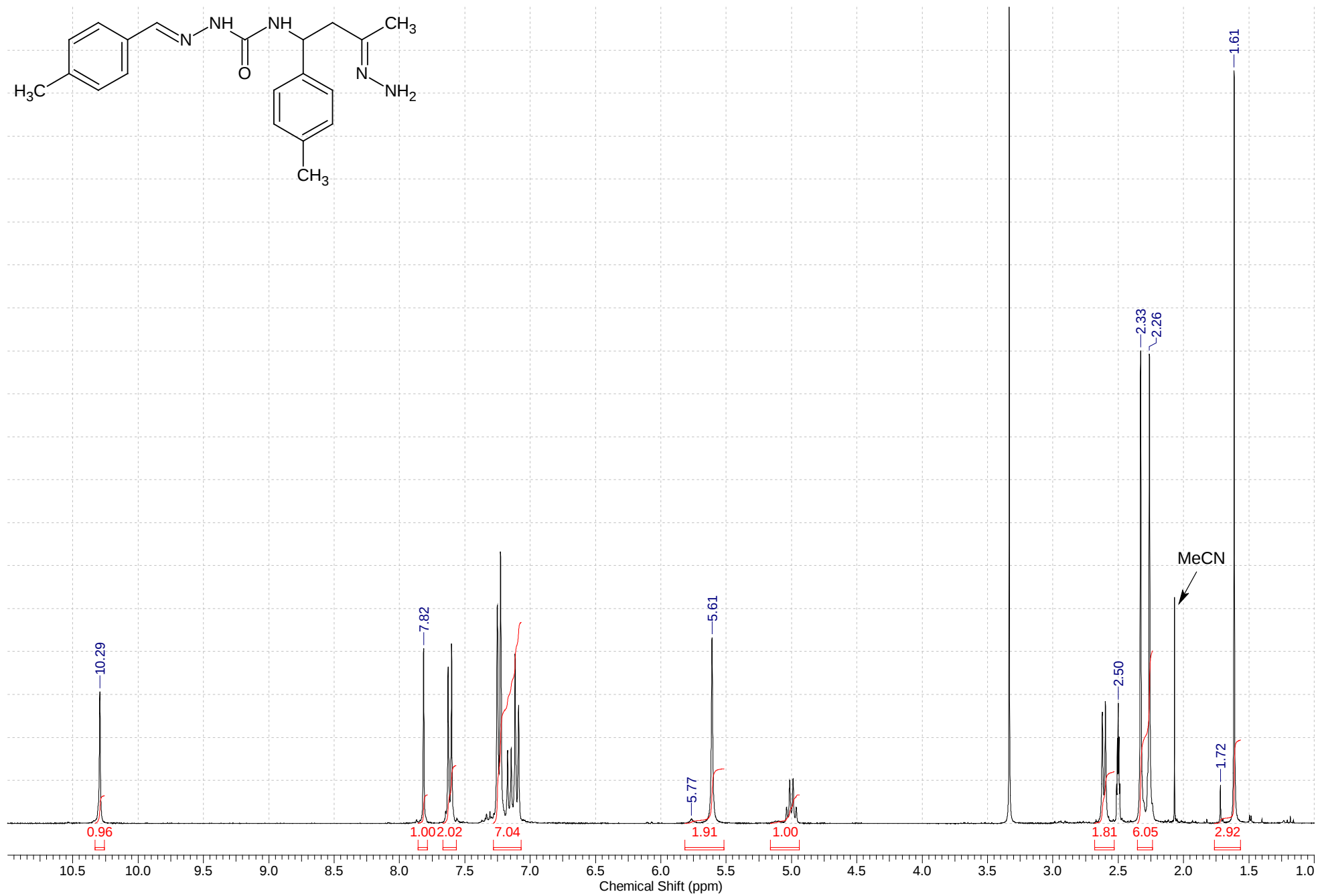
¹H NMR spectrum of compound **14a** (DMSO-*d*₆).



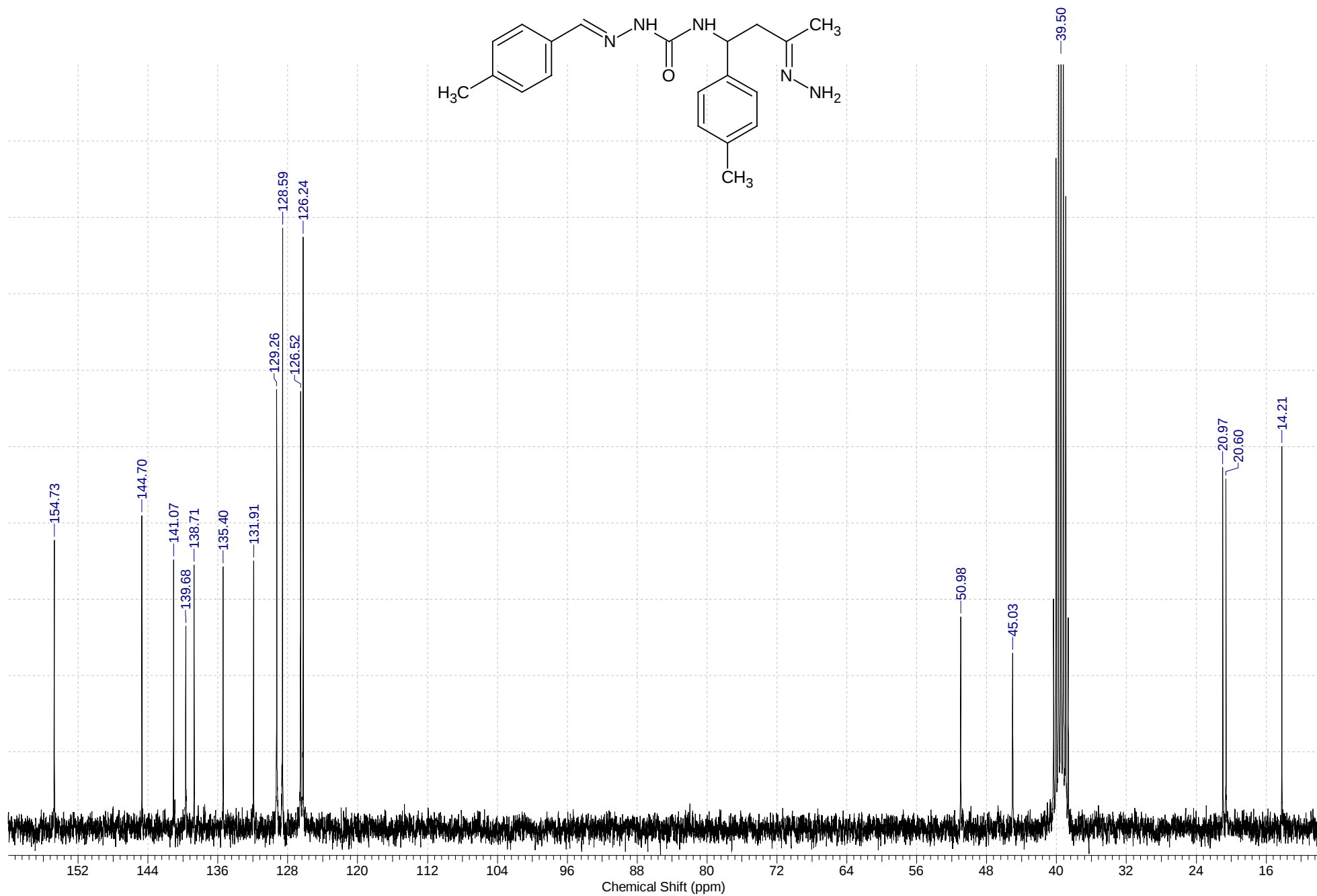
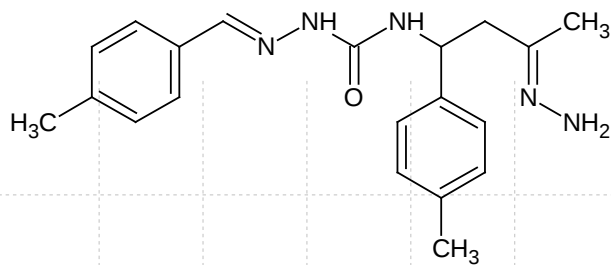
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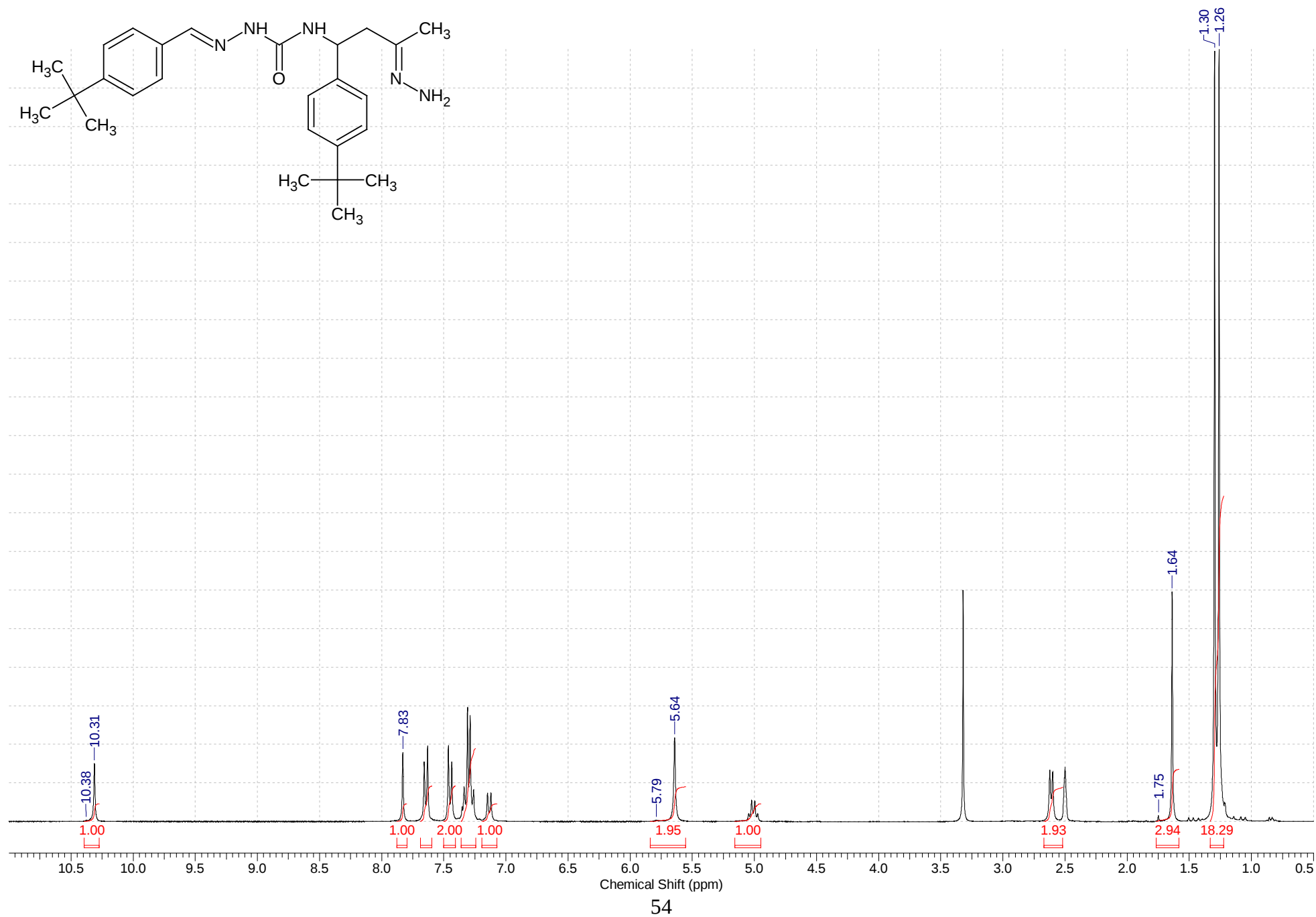
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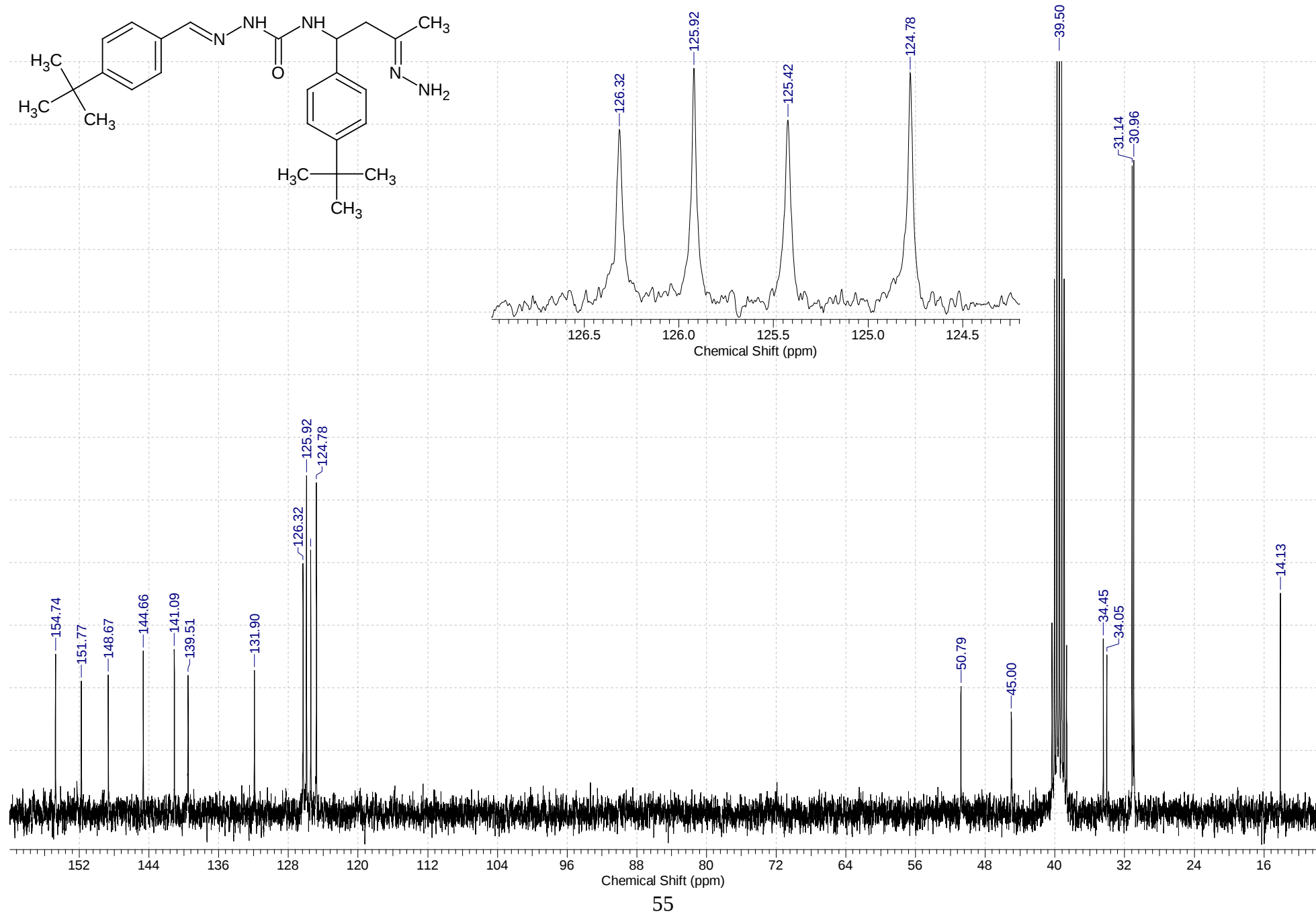
^{13}C NMR spectrum of compound **14b** ($\text{DMSO}-d_6$).



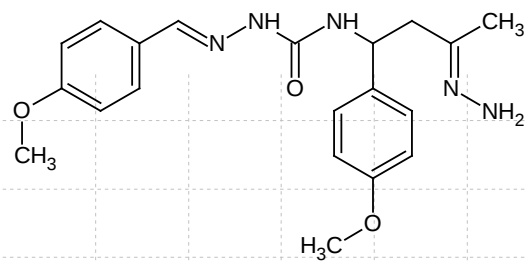
¹H NMR spectrum of compound **14c** (DMSO-*d*₆).



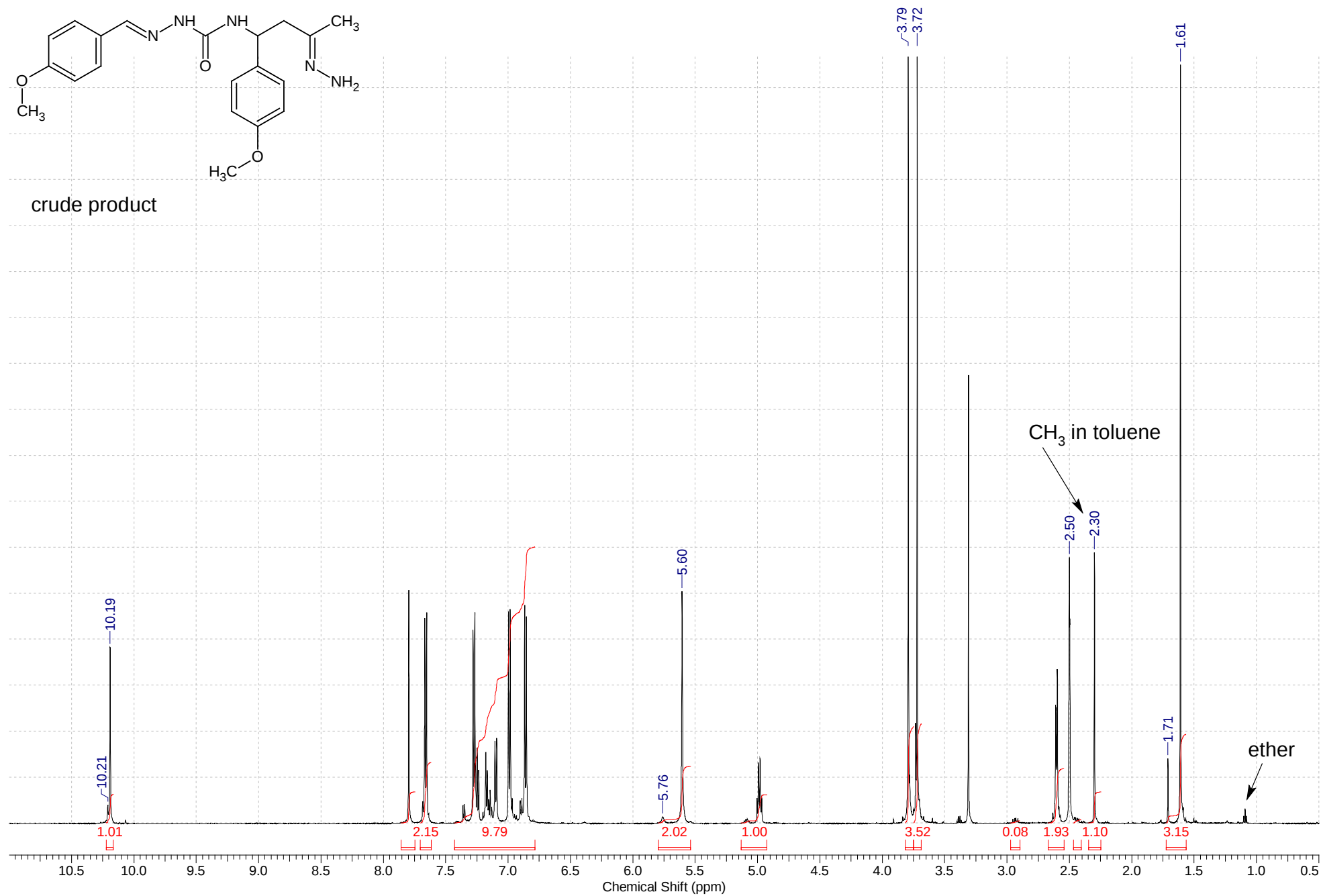
^{13}C NMR spectrum of compound **14c** (DMSO- d_6).



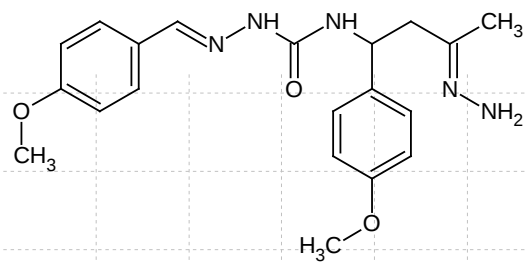
¹H NMR spectrum of compound **14d** (DMSO-*d*₆).



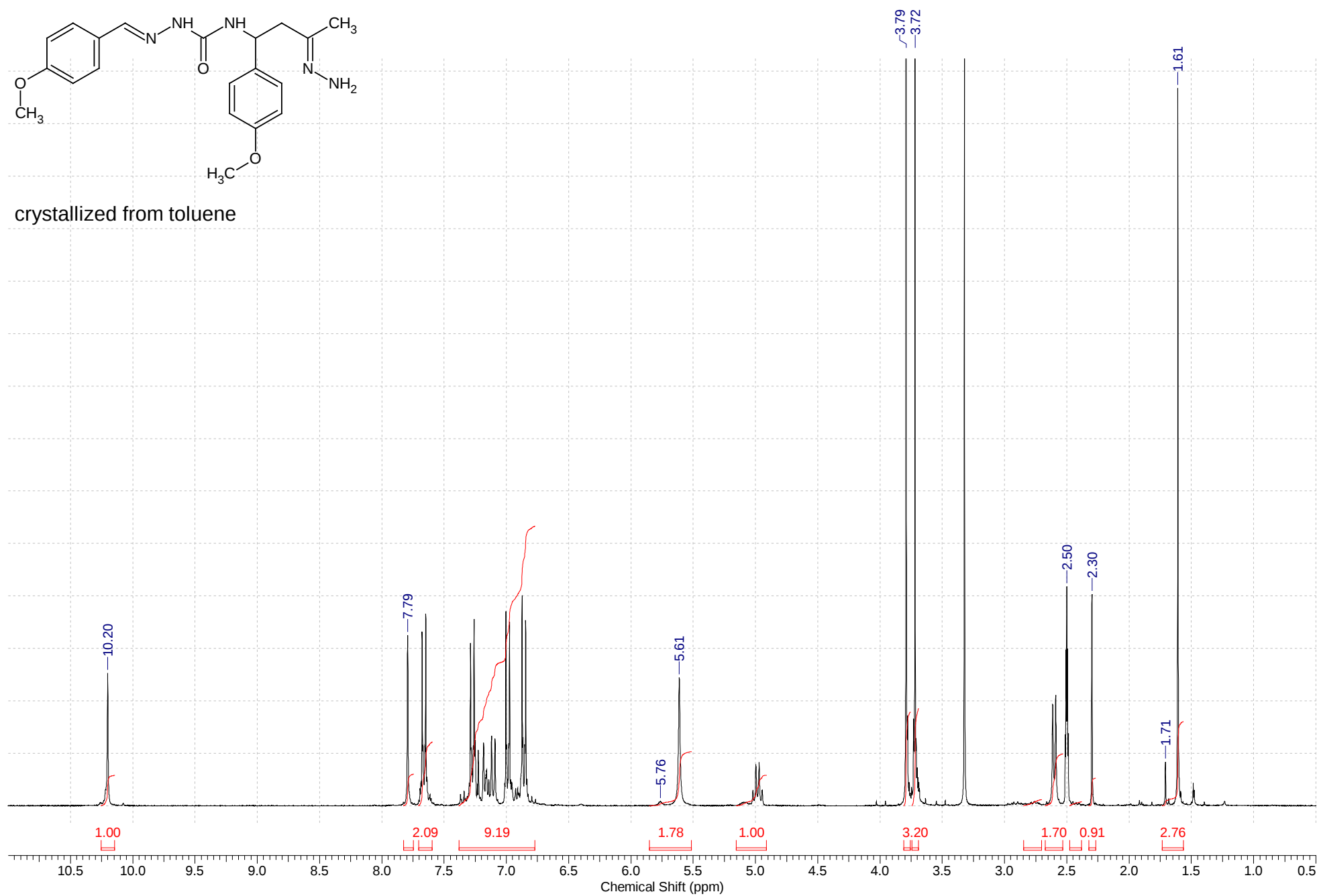
crude product



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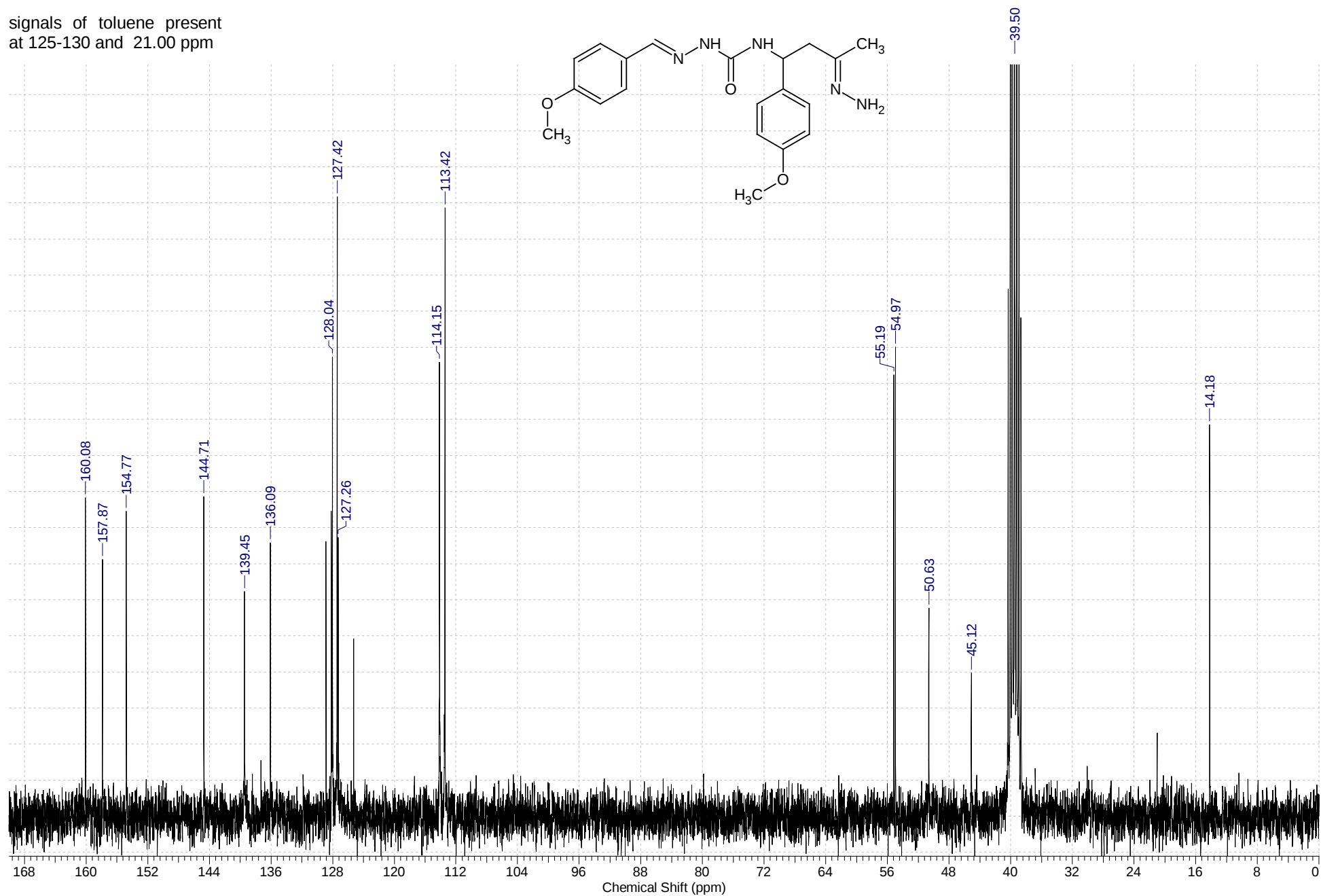
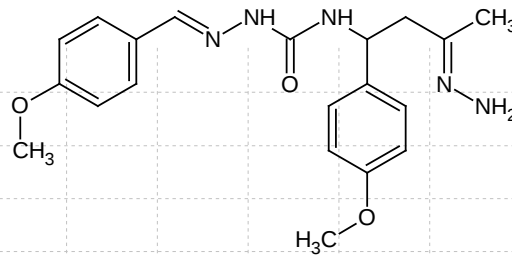


crystallized from toluene

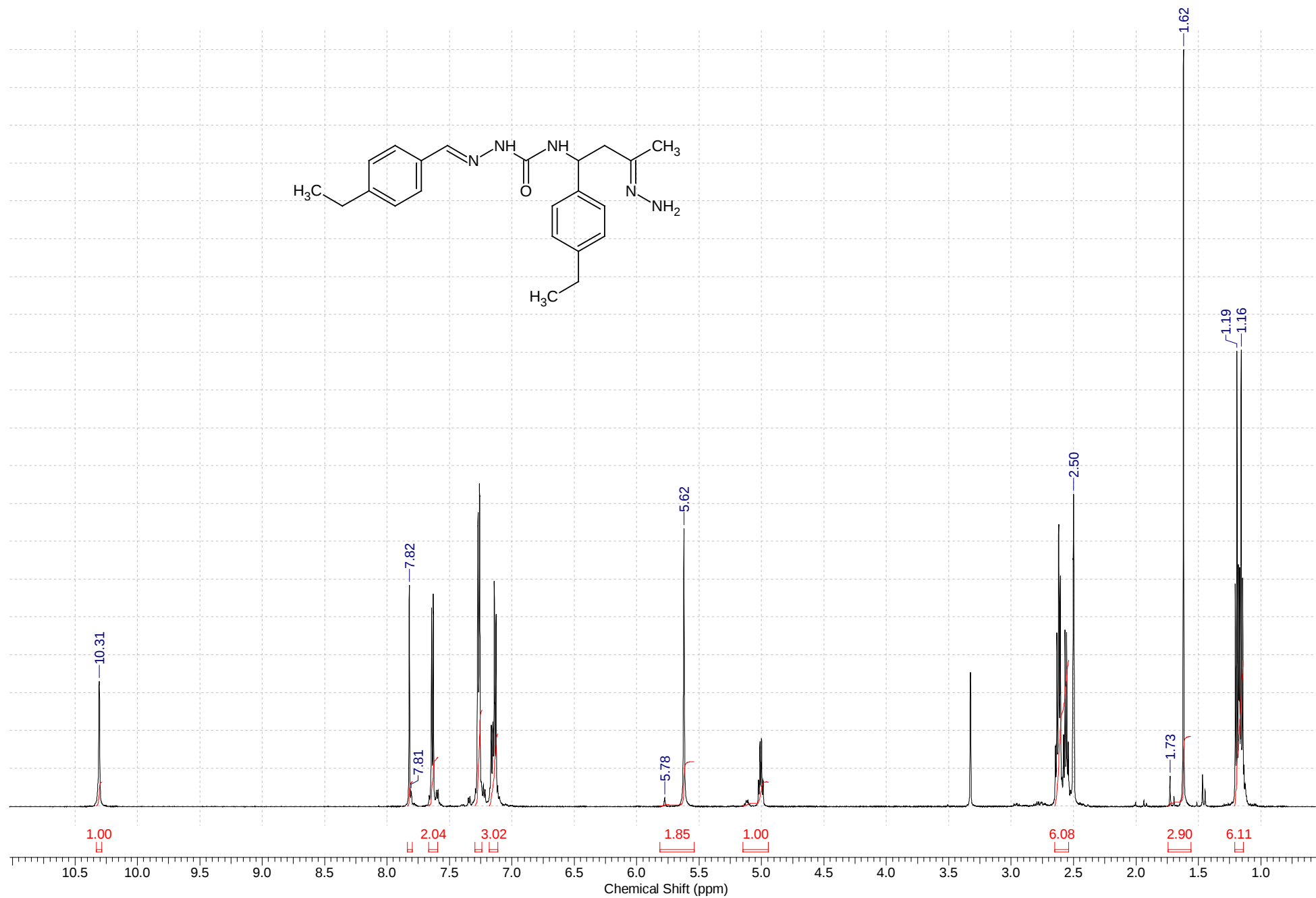


¹³C NMR spectrum of compound **14d** (DMSO-*d*₆).

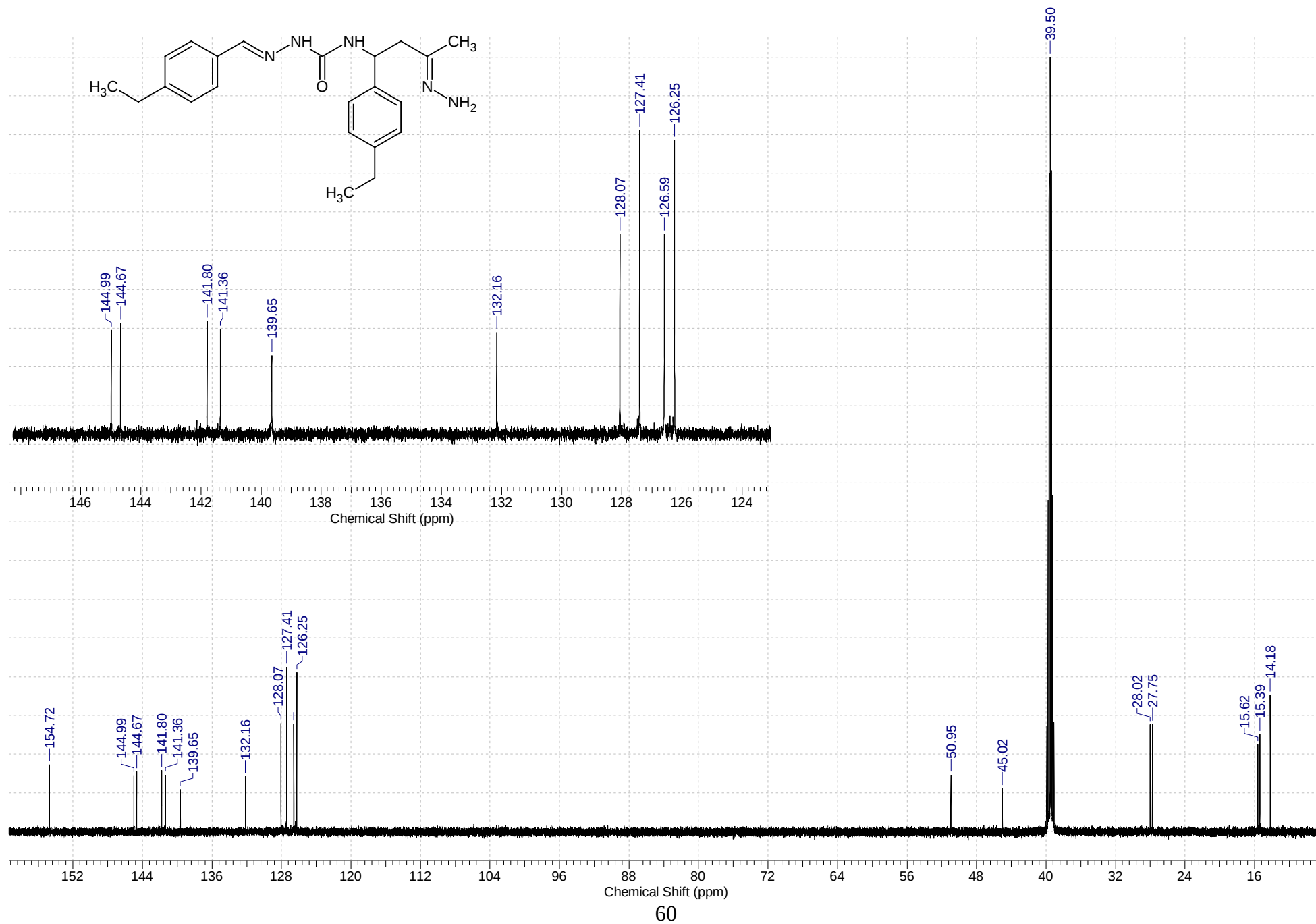
signals of toluene present
at 125-130 and 21.00 ppm



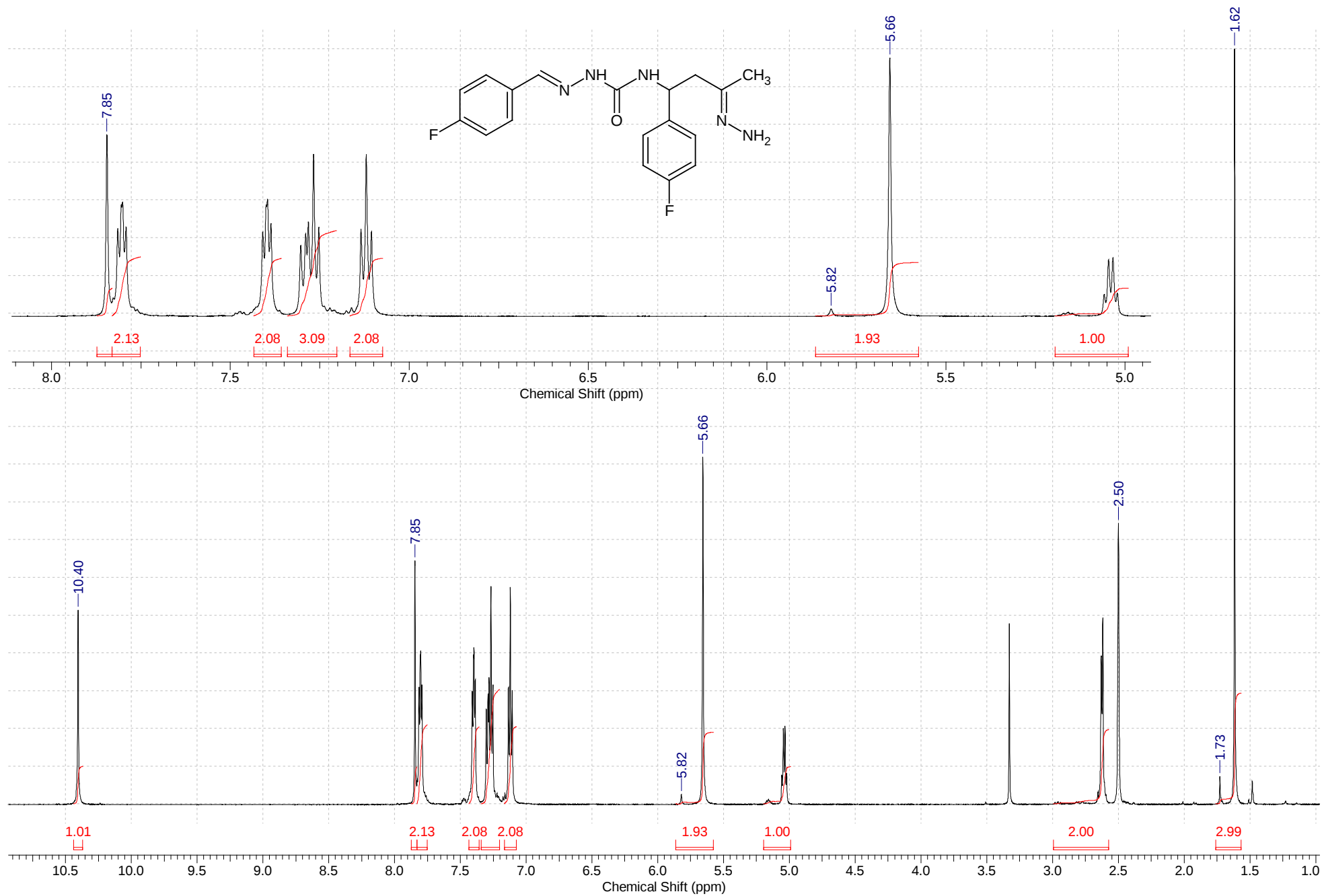
^1H NMR spectrum of compound **14e** ($\text{DMSO-}d_6$).



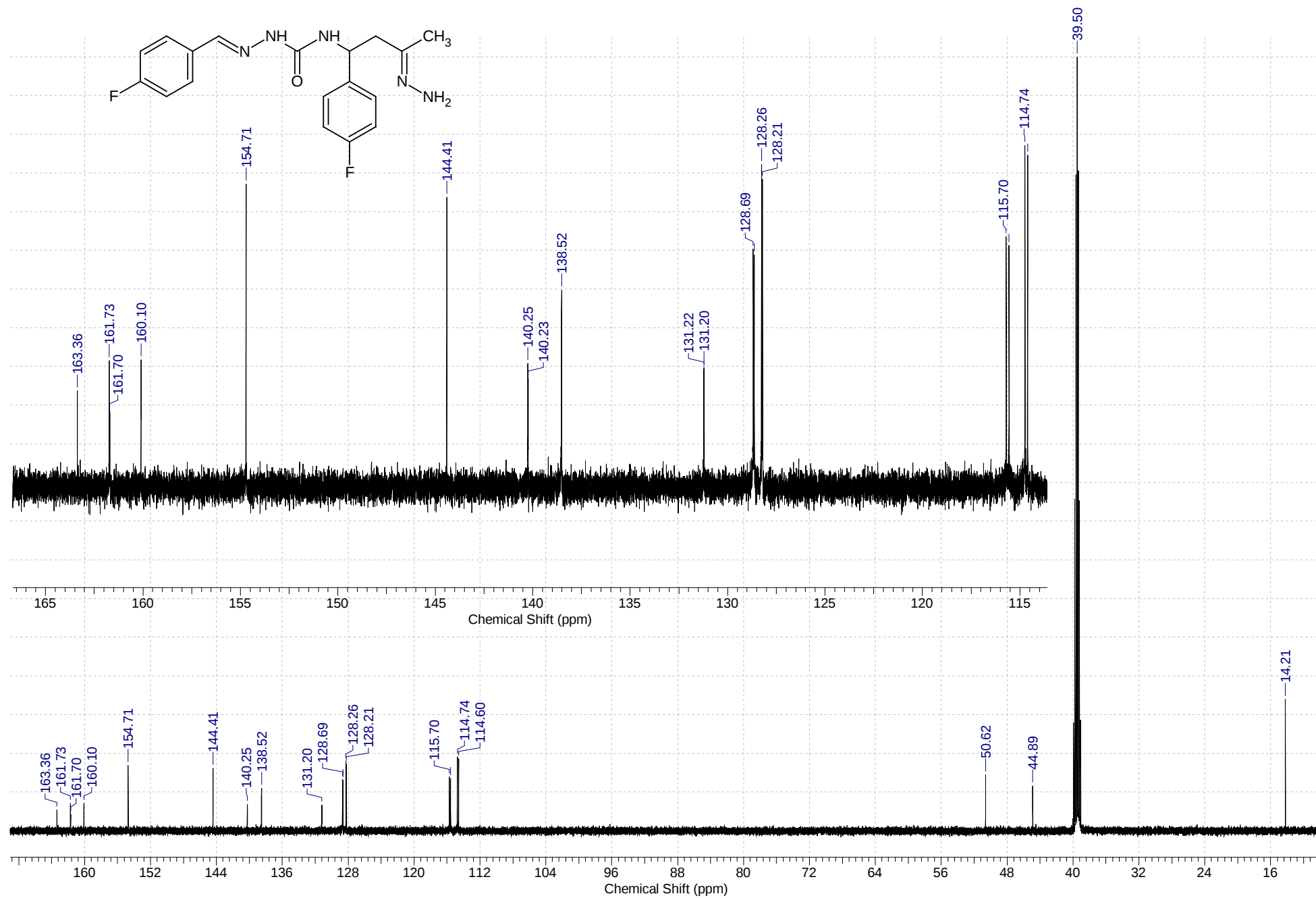
^{13}C NMR spectrum of compound **14e** (DMSO- d_6).



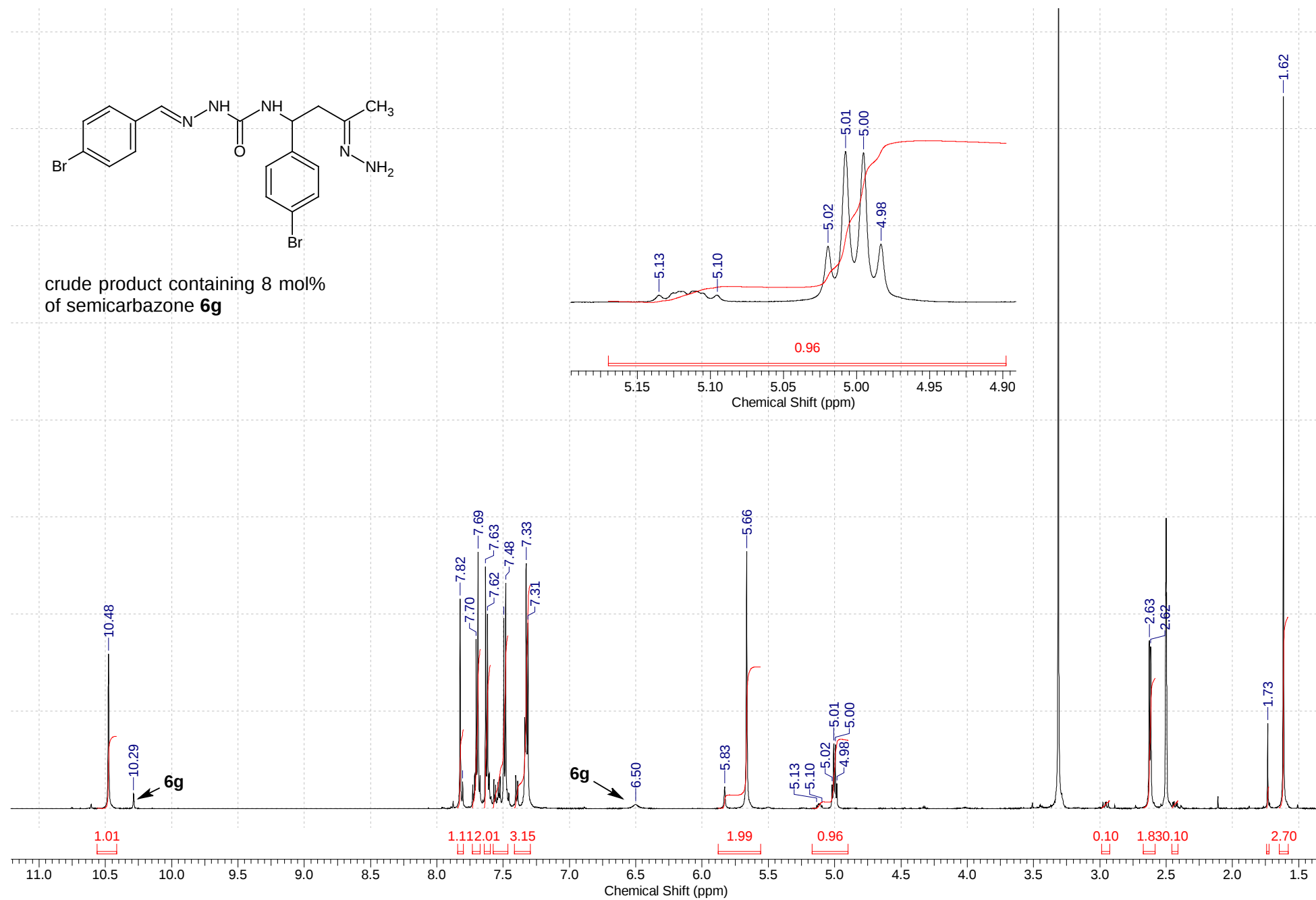
¹H NMR spectrum of compound **14f** (DMSO-*d*₆).



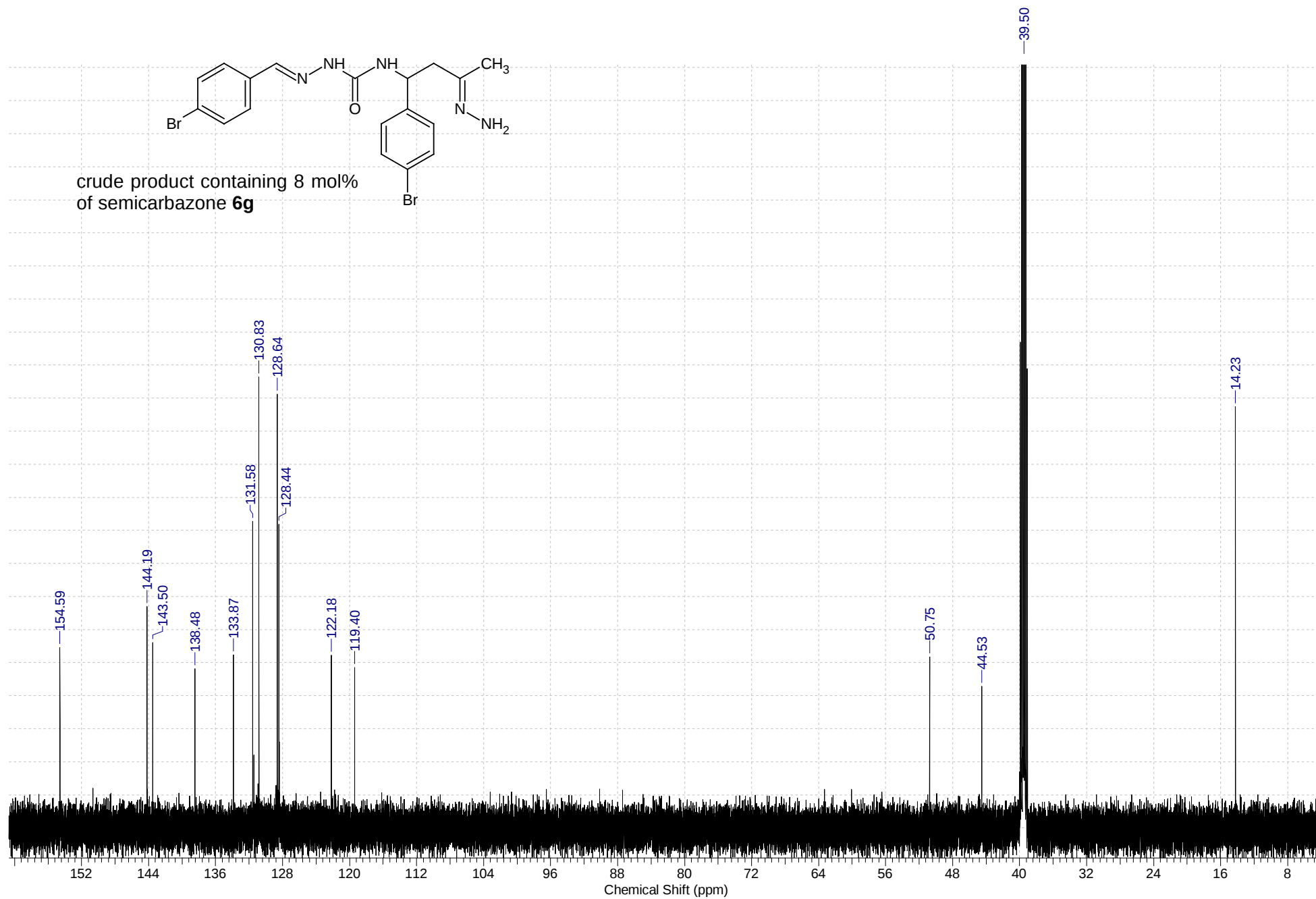
^{13}C NMR spectrum of compound **14f** (DMSO- d_6).



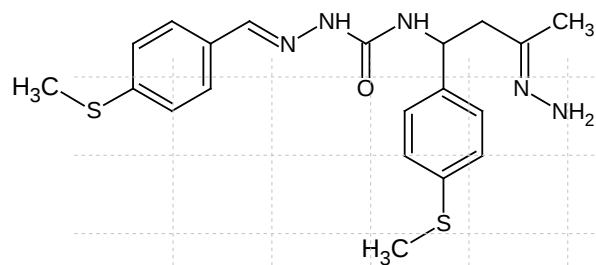
¹H NMR spectrum of crude compound **14g** (DMSO-*d*₆).



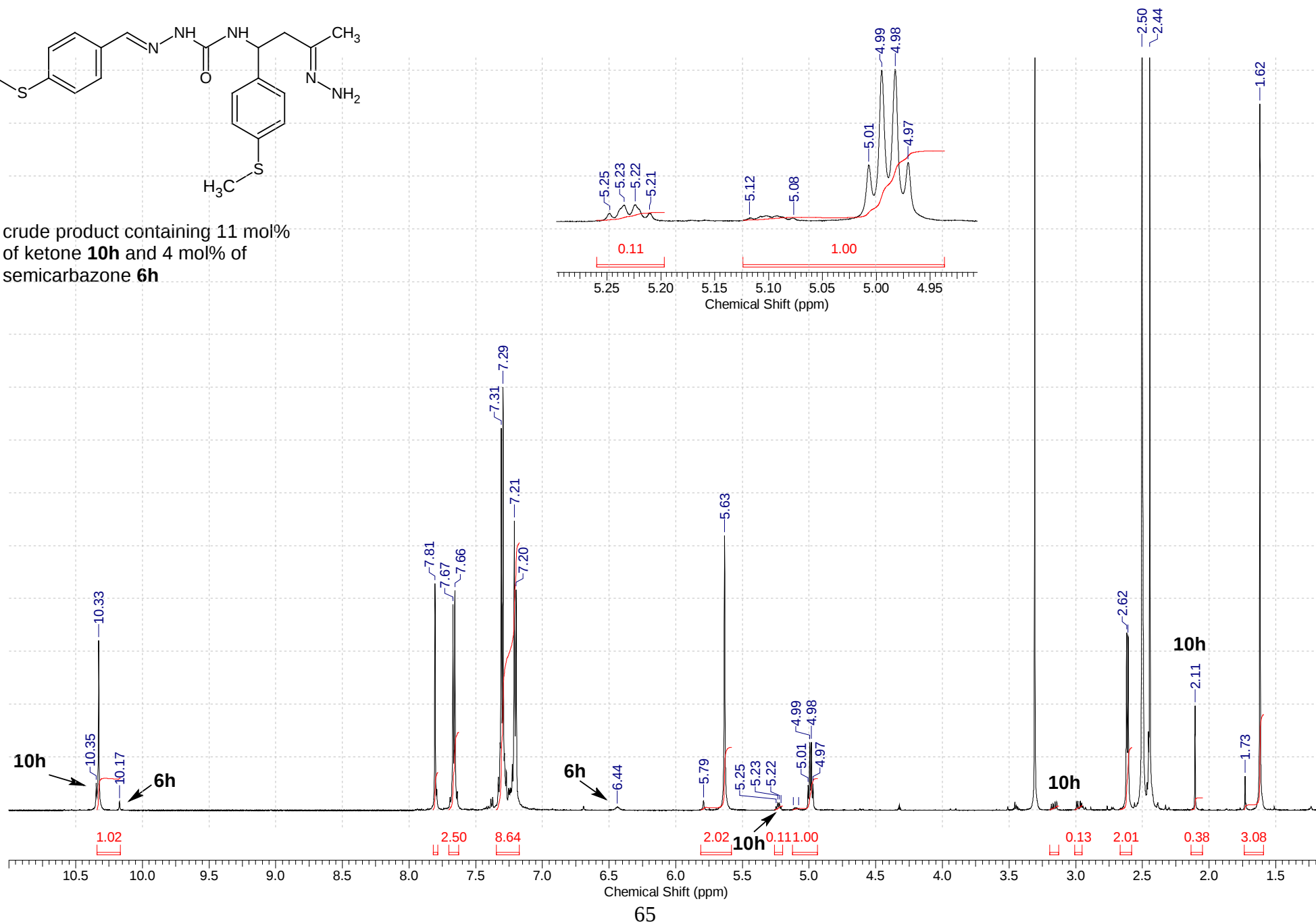
^{13}C NMR spectrum of crude compound **14g** ($\text{DMSO}-d_6$).



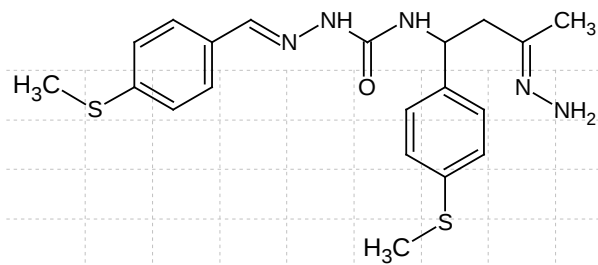
^1H NMR spectrum of crude compound **14h** ($\text{DMSO}-d_6$).



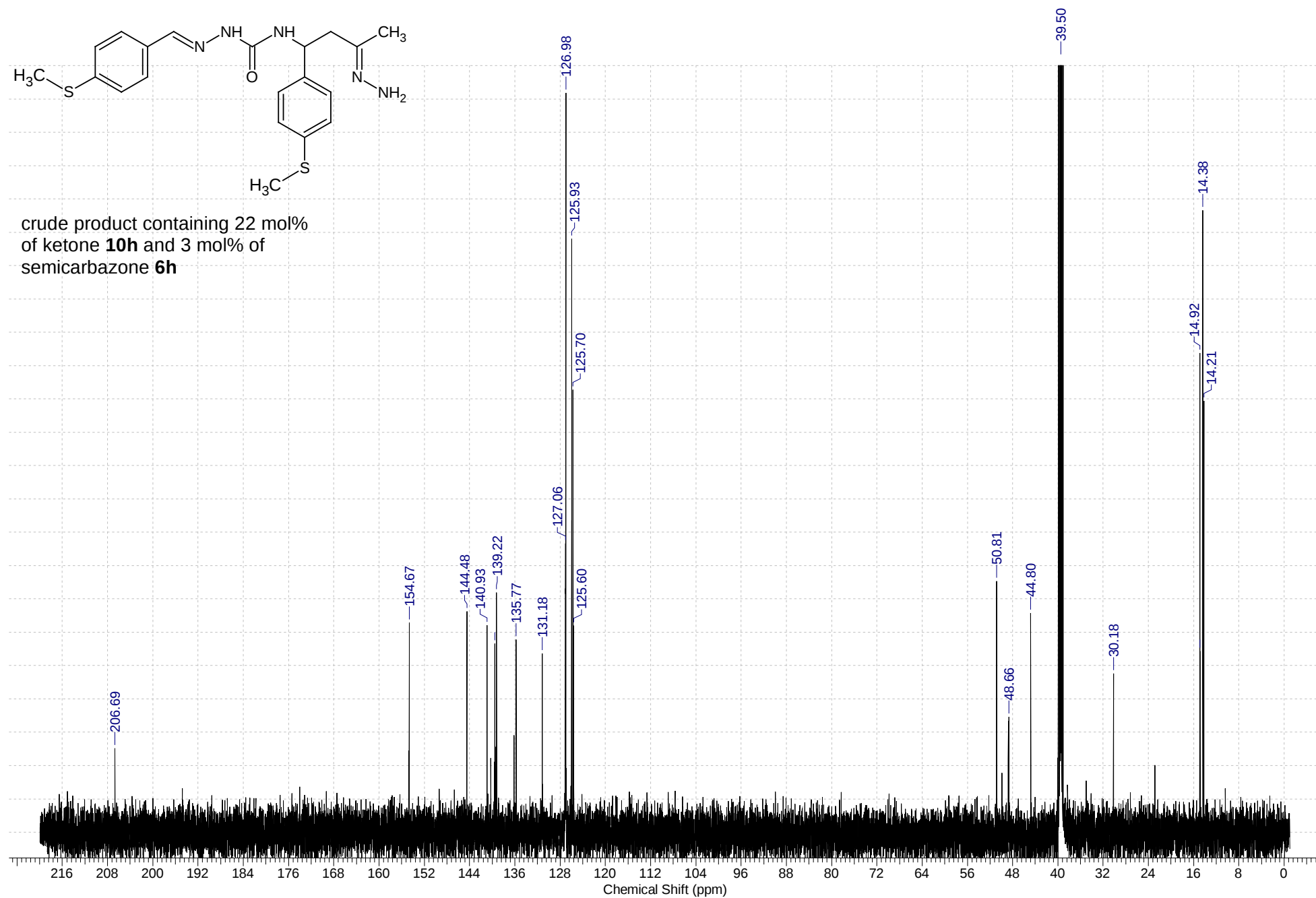
crude product containing 11 mol%
of ketone **10h** and 4 mol% of
semicarbazone **6h**



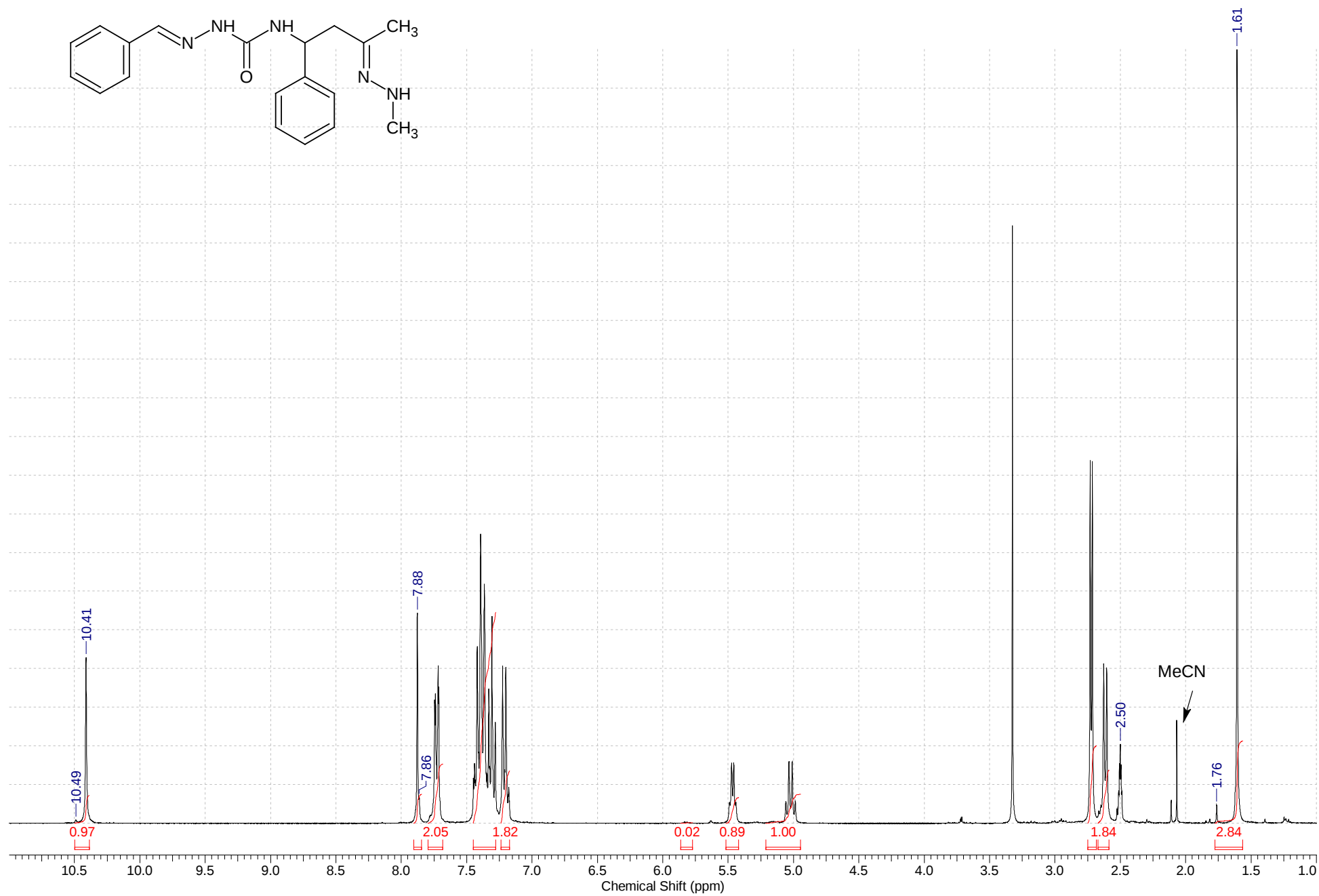
^{13}C NMR spectrum of crude compound **14h** ($\text{DMSO}-d_6$).



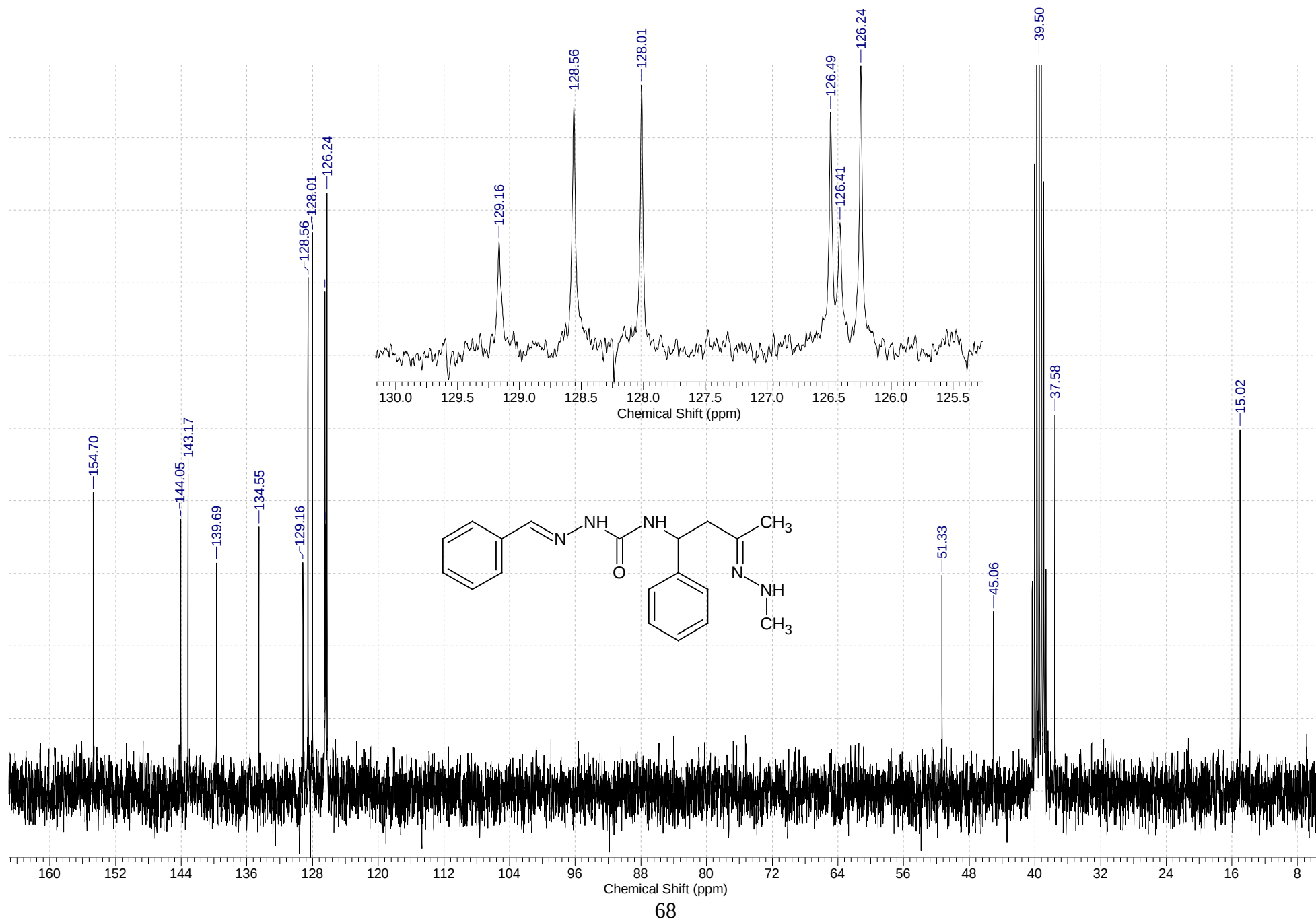
crude product containing 22 mol%
of ketone **10h** and 3 mol% of
semicarbazone **6h**



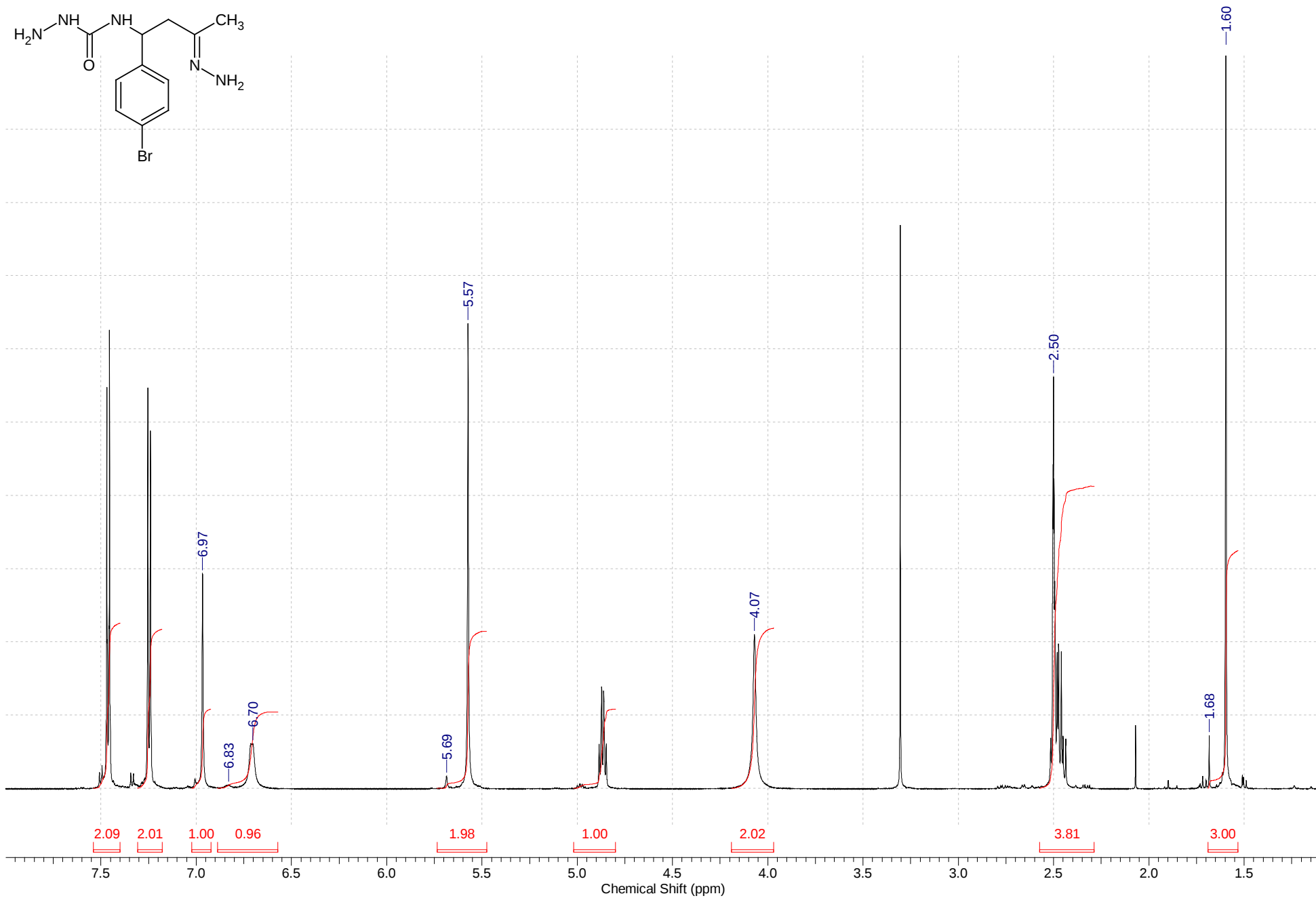
¹H NMR spectrum of compound 15 (DMSO-*d*₆).



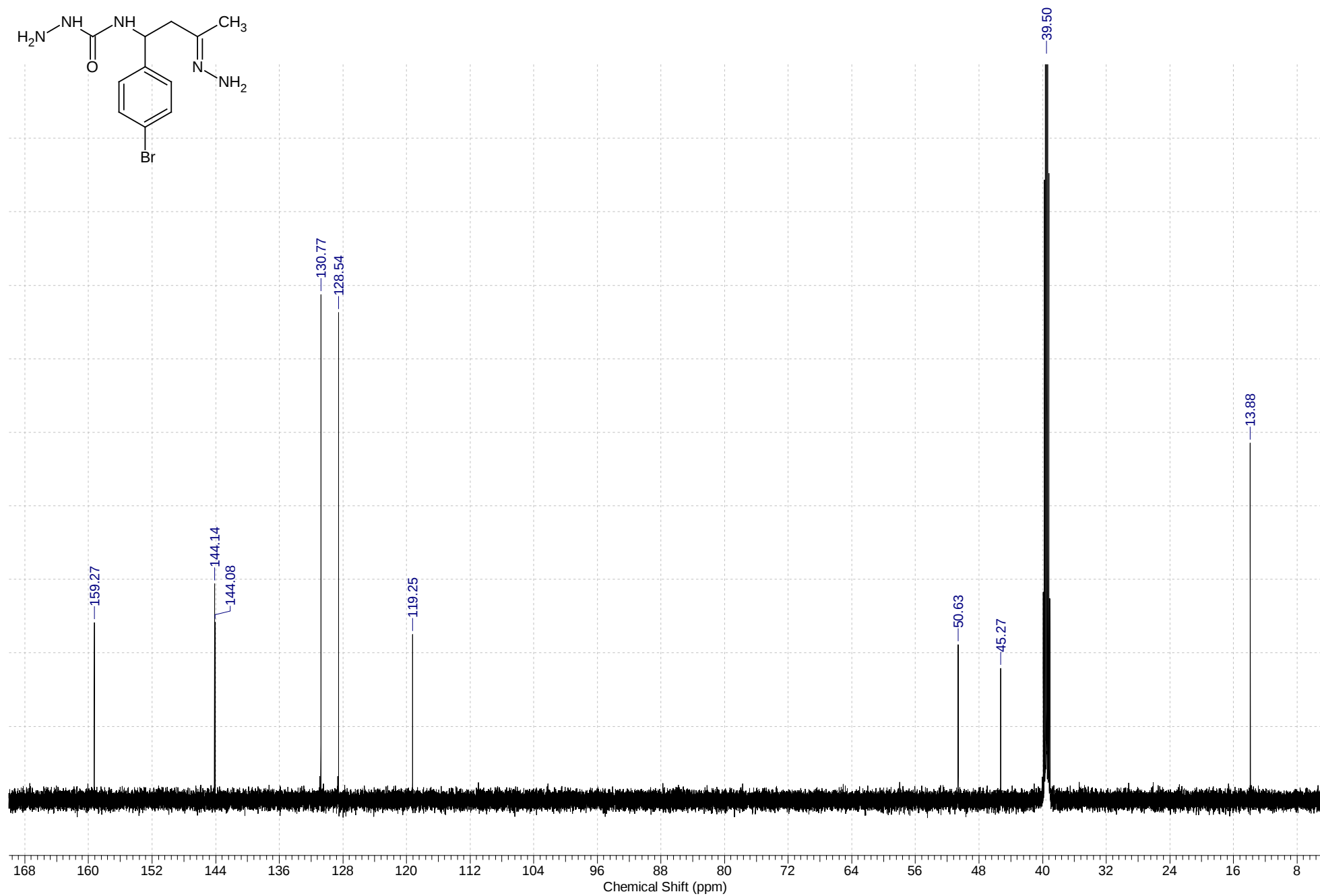
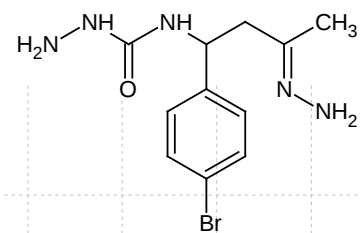
^{13}C NMR spectrum of compound 15 (DMSO- d_6).



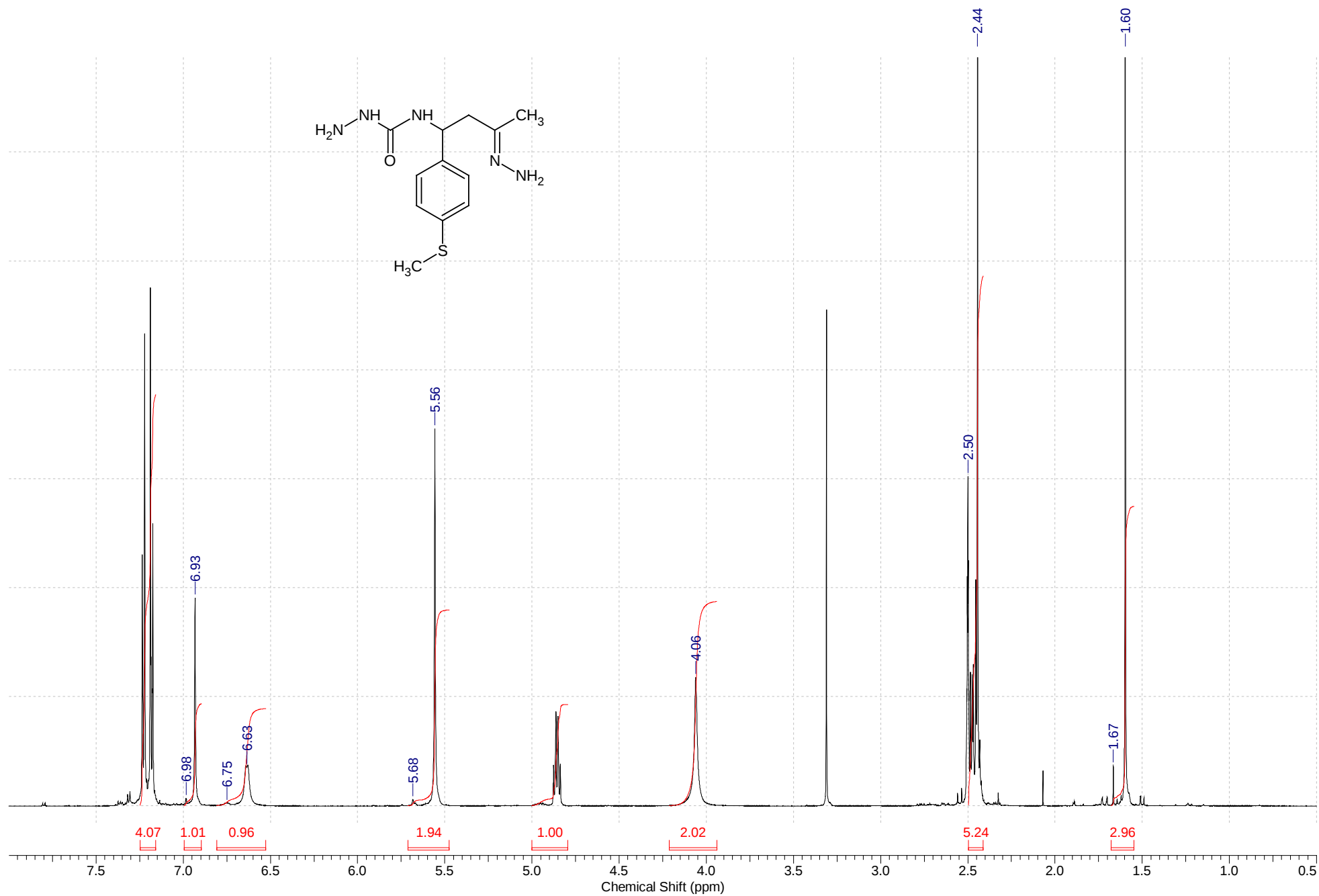
¹H NMR spectrum of compound **16a** (DMSO-*d*₆).



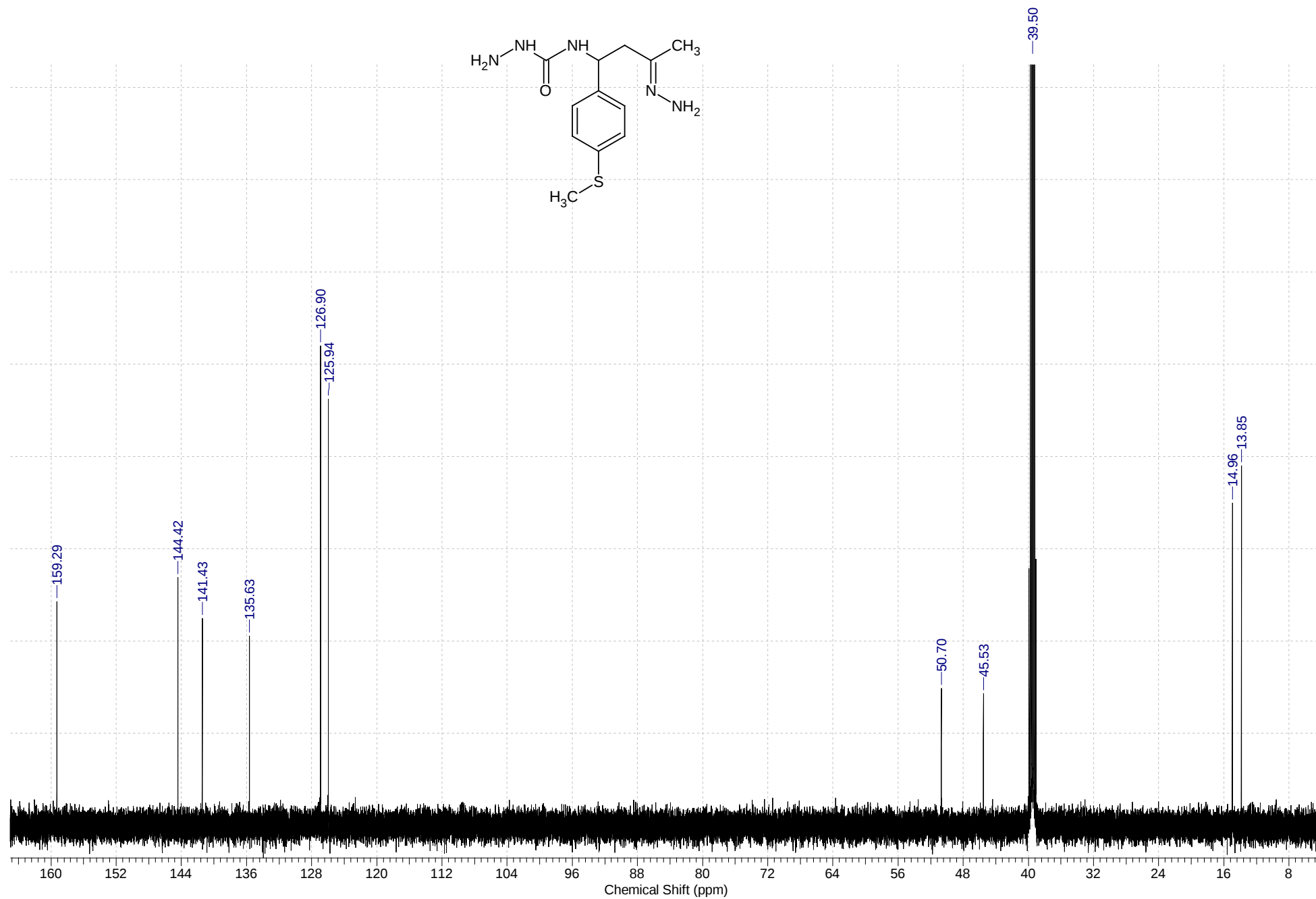
^{13}C NMR spectrum of compound **16a** ($\text{DMSO-}d_6$).



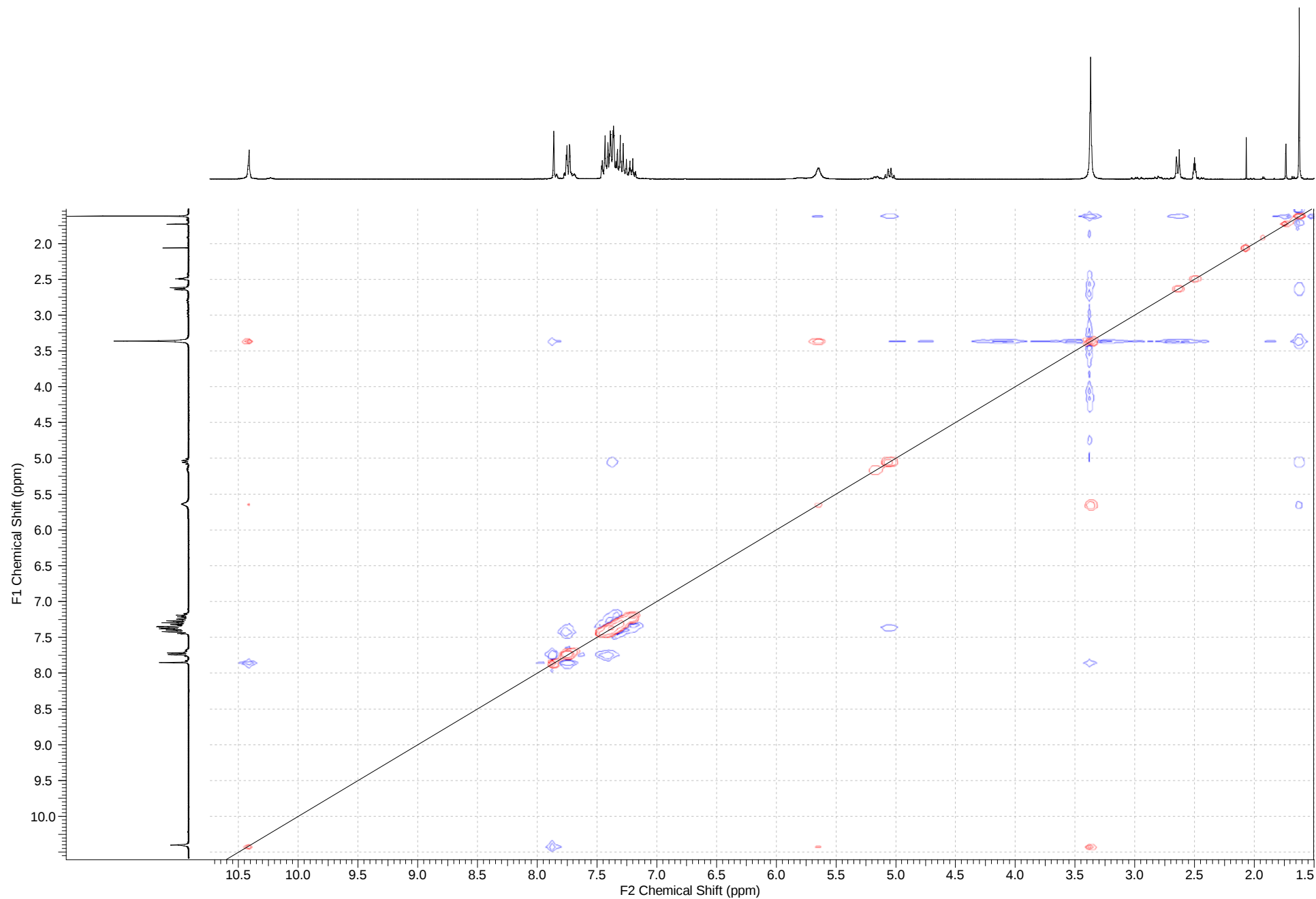
^1H NMR spectrum of compound **16b** (DMSO- d_6).



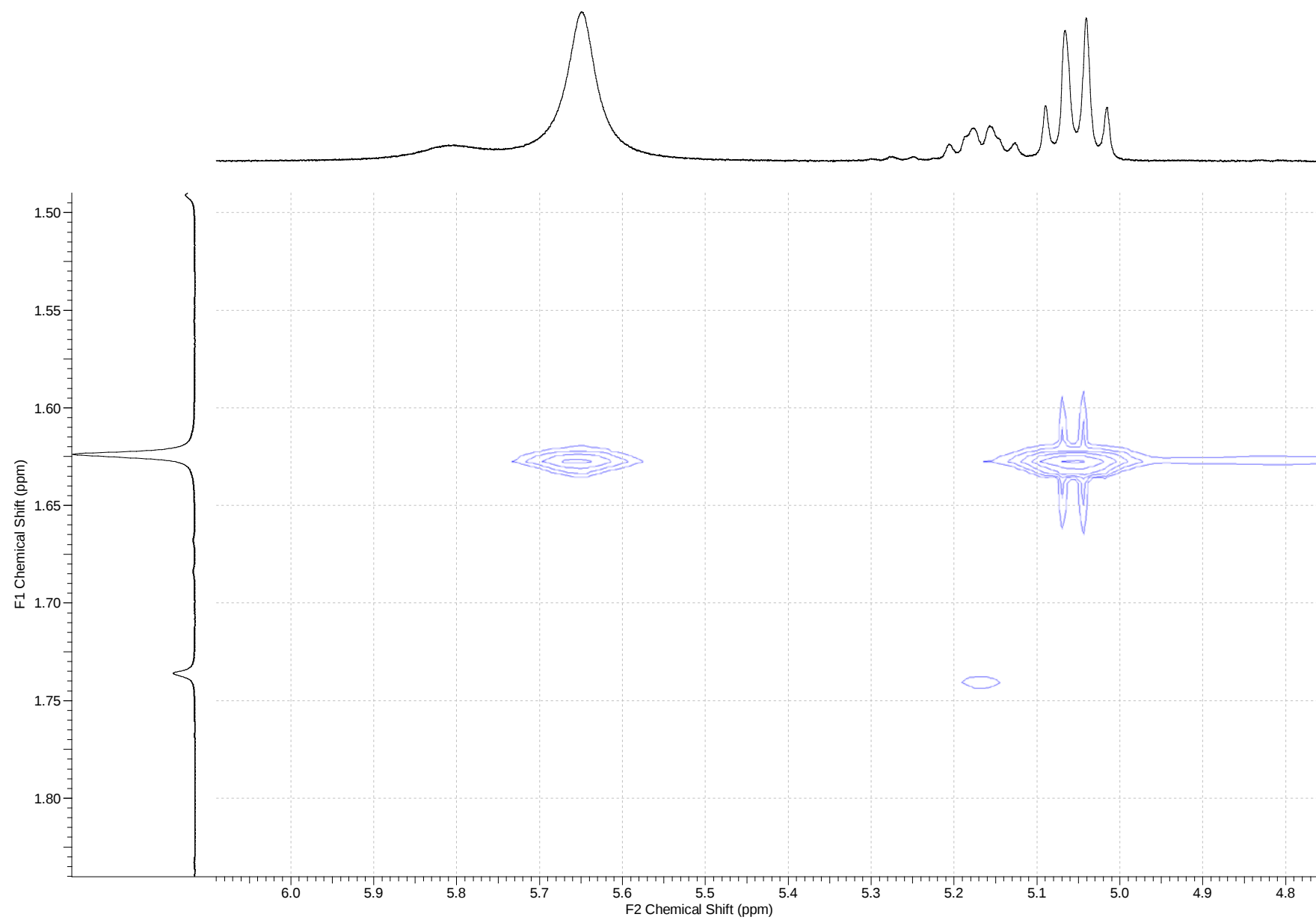
^{13}C NMR spectrum of compound **16b** ($\text{DMSO}-d_6$).



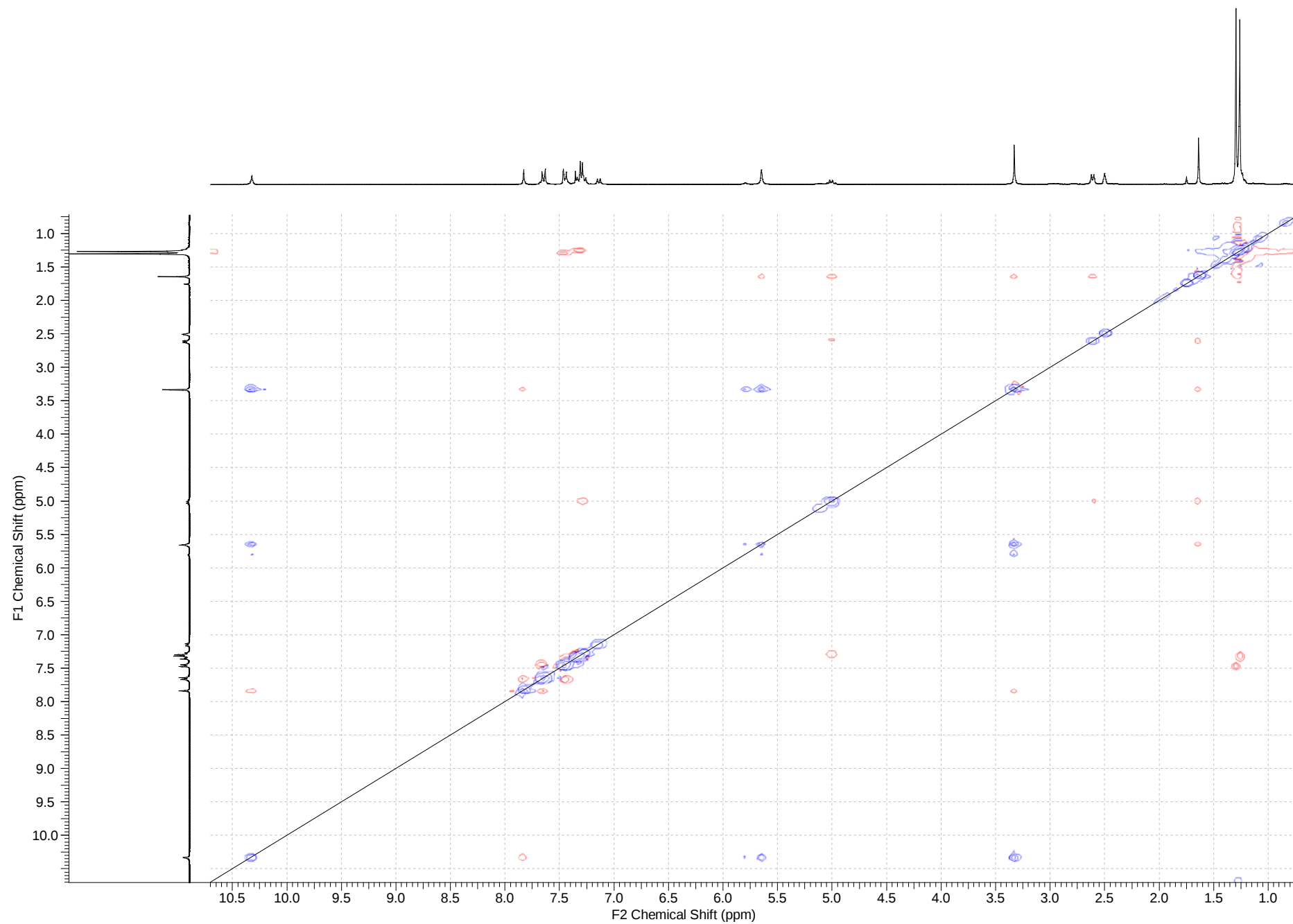
^1H , ^1H NOESY spectrum of a 84:16 mixture of (*E*)- and (*Z*)-**14a** (DMSO- d_6)



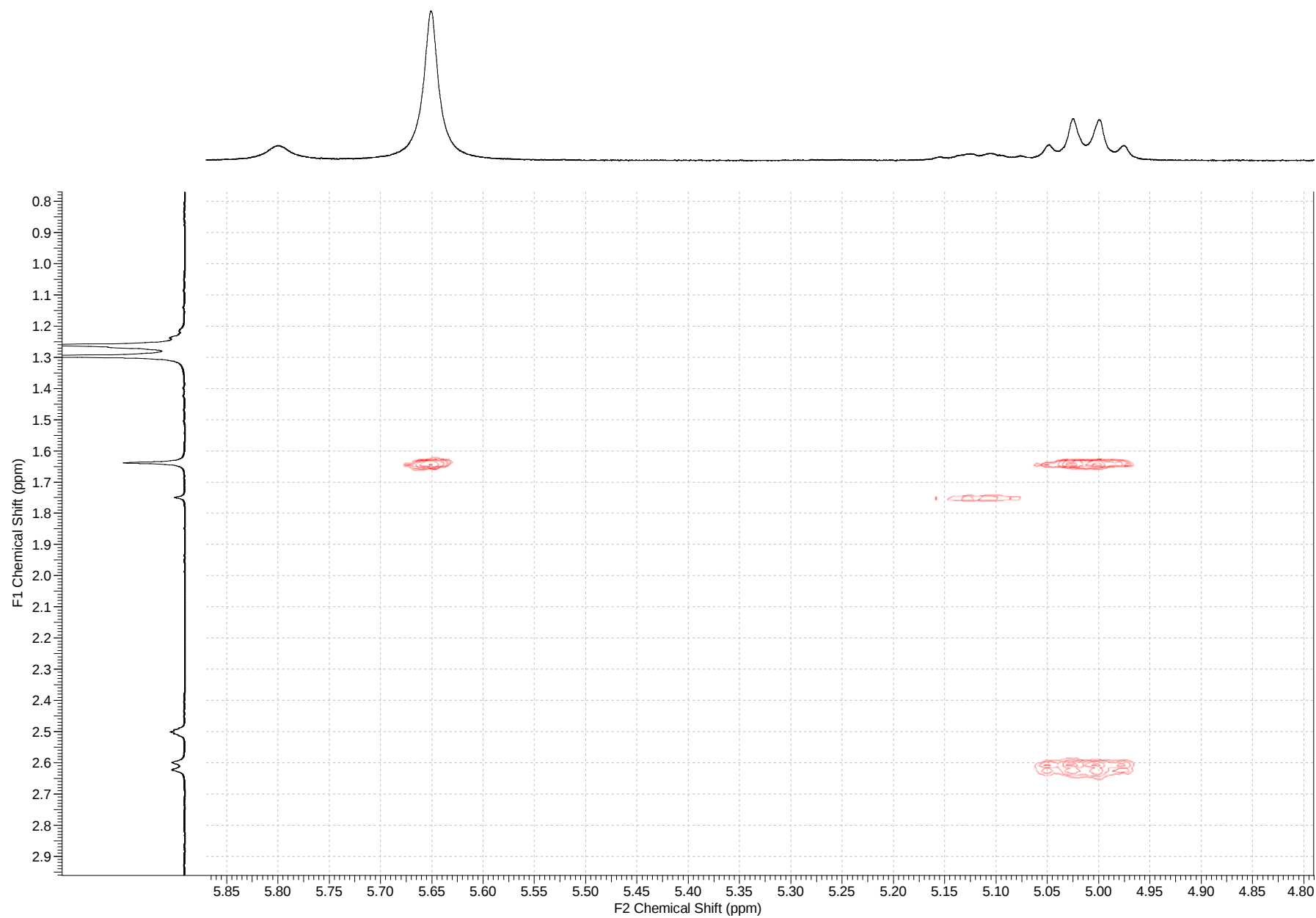
^1H , ^1H NOESY spectrum of a 84:16 mixture of (*E*)- and (*Z*)-**14a** (DMSO- d_6)



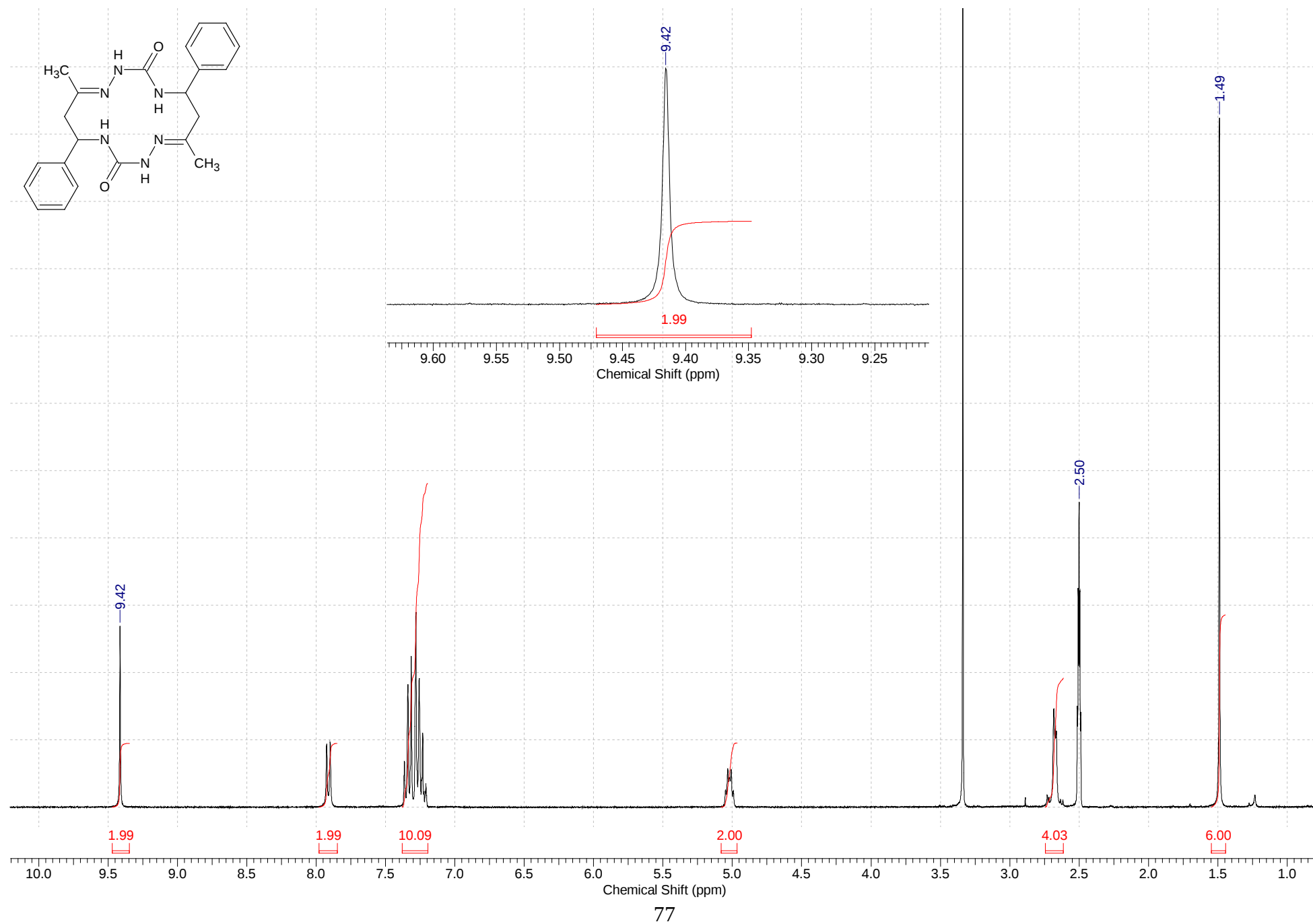
^1H , ^1H NOESY spectrum of a 86:14 mixture of (*E*)- and (*Z*)-14c (DMSO- d_6)



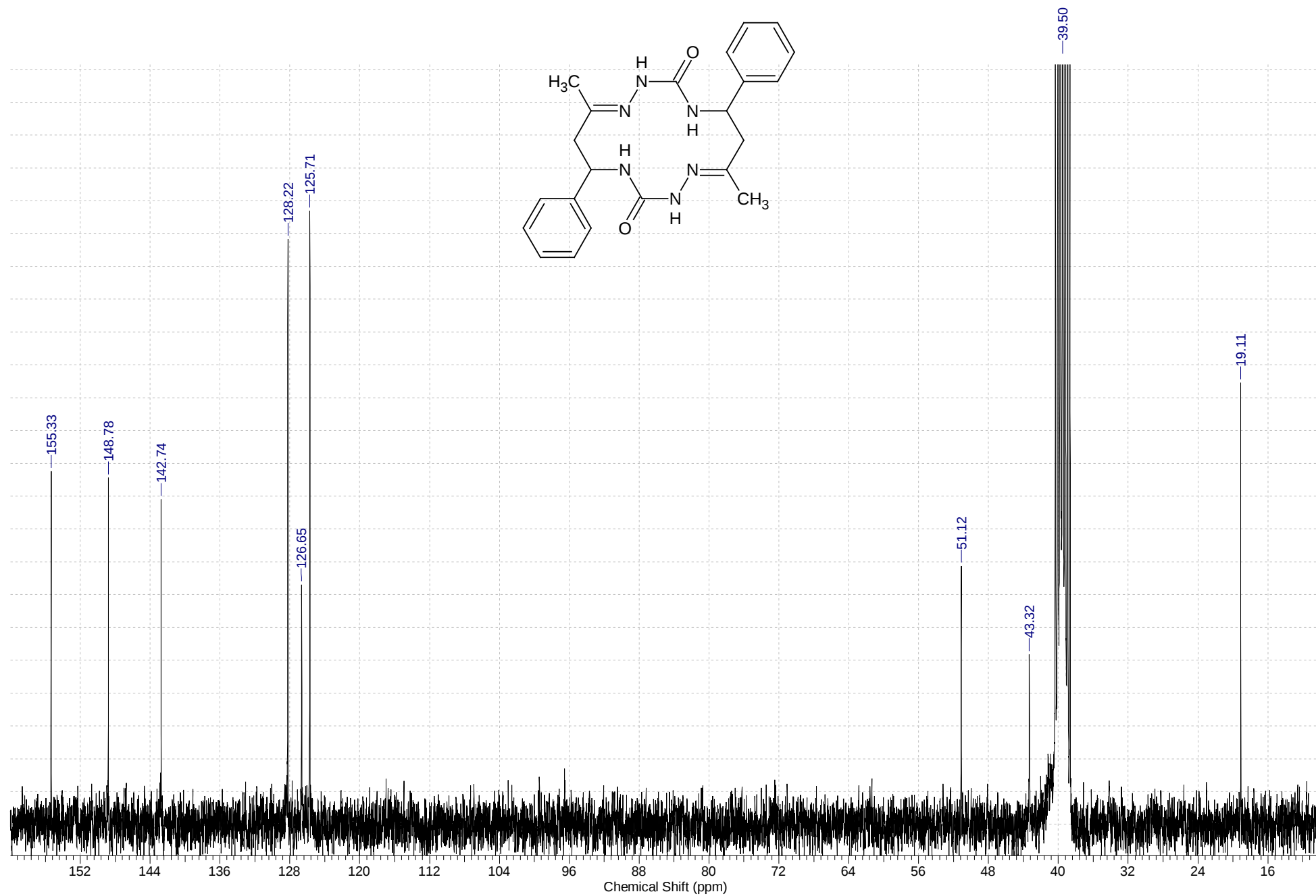
^1H , ^1H NOESY spectrum of a 86:14 mixture of (*E*)- and (*Z*)-14c (DMSO- d_6)



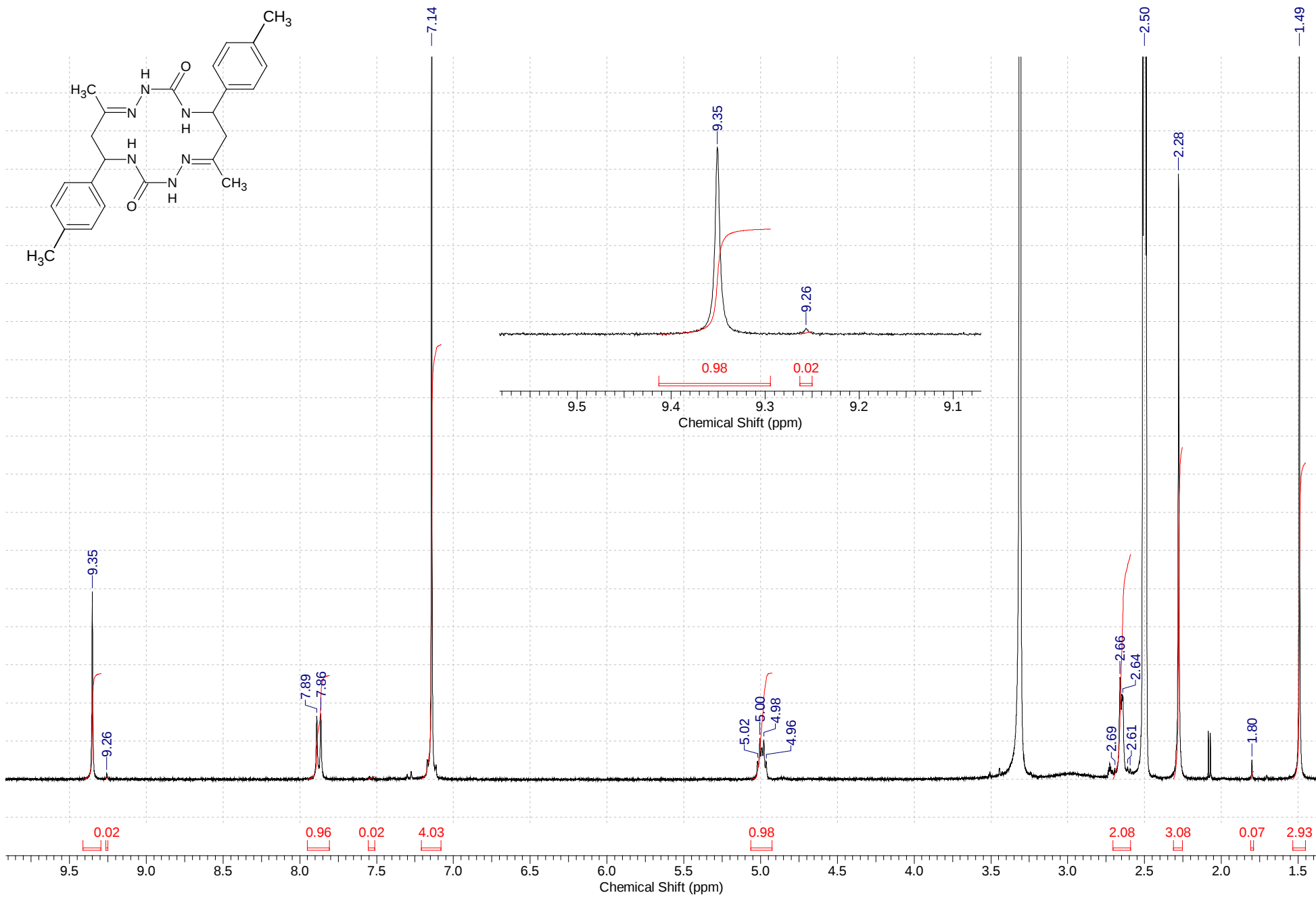
¹H NMR spectrum of compound **17a** (DMSO-*d*₆).



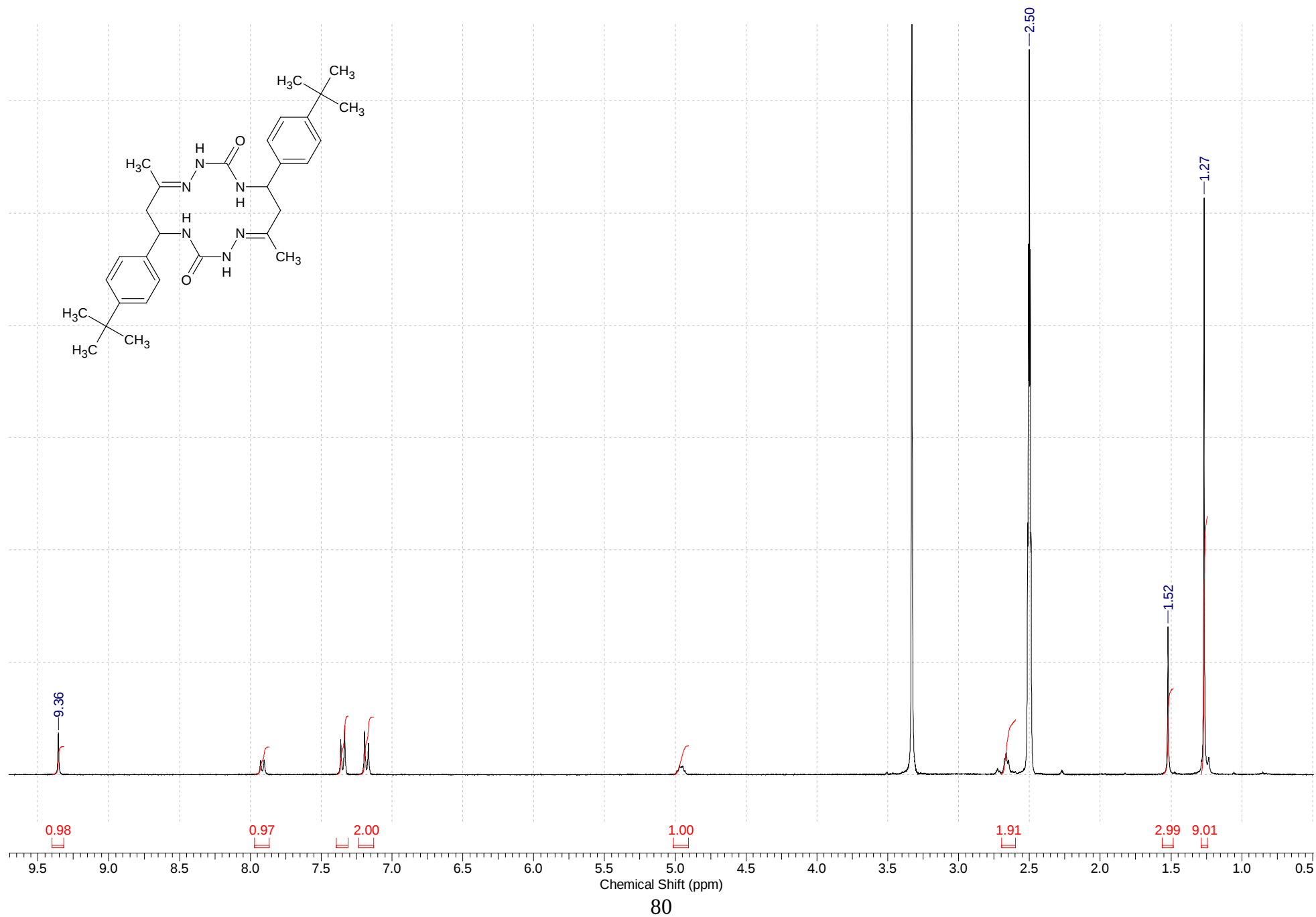
^{13}C NMR spectrum of compound **17a** ($\text{DMSO-}d_6$).



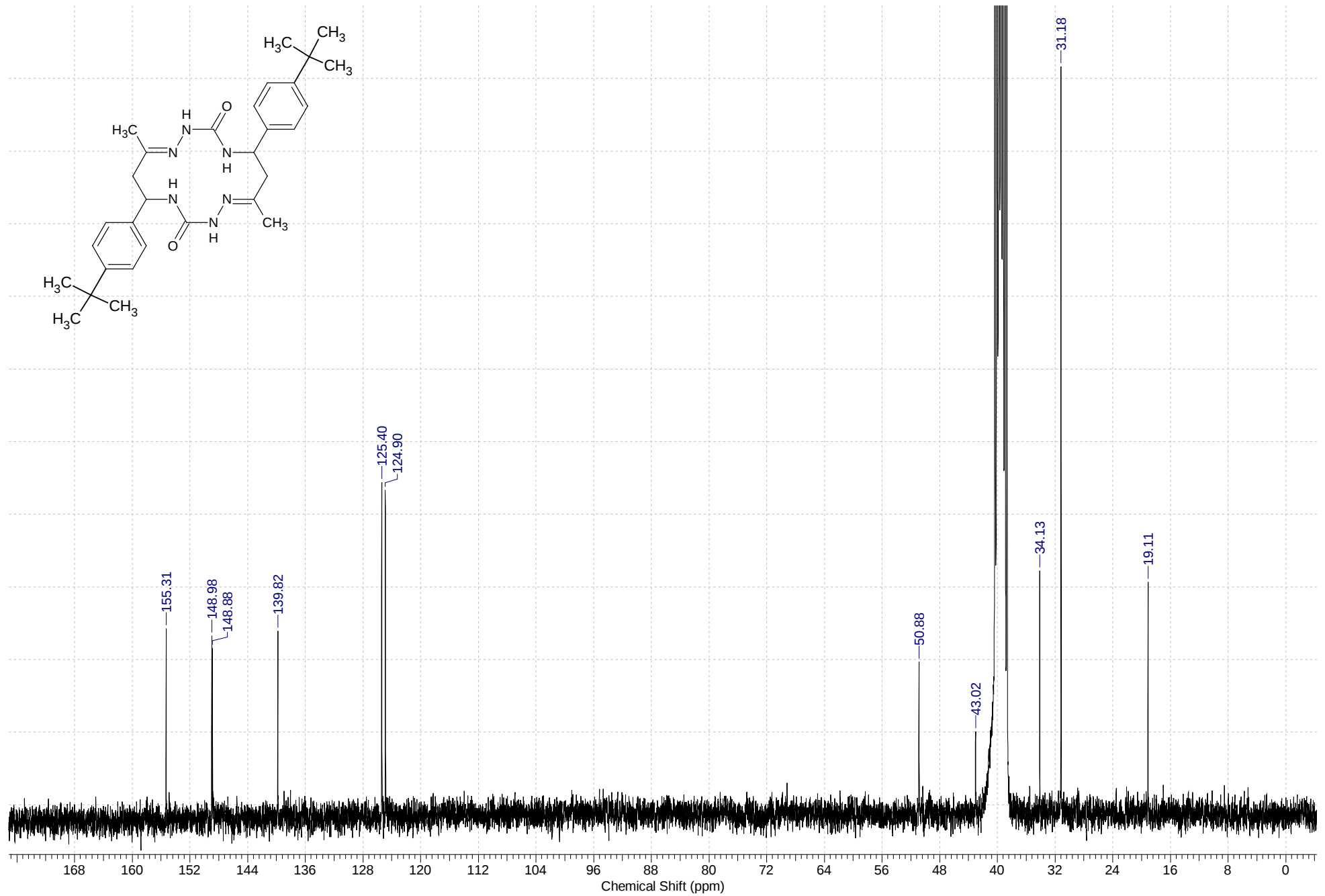
¹H NMR spectrum of crude compound **17b** (DMSO-*d*₆).



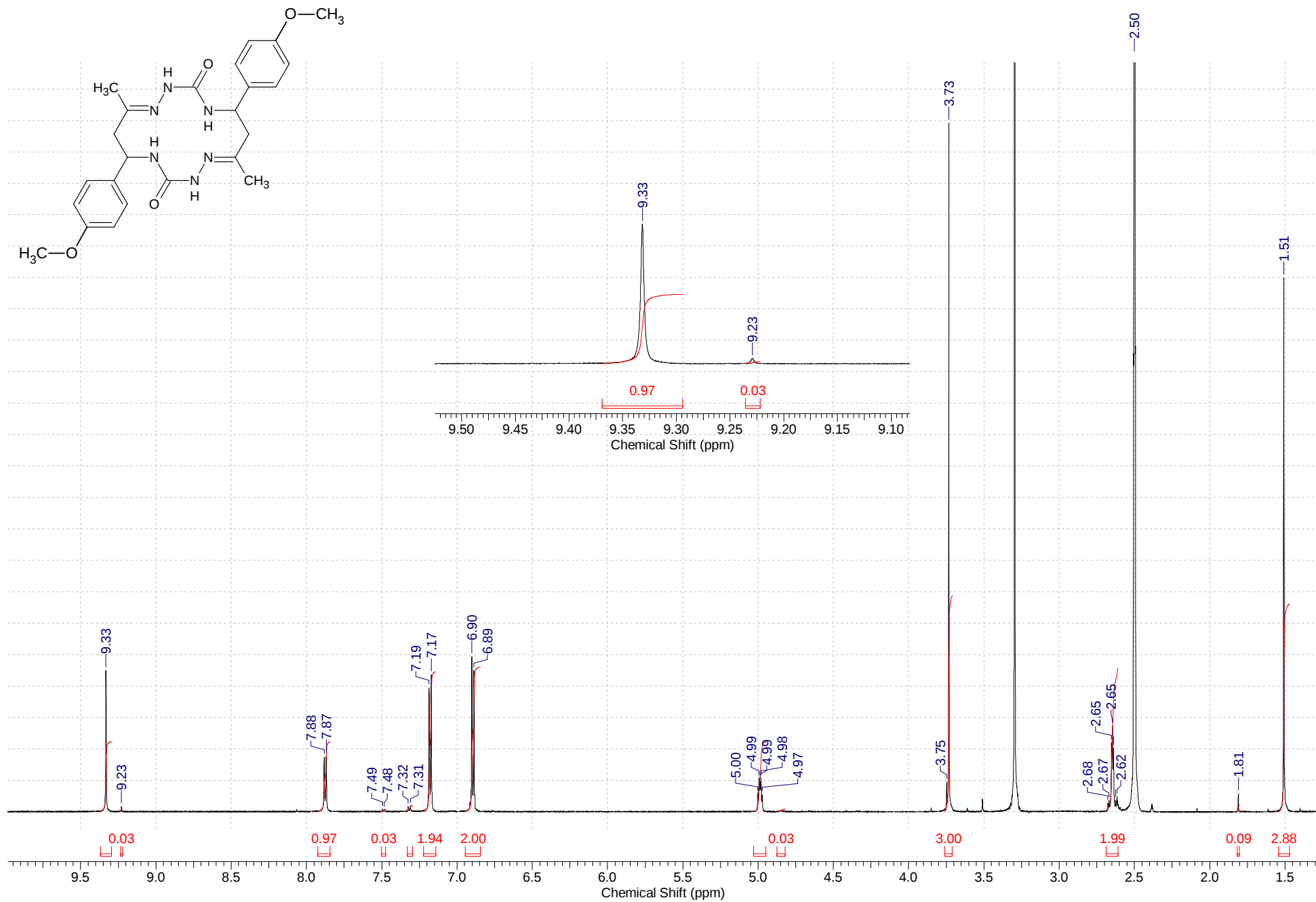
^1H NMR spectrum of crude compound **17c** (DMSO- d_6).



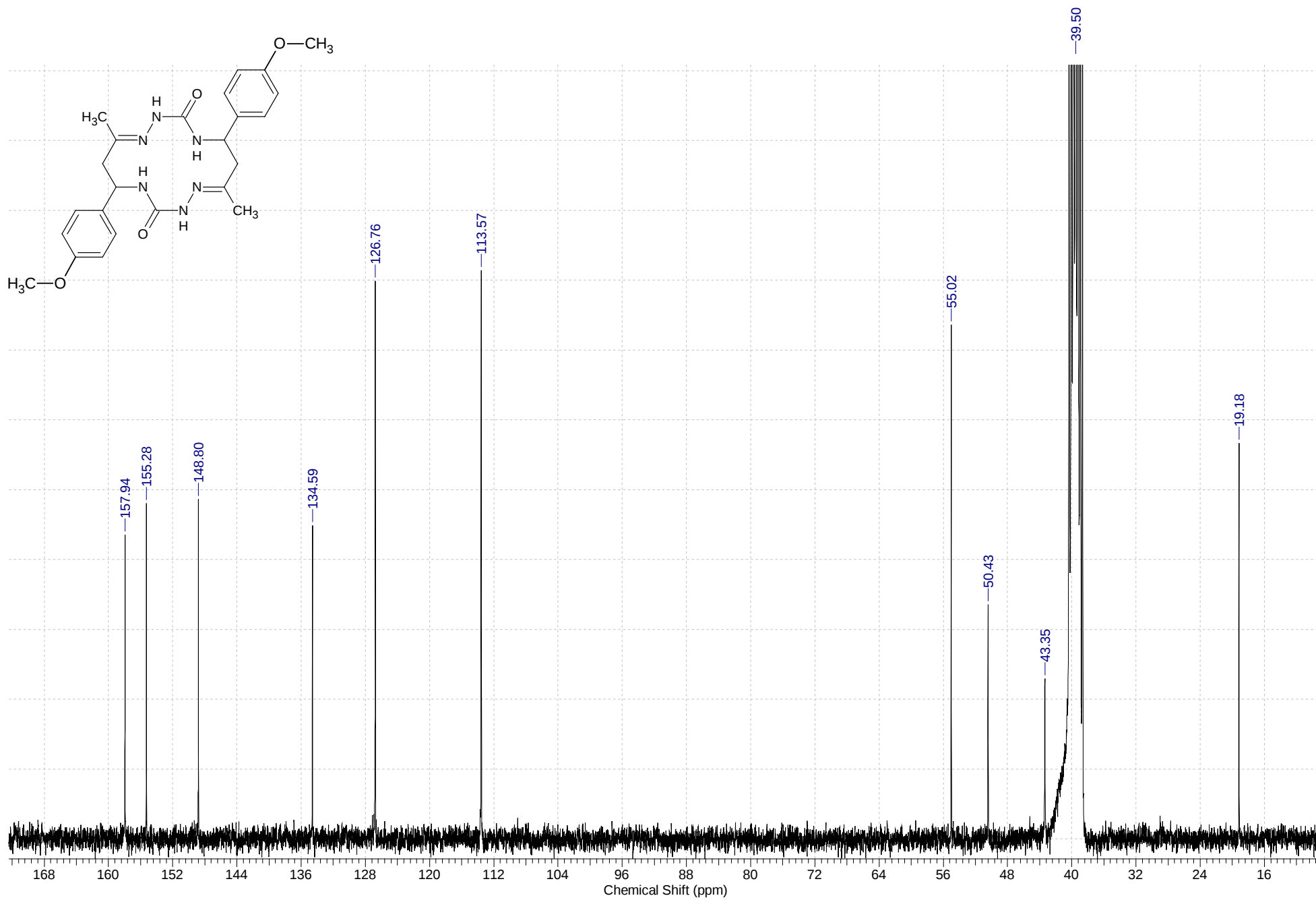
^{13}C NMR spectrum of crude compound **17c** (DMSO- d_6).



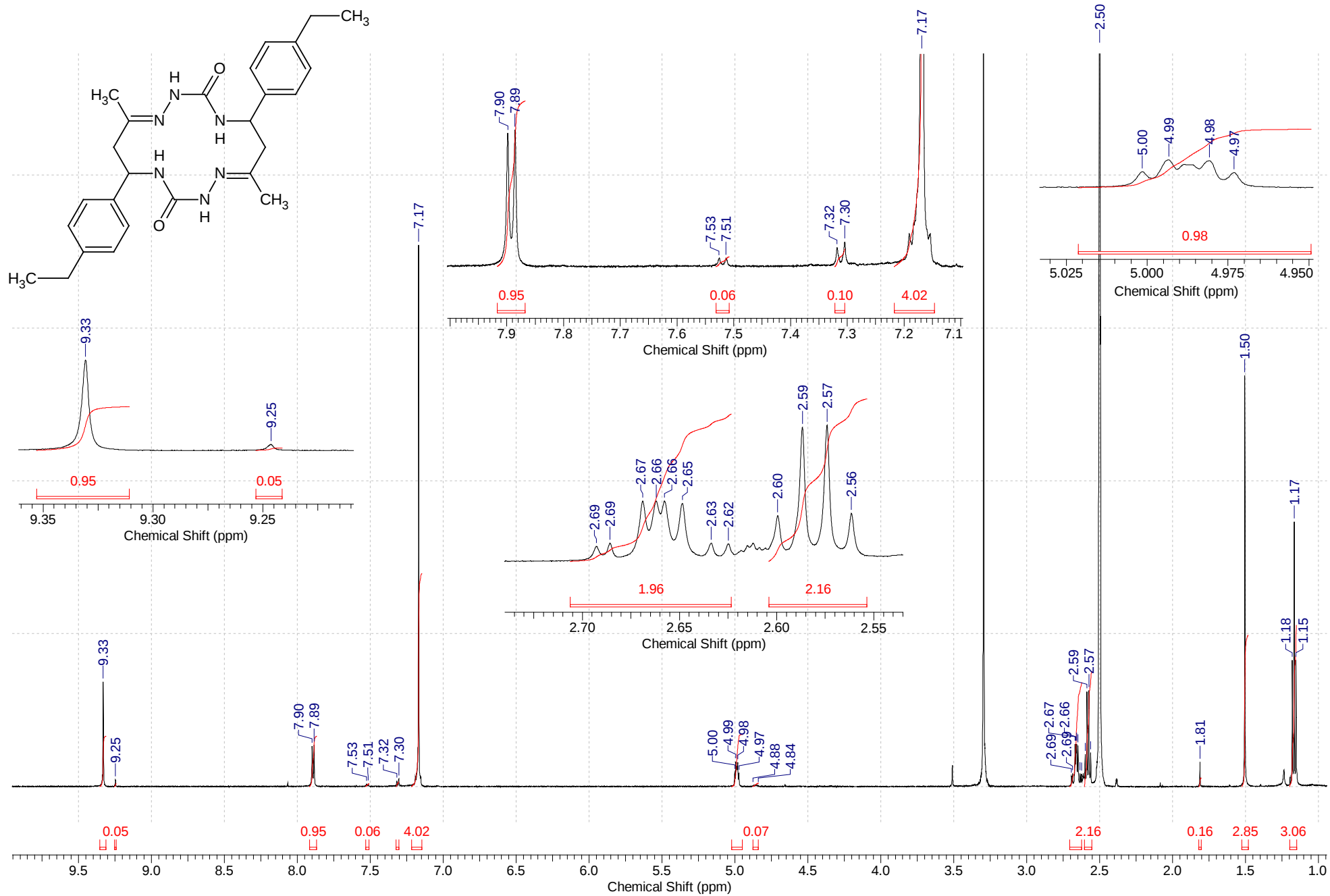
¹H NMR spectrum of crude compound **17d** (DMSO-*d*₆).



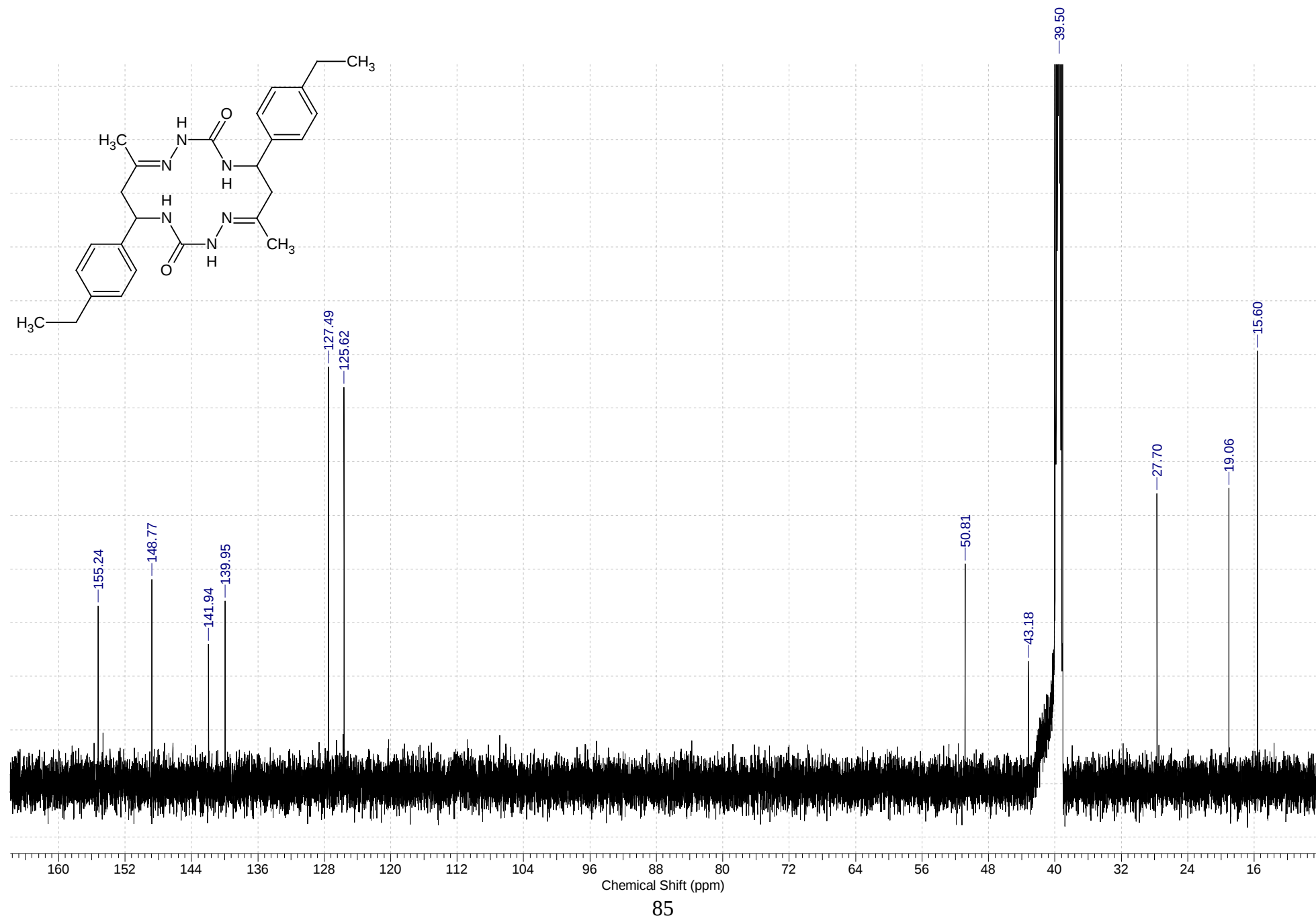
^{13}C NMR spectrum of crude compound **17d** (DMSO- d_6).



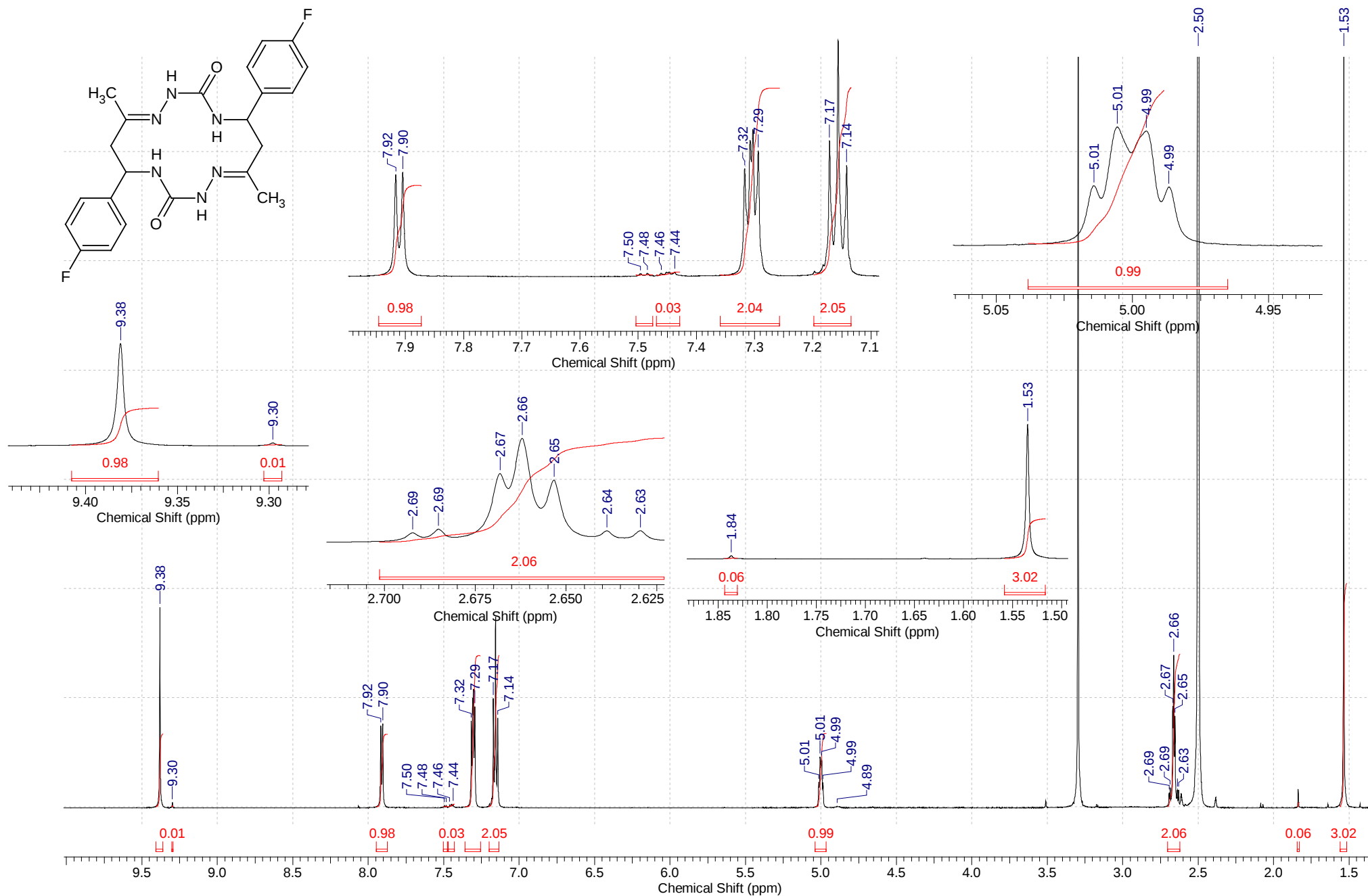
¹H NMR spectrum of crude compound **17e** (DMSO-*d*₆).



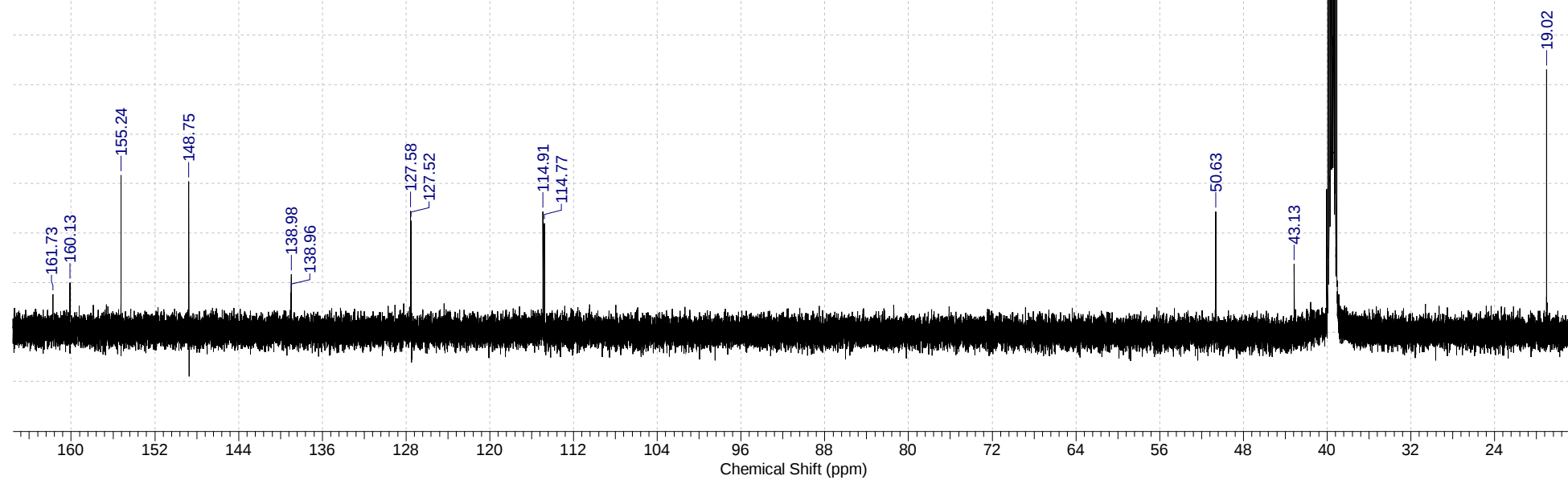
¹³C NMR spectrum of crude compound **17e** (DMSO-*d*₆).



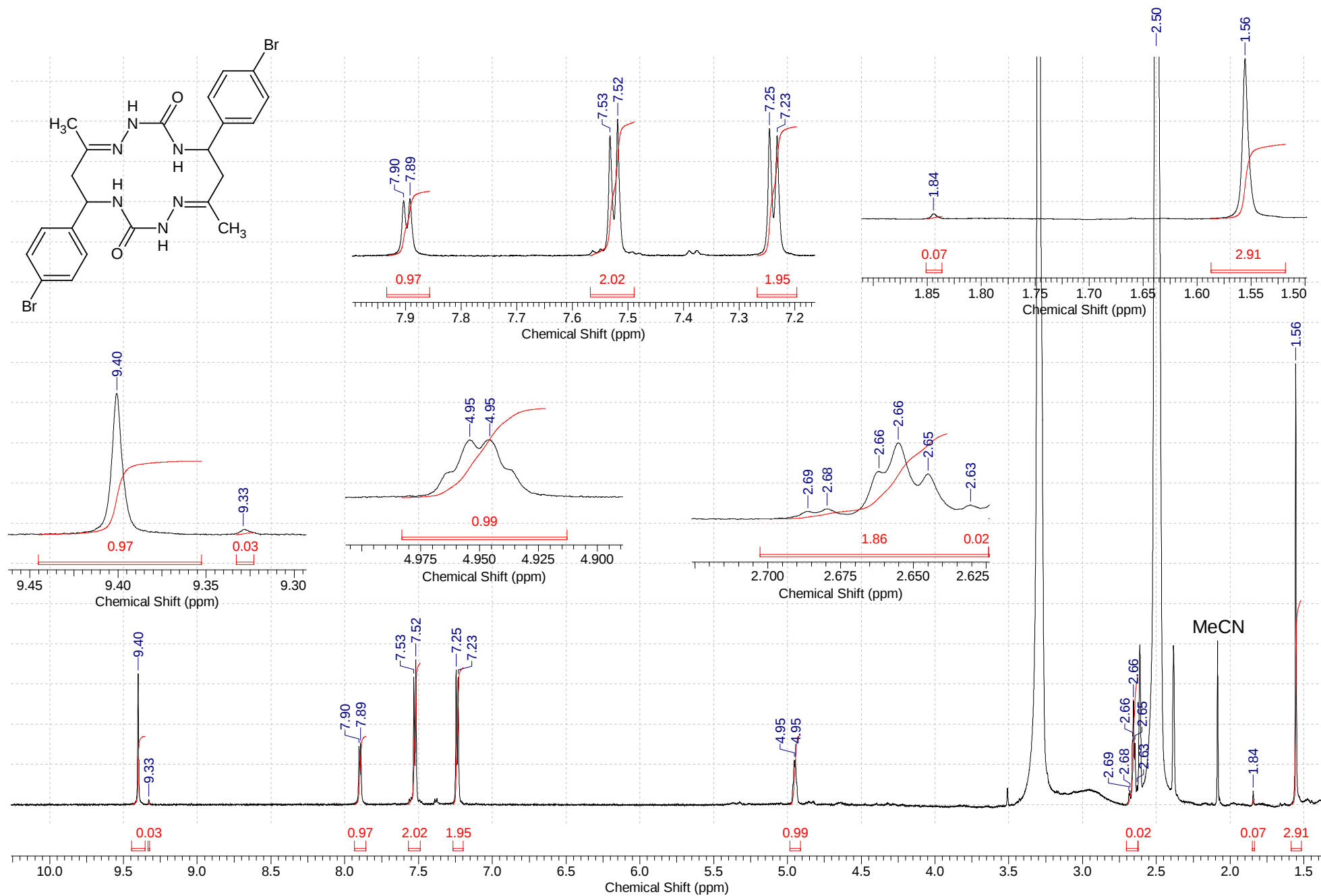
^1H NMR spectrum of crude compound **17f** (DMSO- d_6).



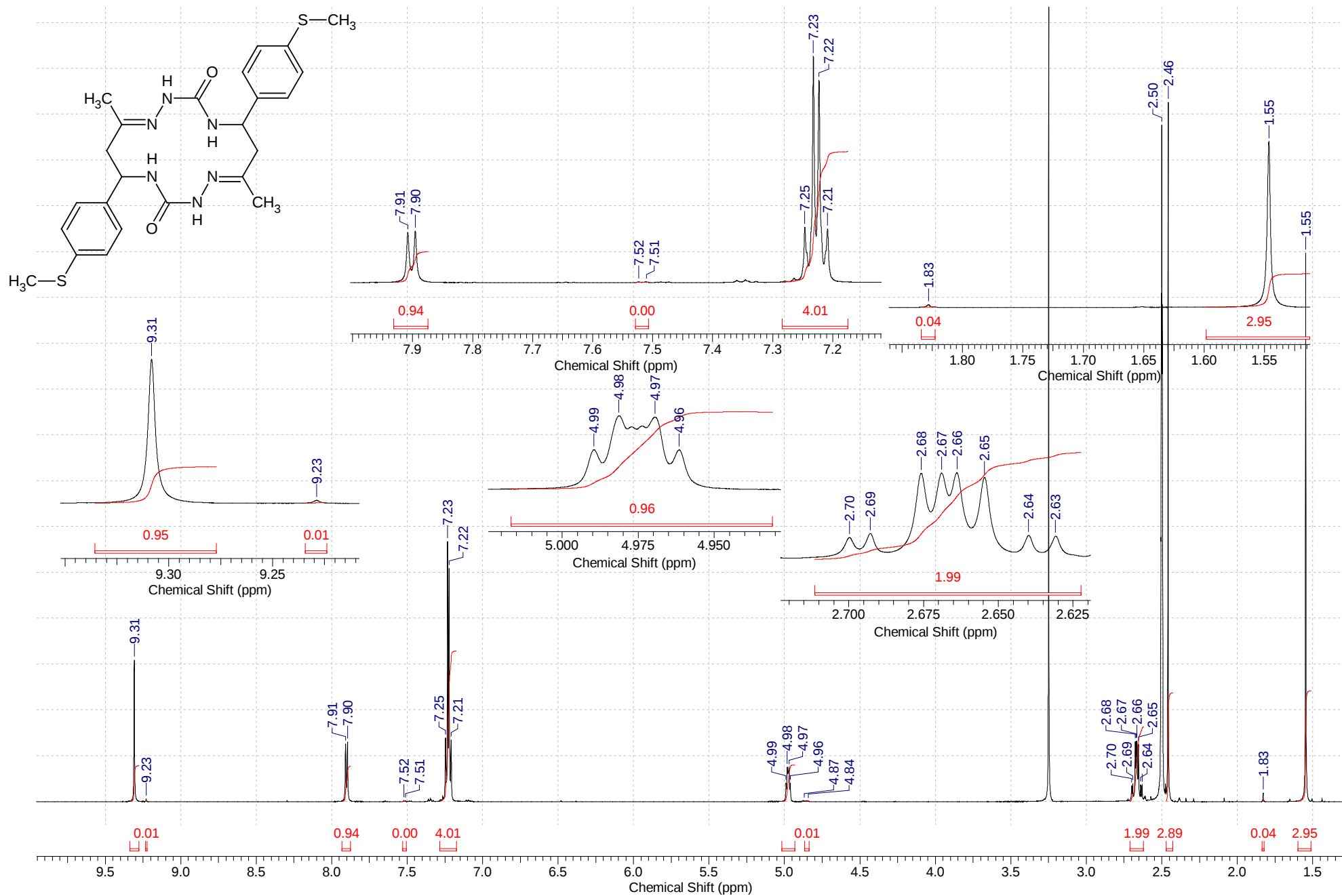
^{13}C NMR spectrum of crude compound **17f** ($\text{DMSO-}d_6$).



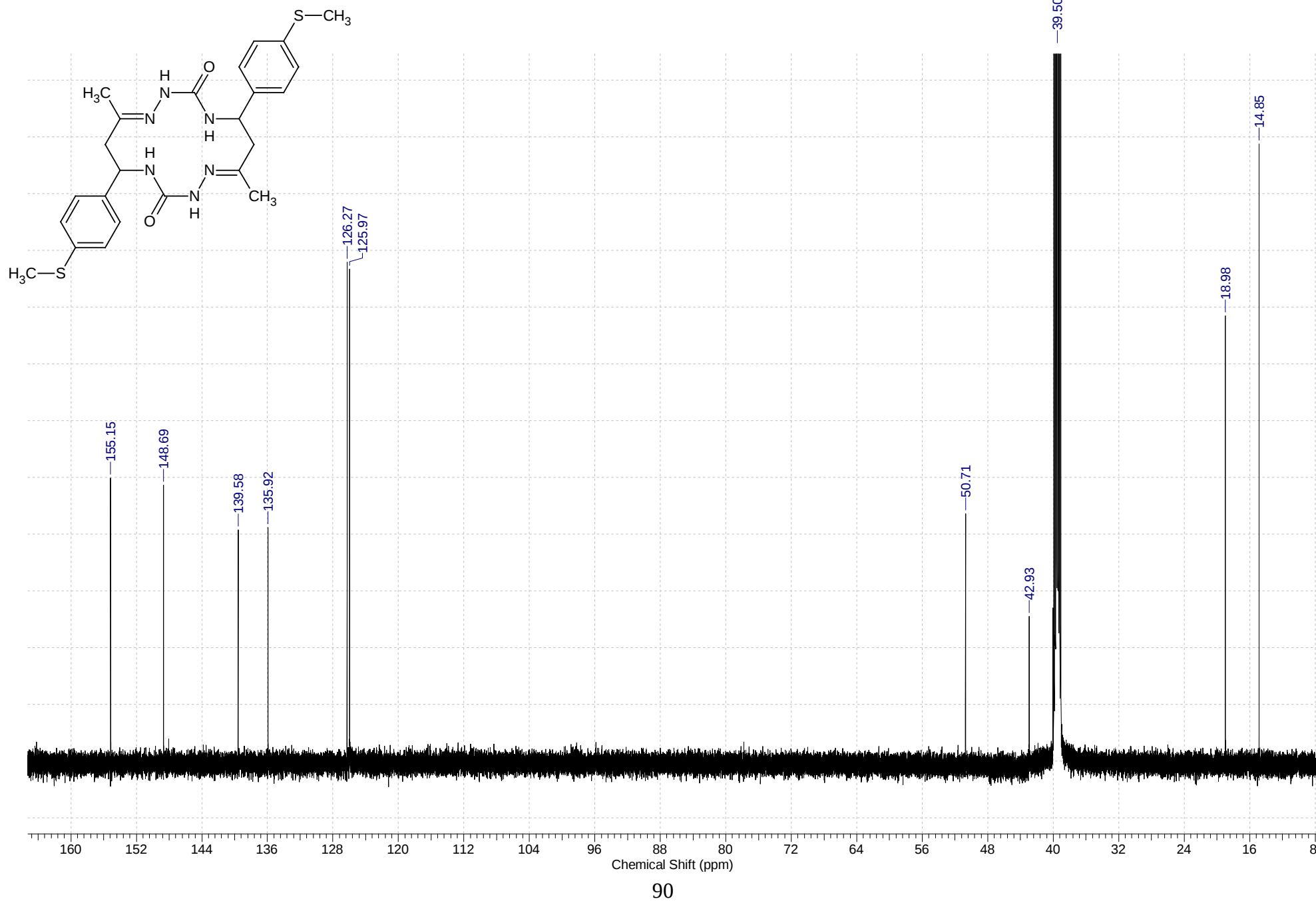
¹H NMR spectrum of crude compound **17g** (DMSO-*d*₆).



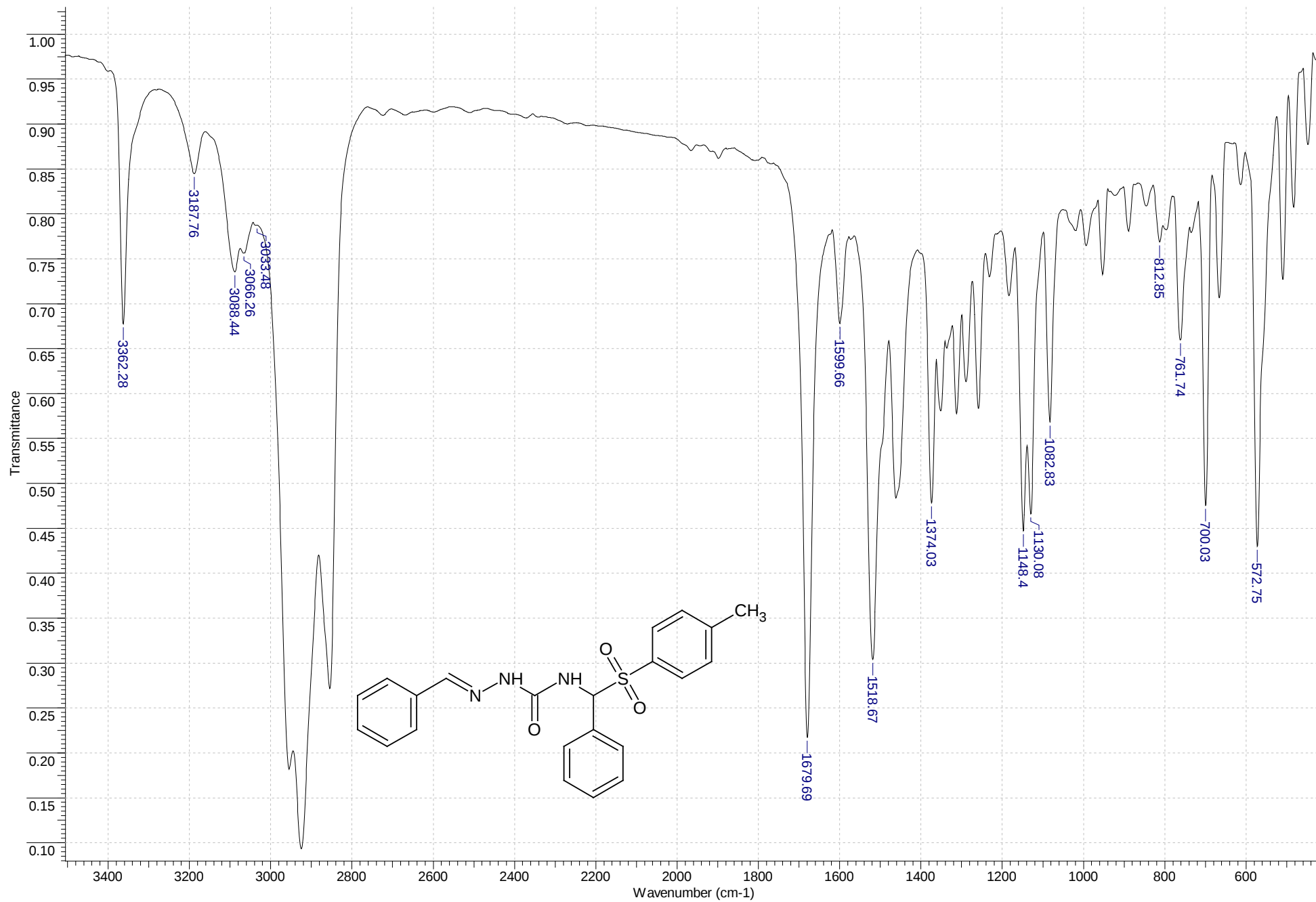
¹H NMR spectrum of crude compound **17h** (DMSO-*d*₆).



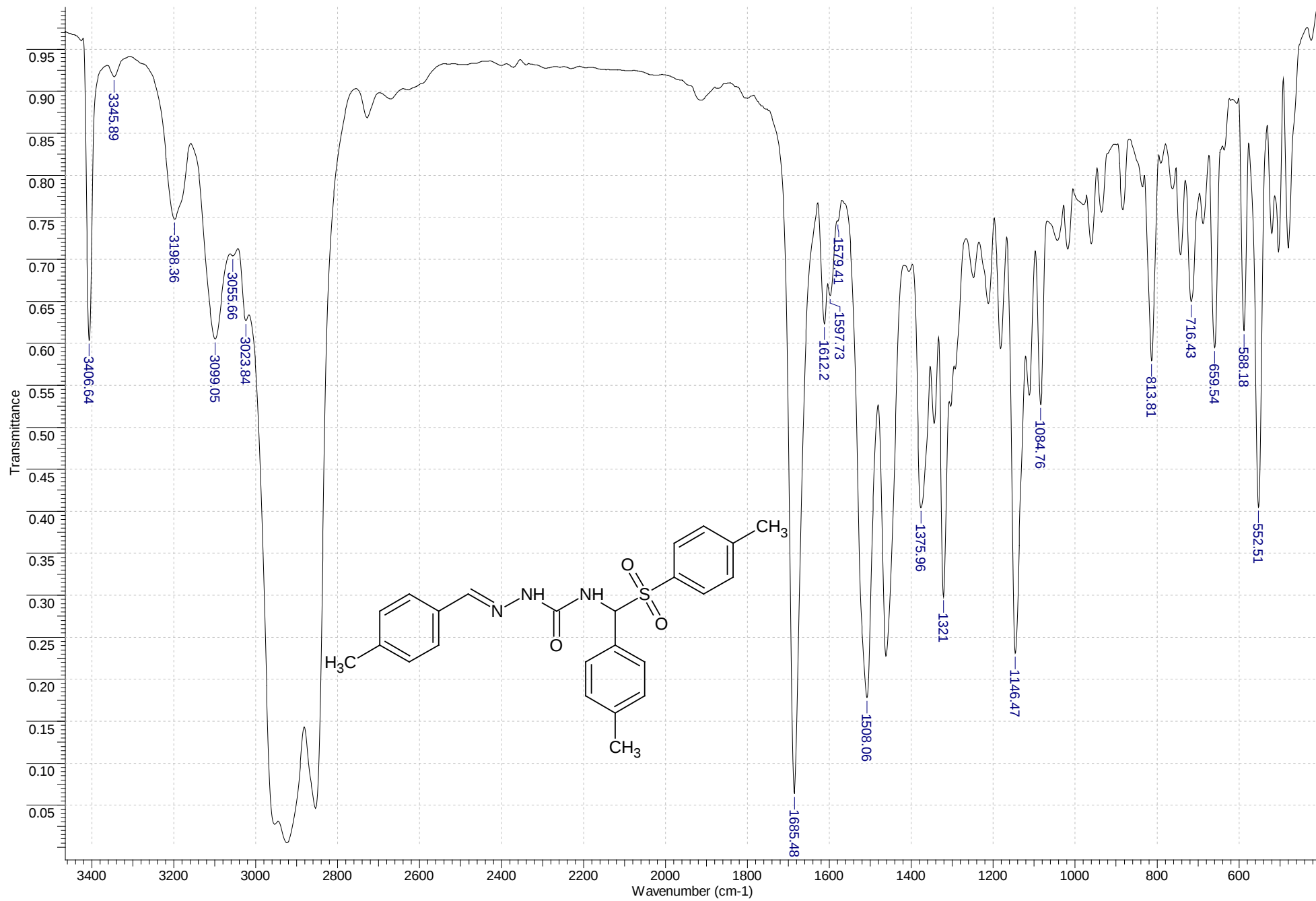
^{13}C NMR spectrum of crude compound **17h** (DMSO- d_6).



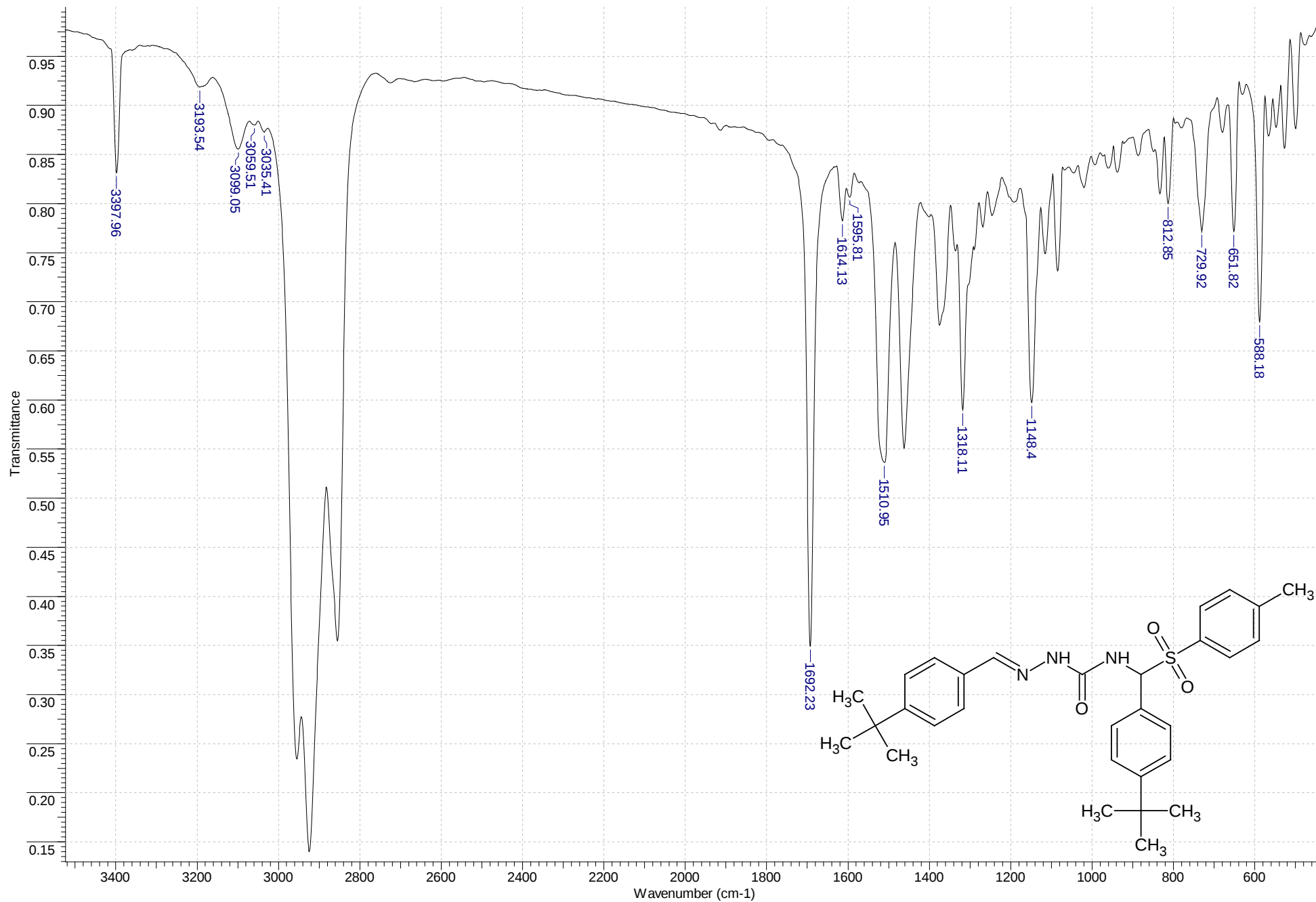
IR spectrum of compound 5a (Nujol).



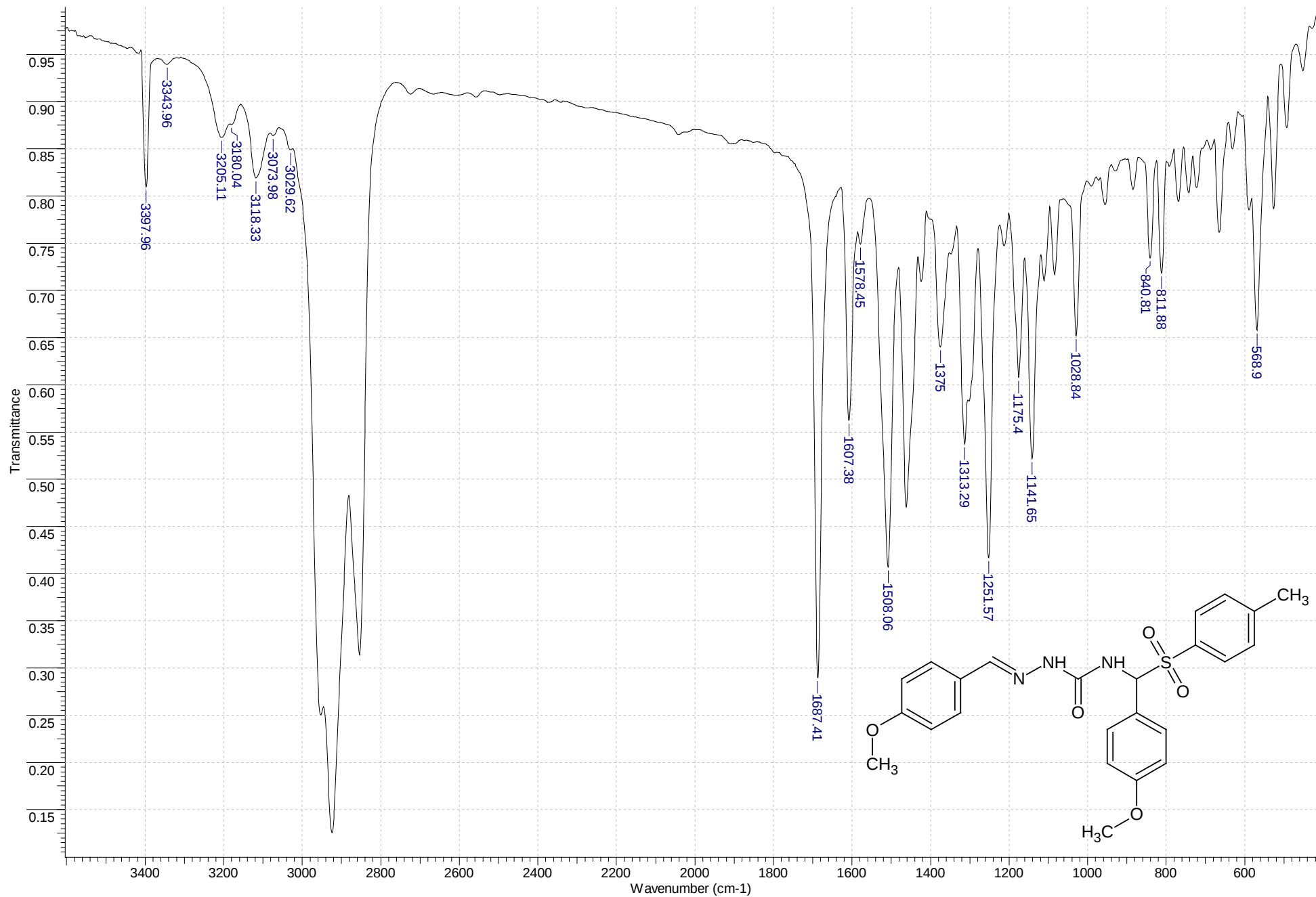
IR spectrum of compound **5b** (Nujol).



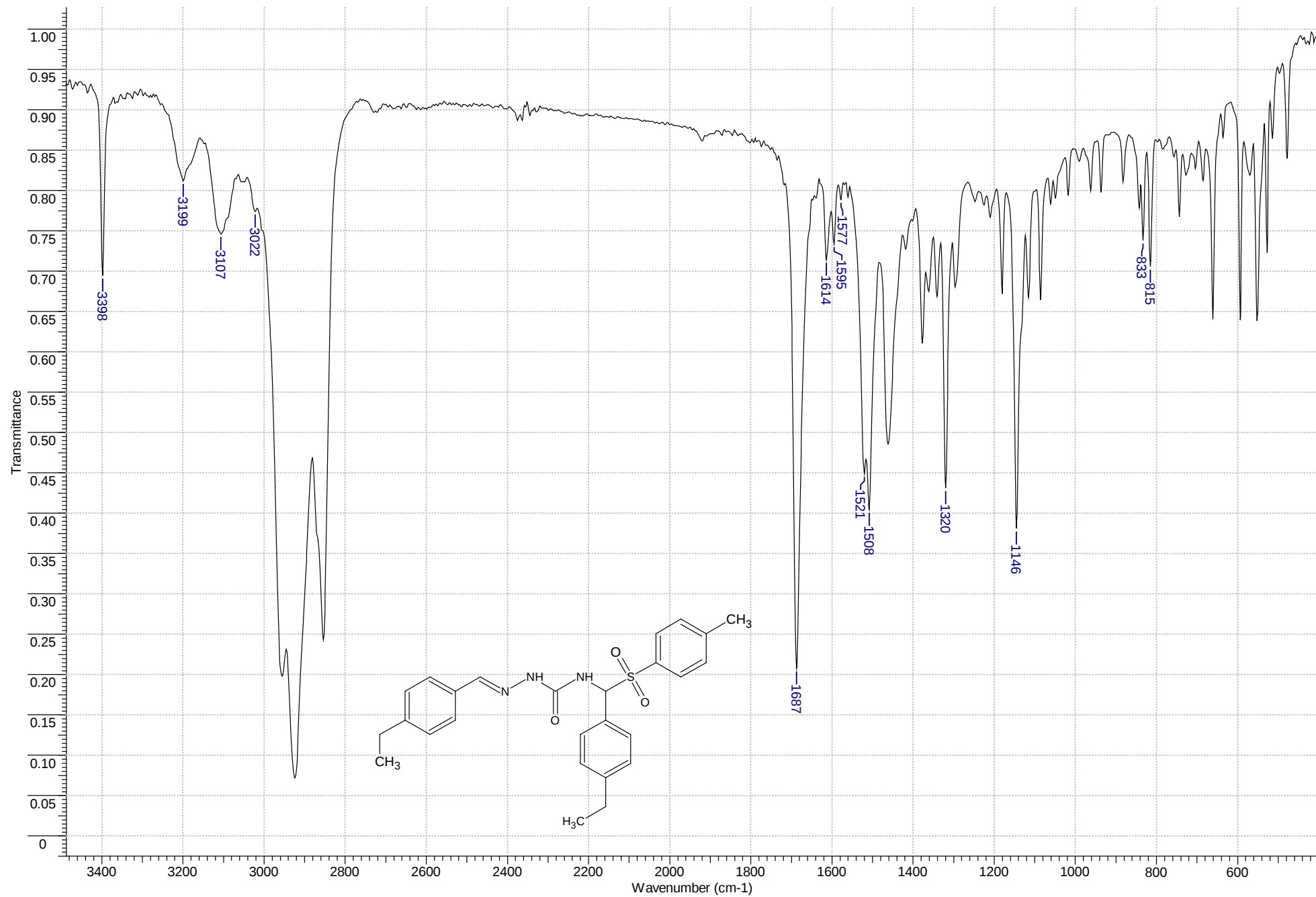
IR spectrum of compound 5c (Nujol).



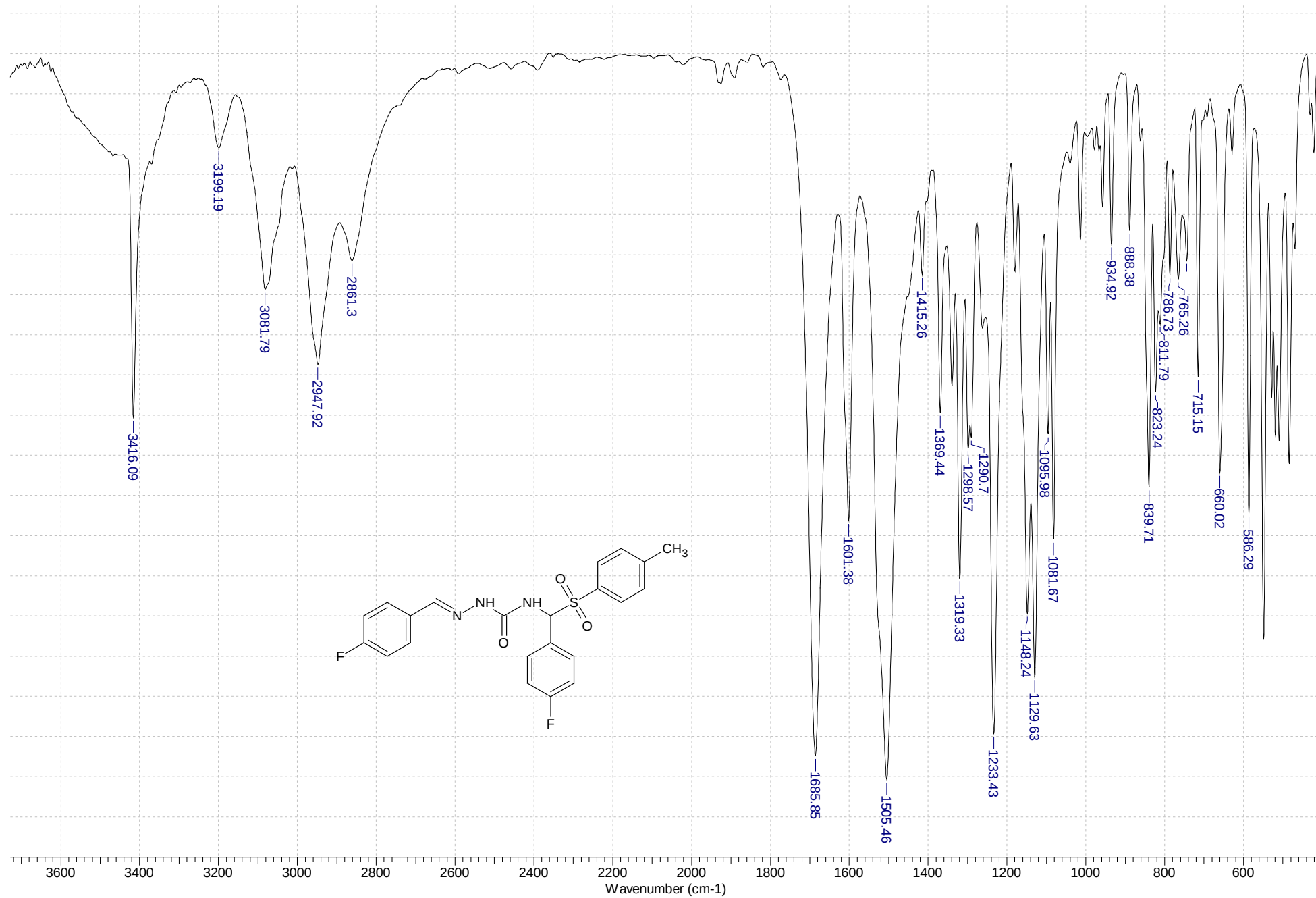
IR spectrum of compound **5d** (Nujol).



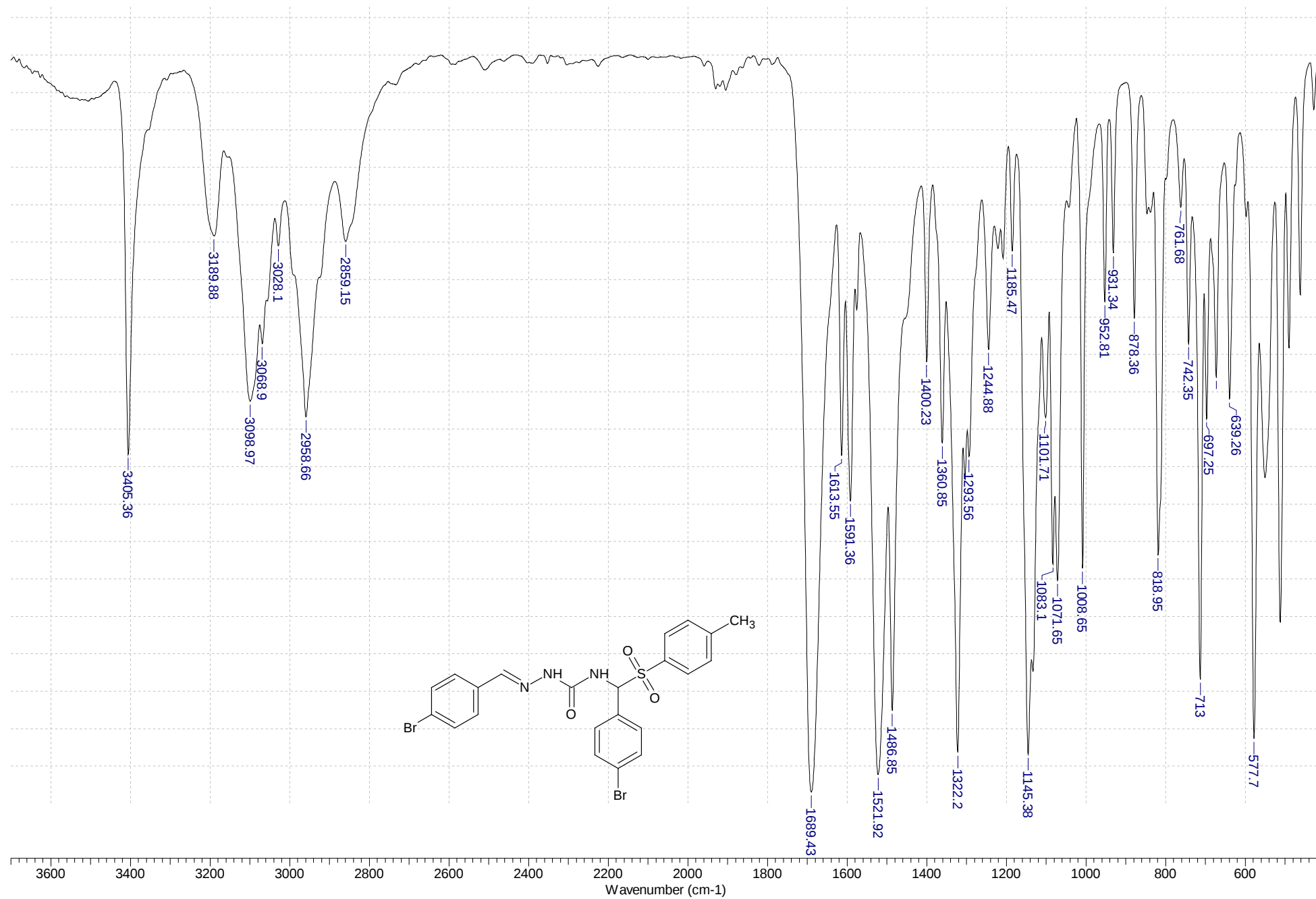
IR spectrum of compound 5e (Nujol).



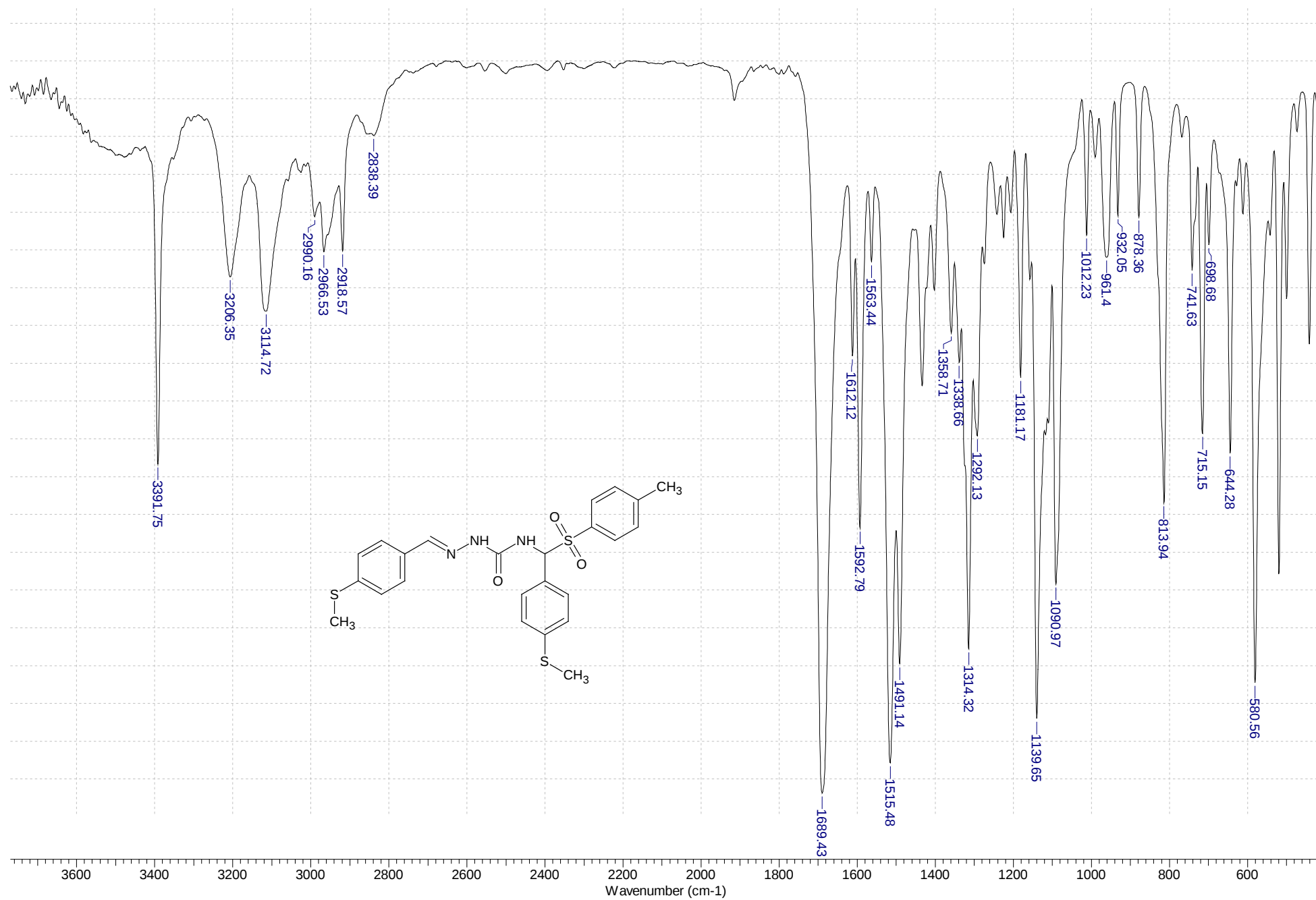
IR spectrum of compound 5f (KBr).



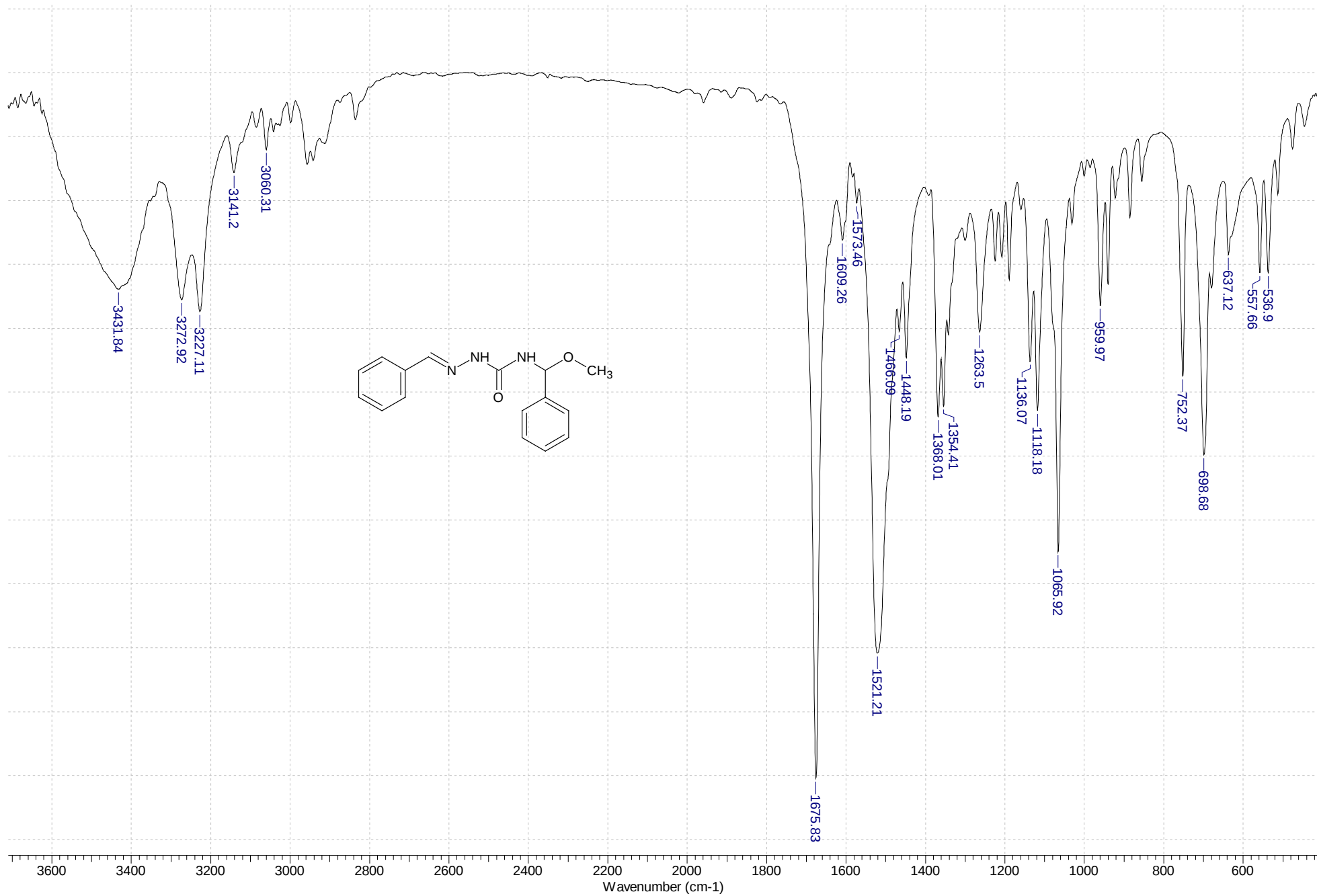
IR spectrum of compound 5g (KBr).



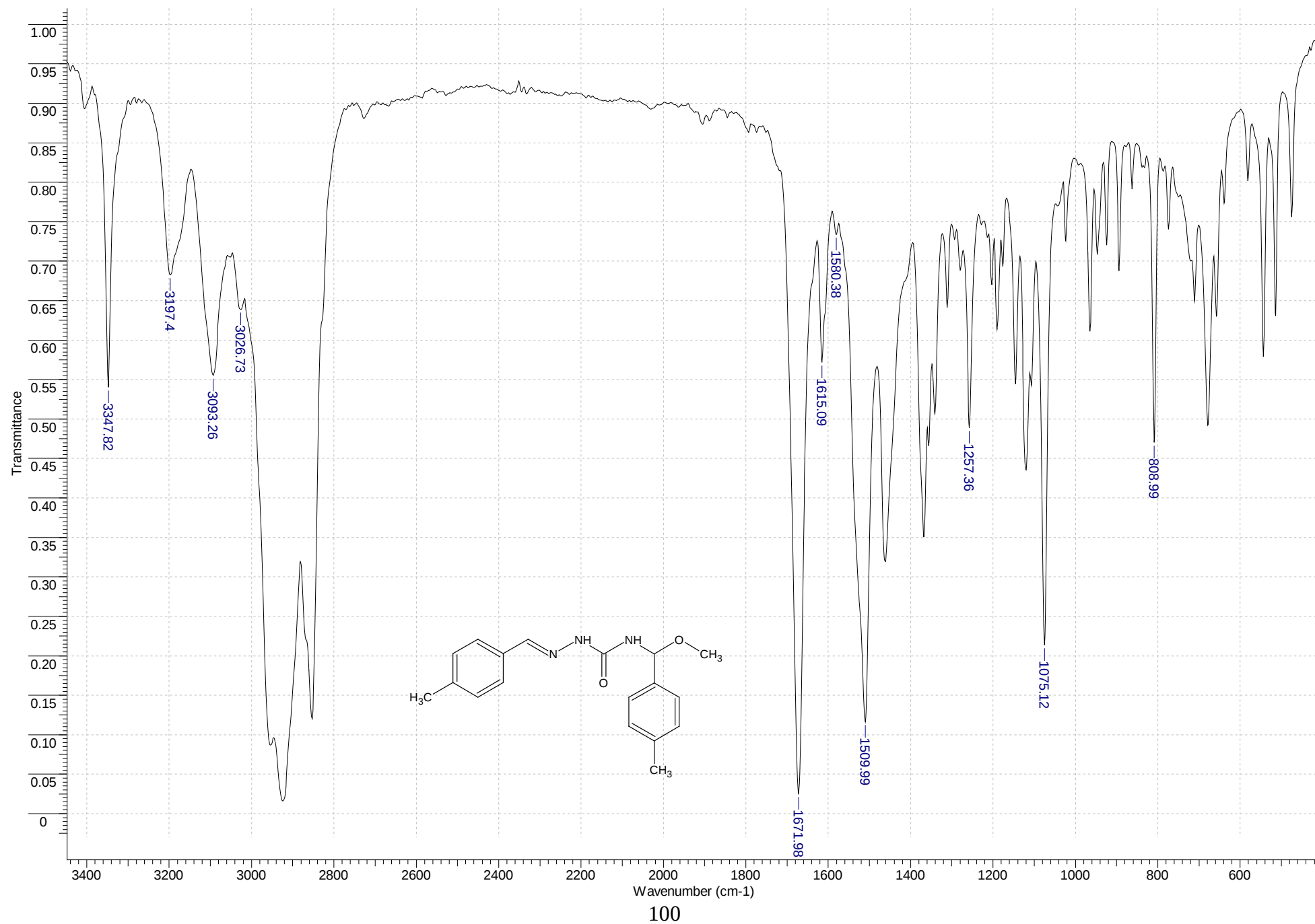
IR spectrum of compound **5h** (KBr).



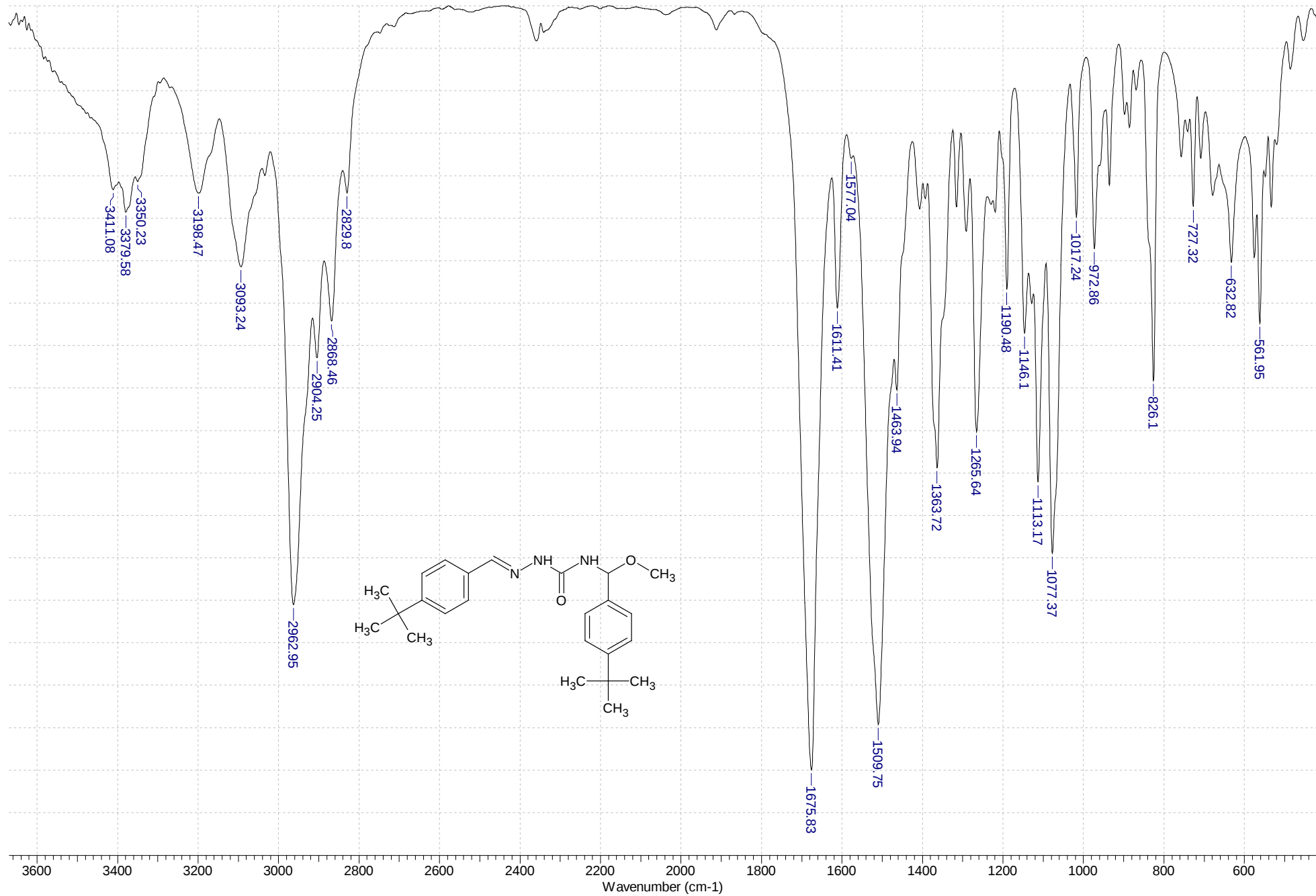
IR spectrum of compound **9a** (KBr).



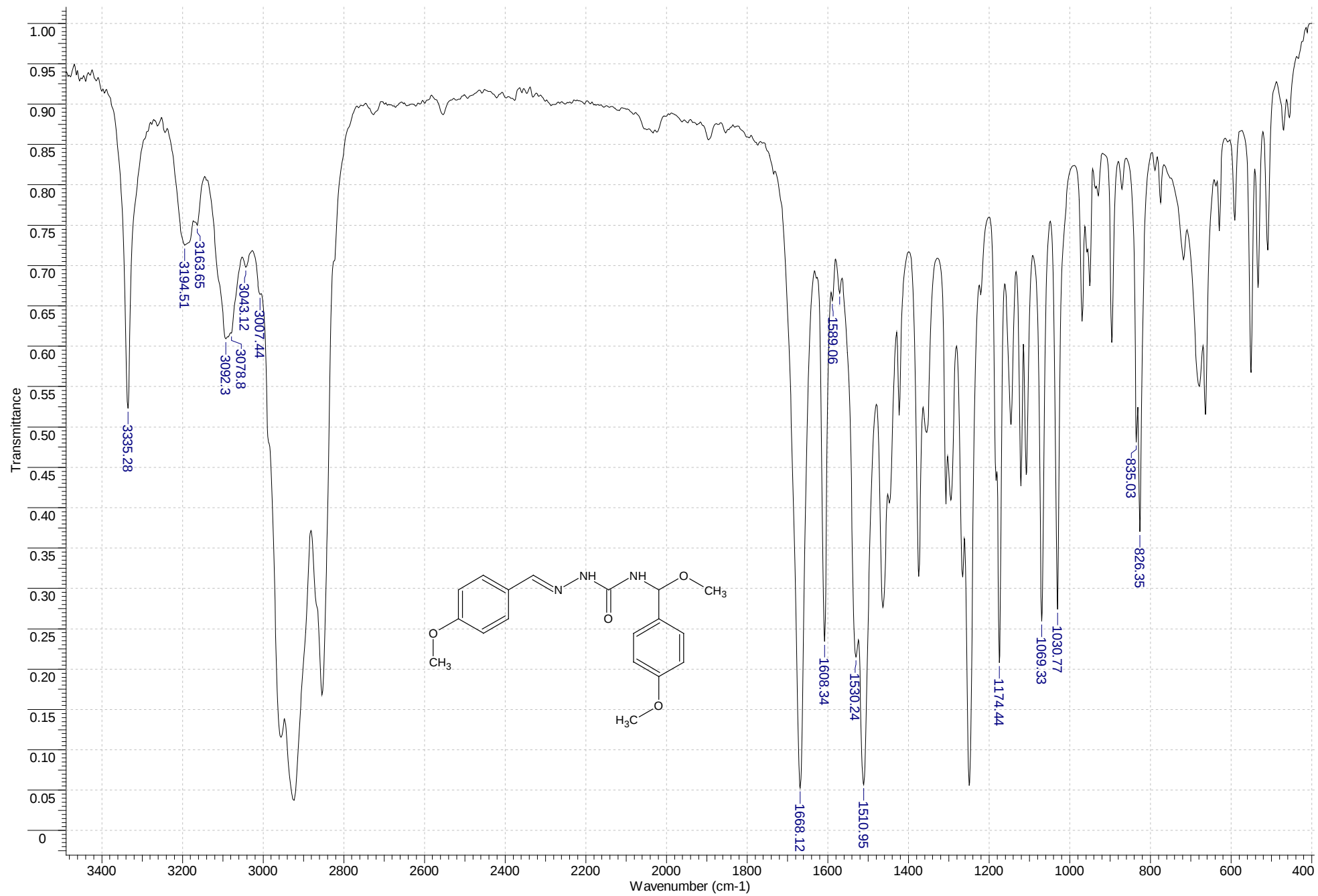
IR spectrum of compound **9b** (Nujol).



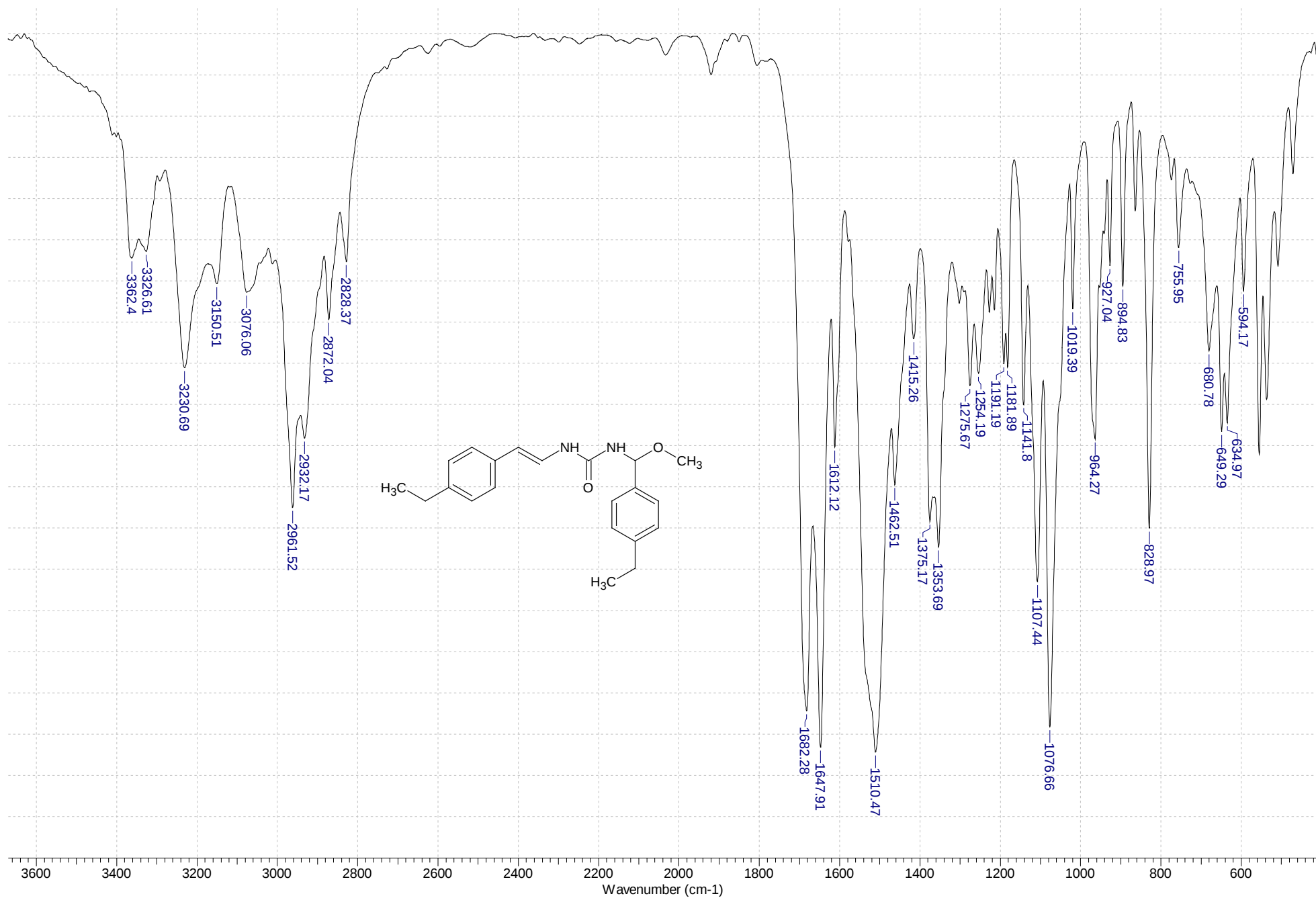
IR spectrum of compound **9c** (KBr).



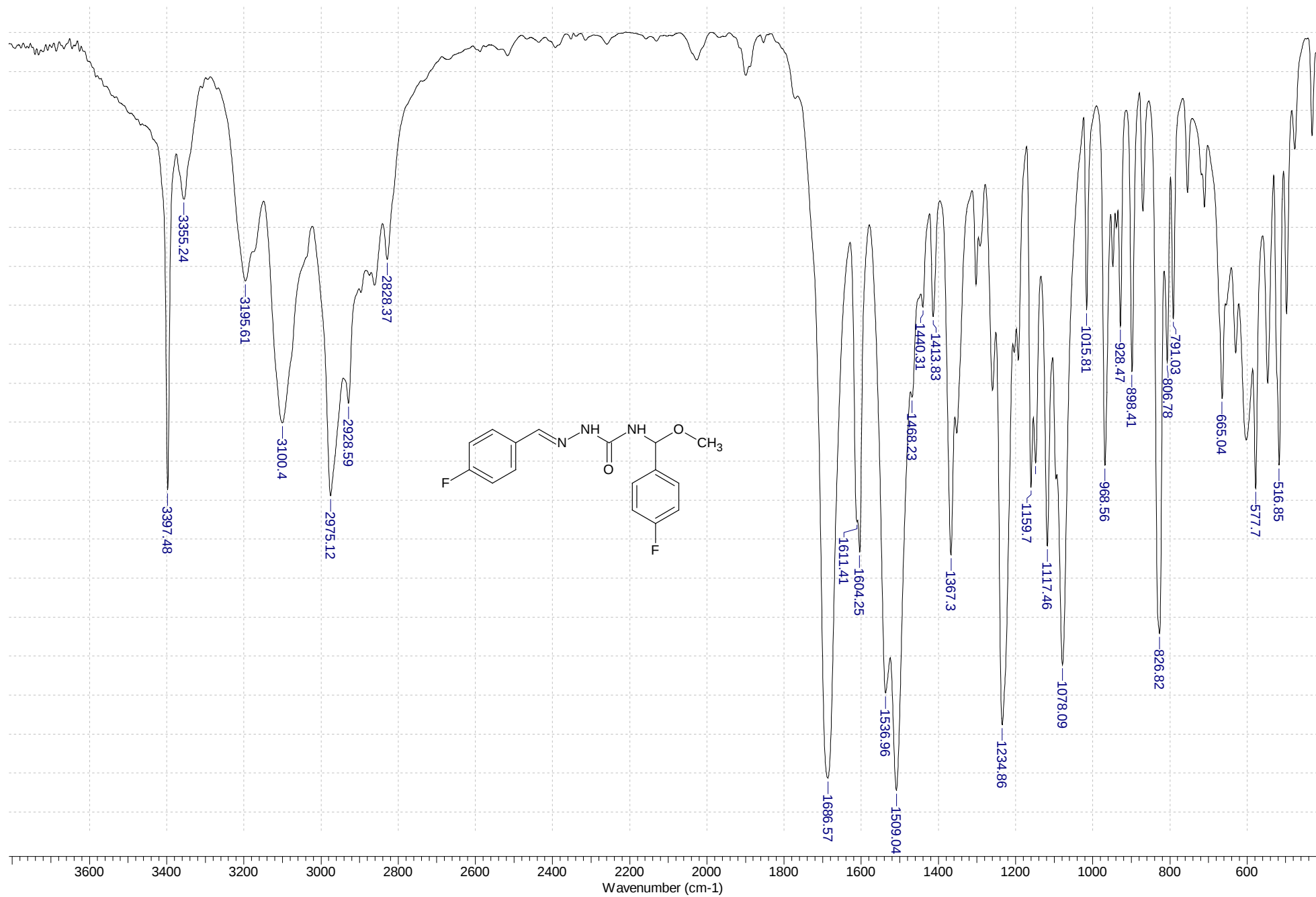
IR spectrum of compound **9d** (Nujol).



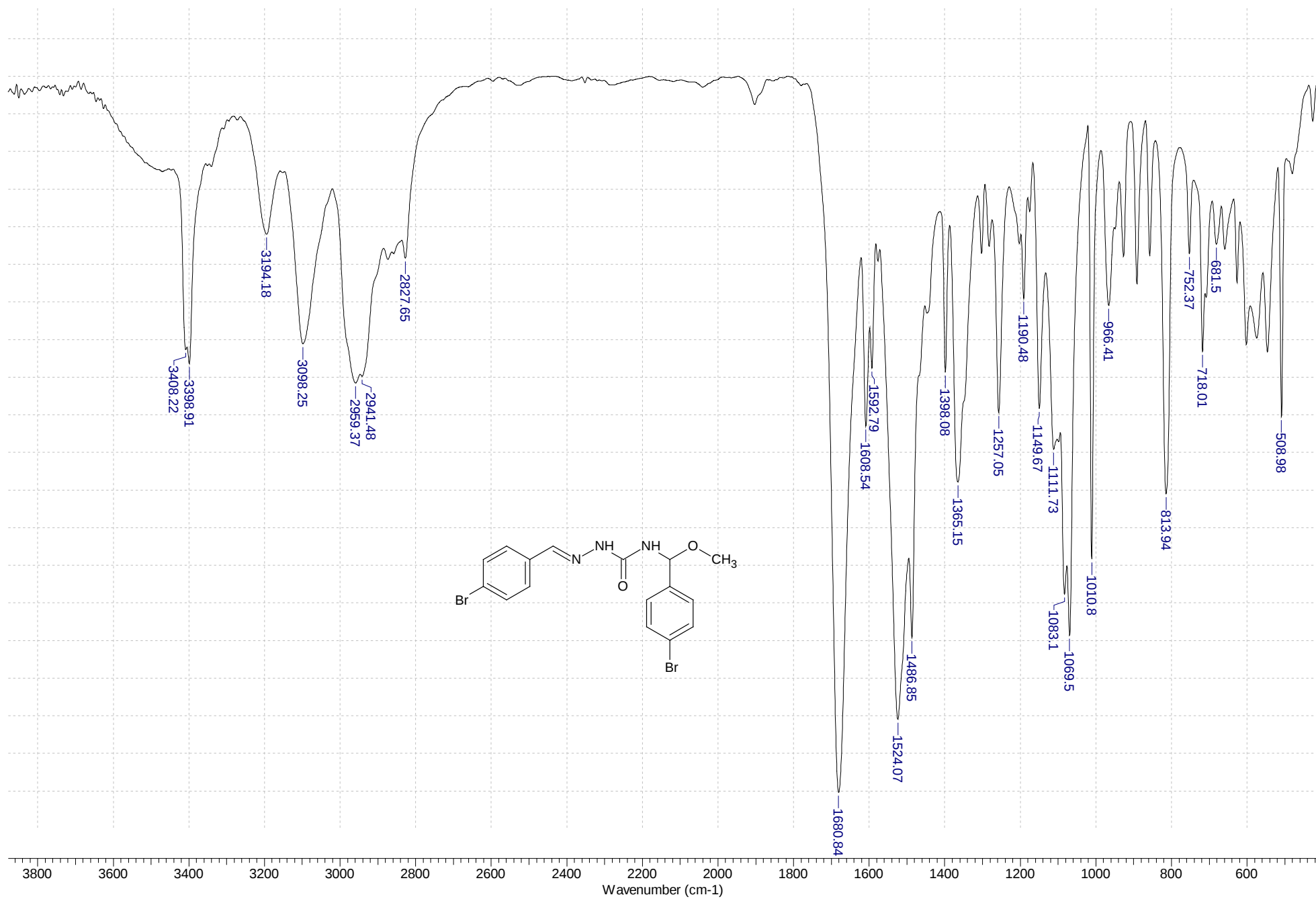
IR spectrum of compound **9e** (KBr).



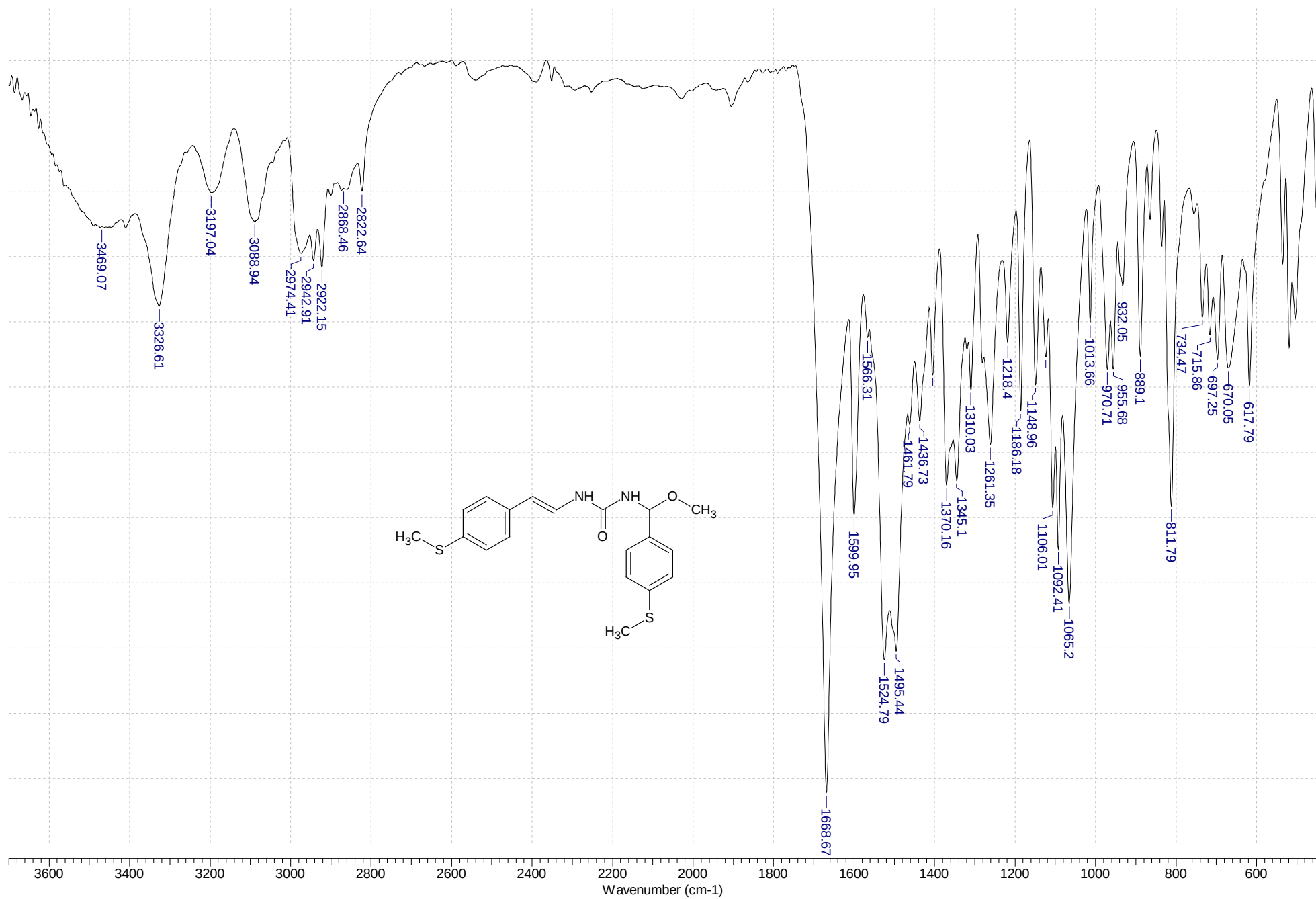
IR spectrum of compound **9f** (KBr).



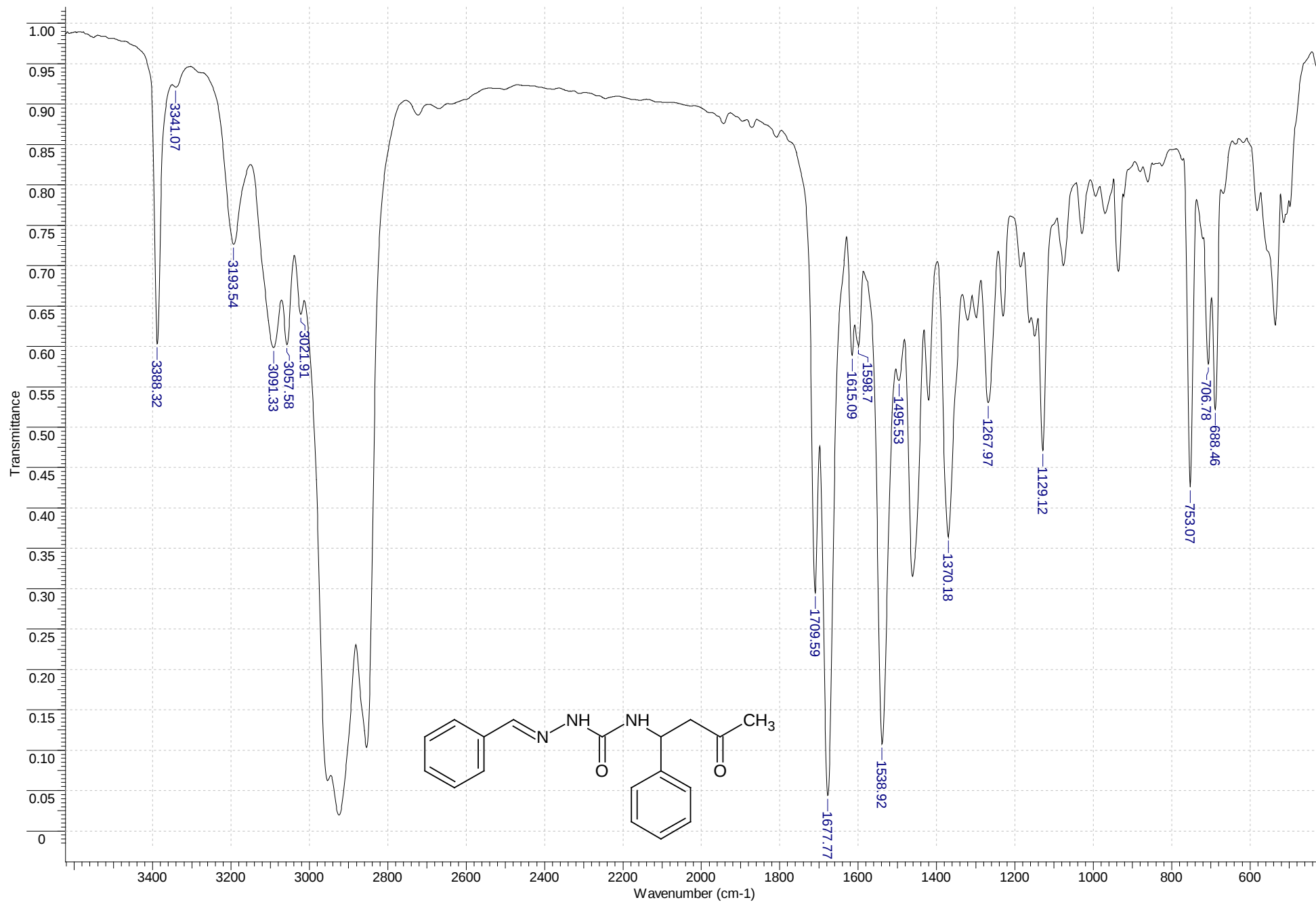
IR spectrum of compound **9g** (KBr).



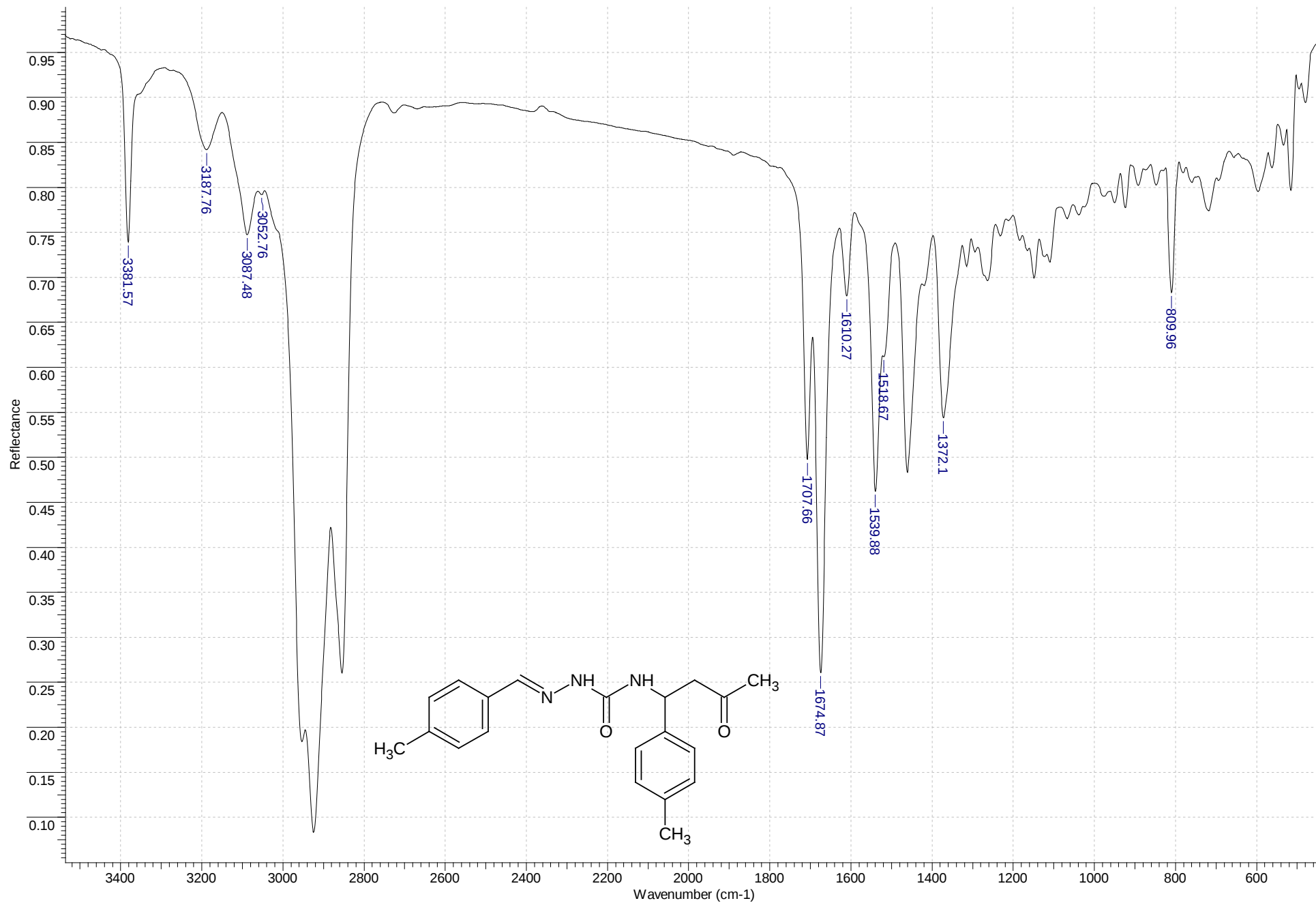
IR spectrum of compound **9h** (KBr).



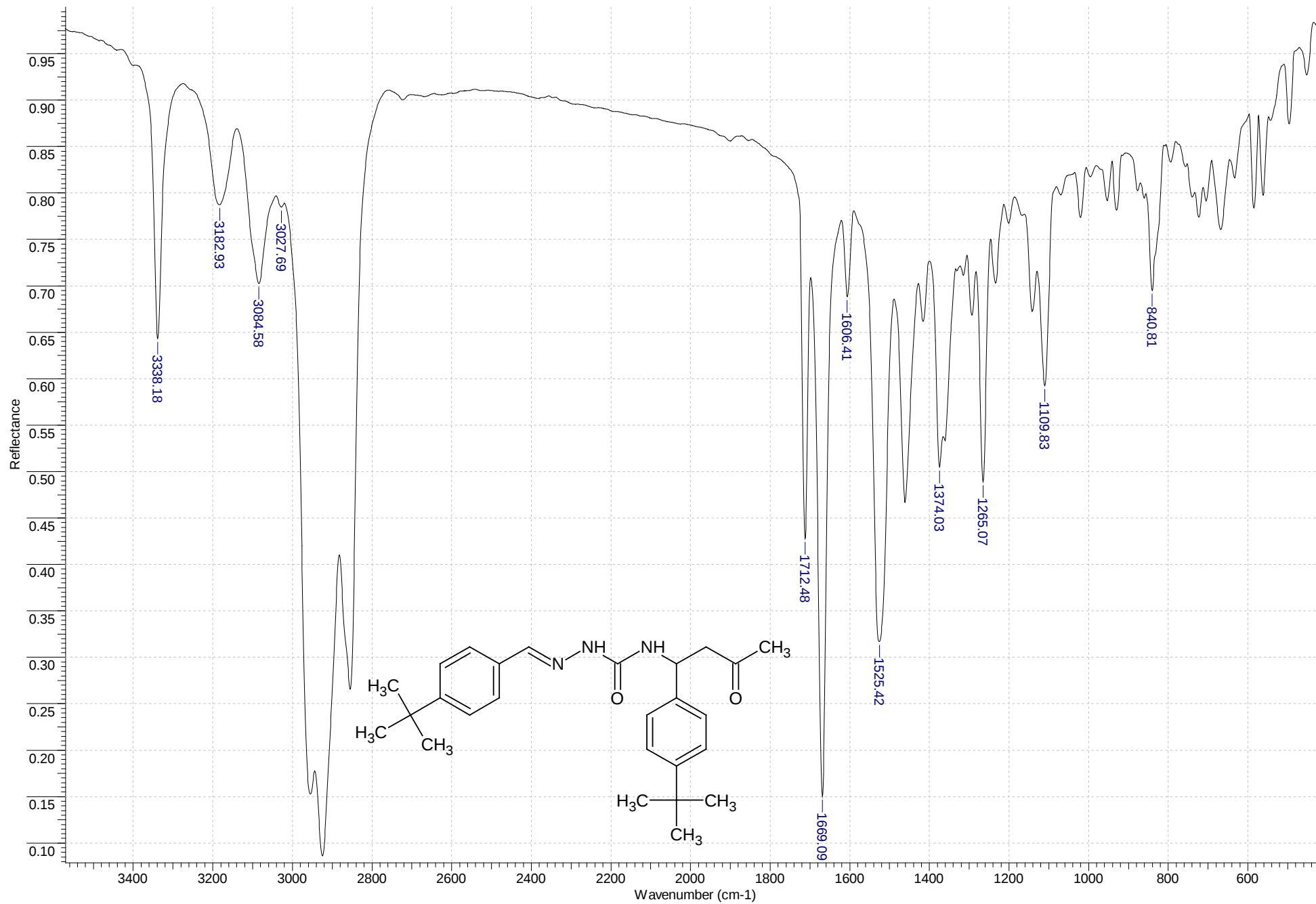
IR spectrum of compound **10a** (Nujol).



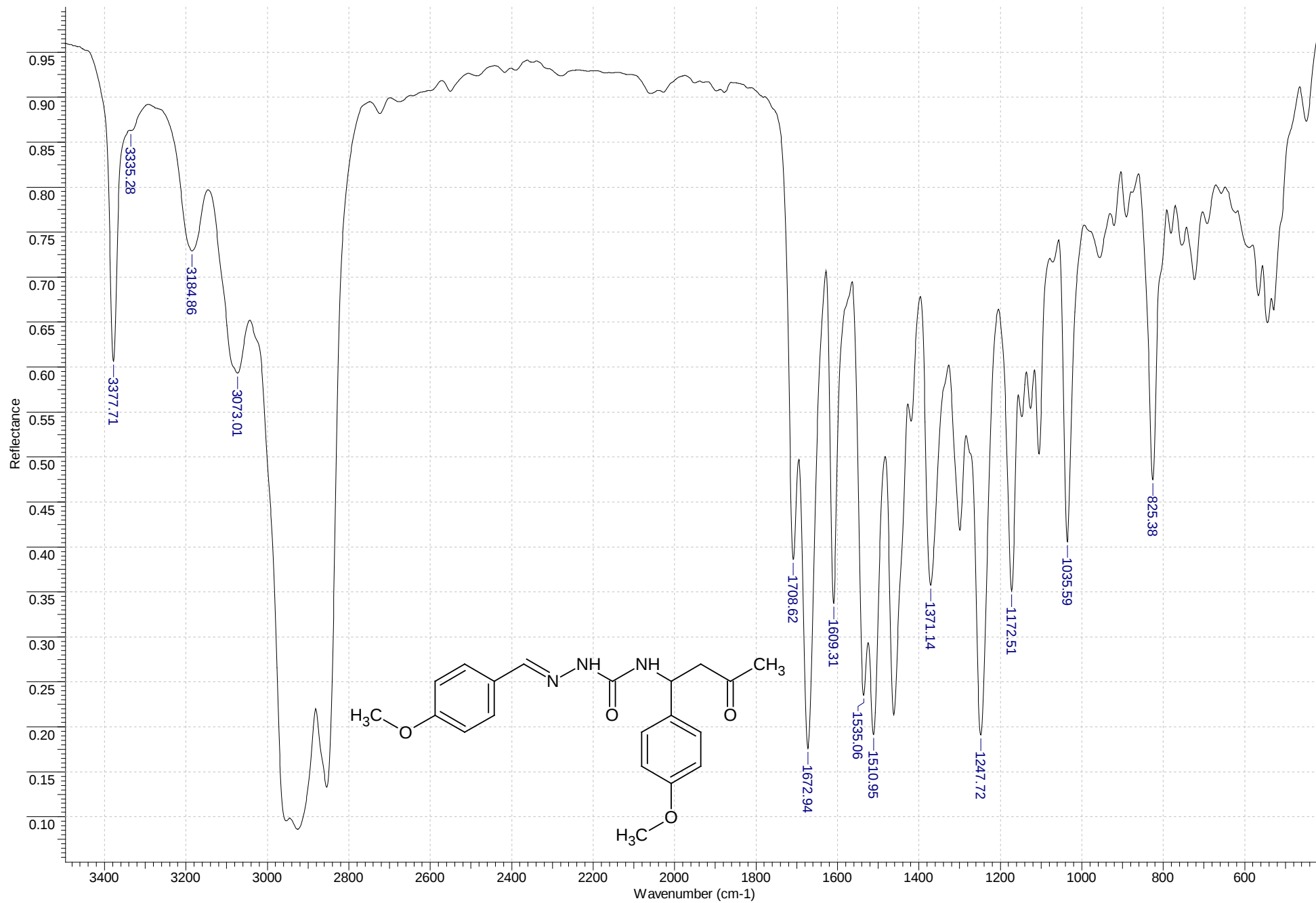
IR spectrum of compound **10b** (Nujol).



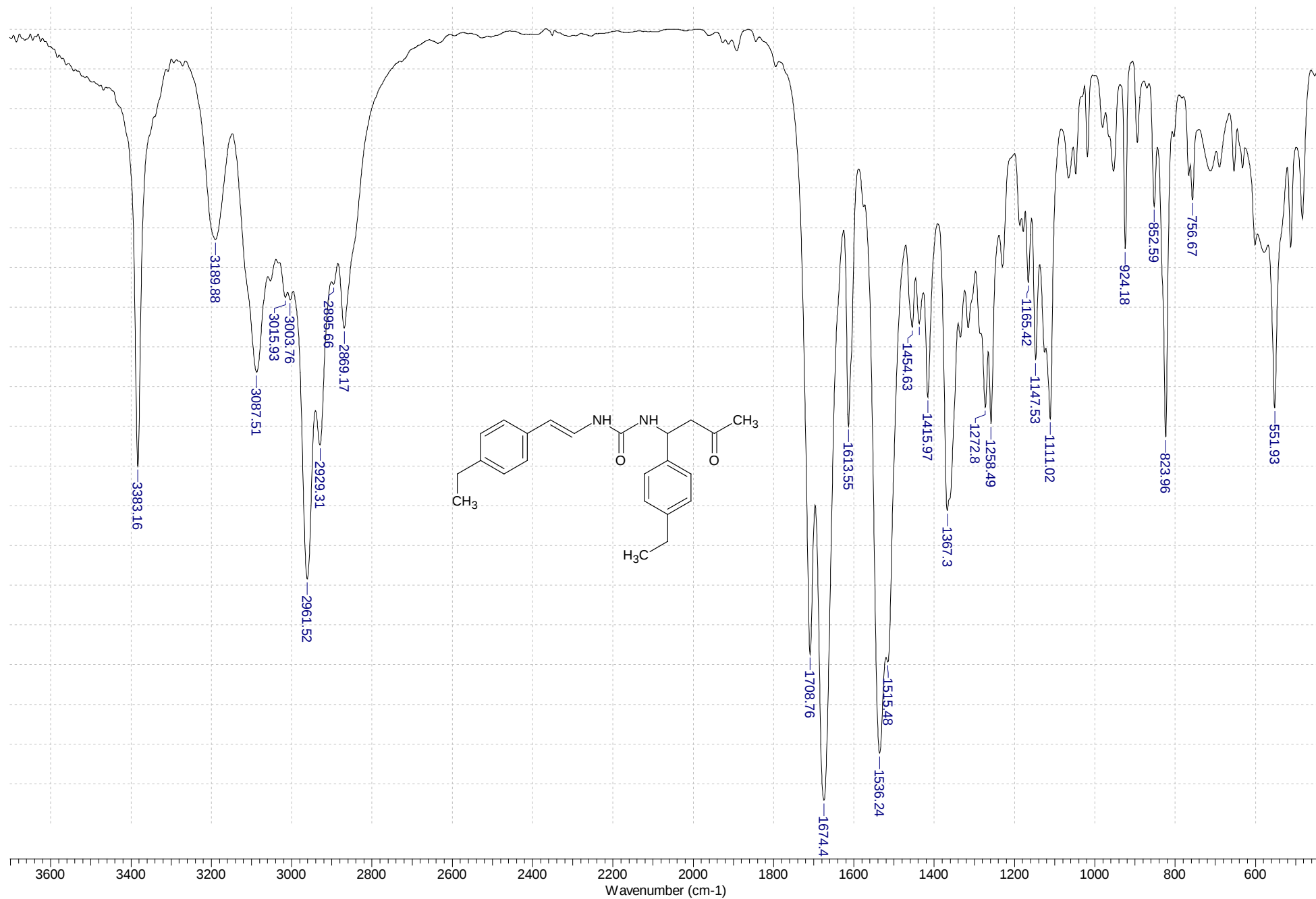
IR spectrum of compound **10c** (Nujol).



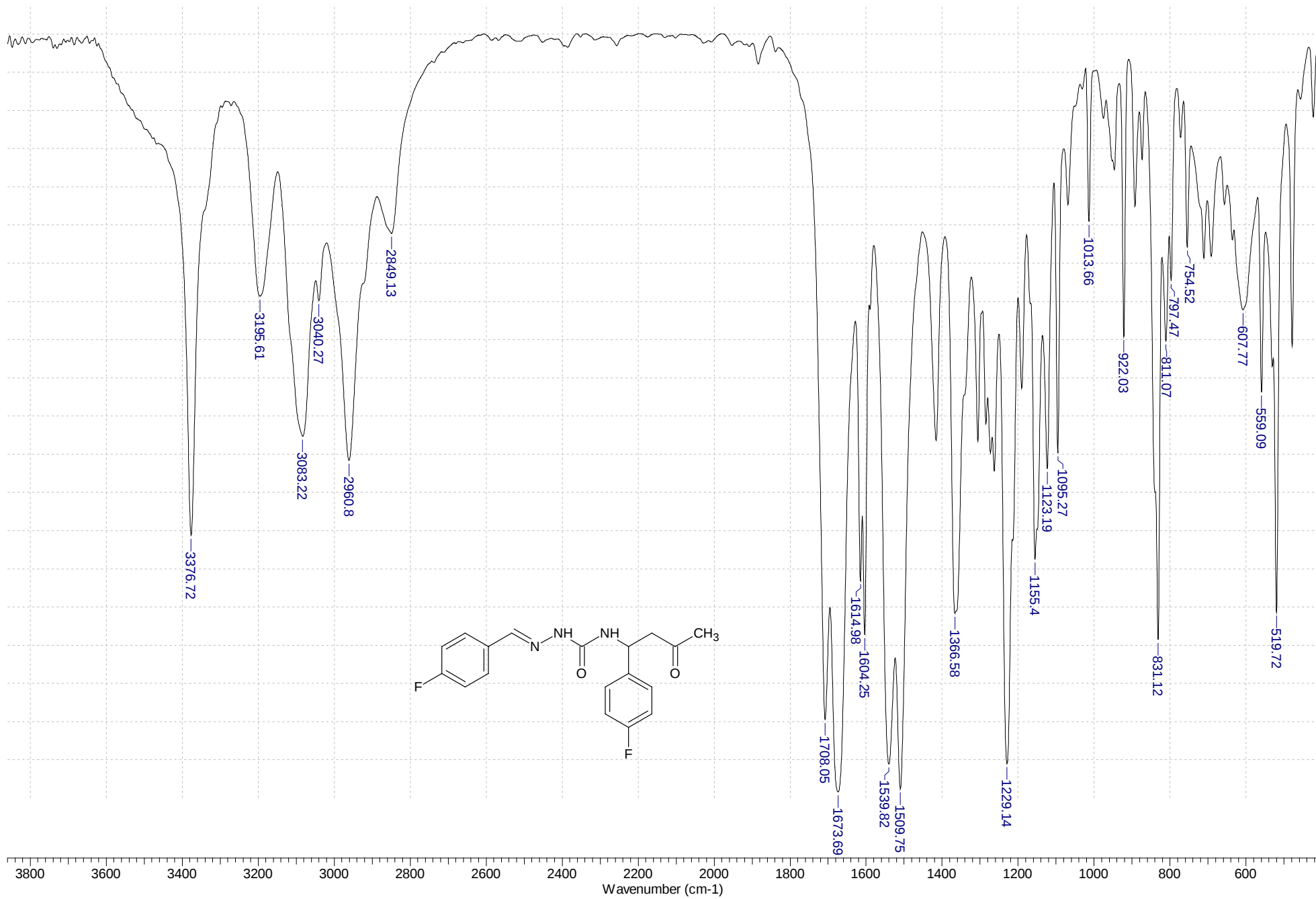
IR spectrum of compound **10d** (Nujol).



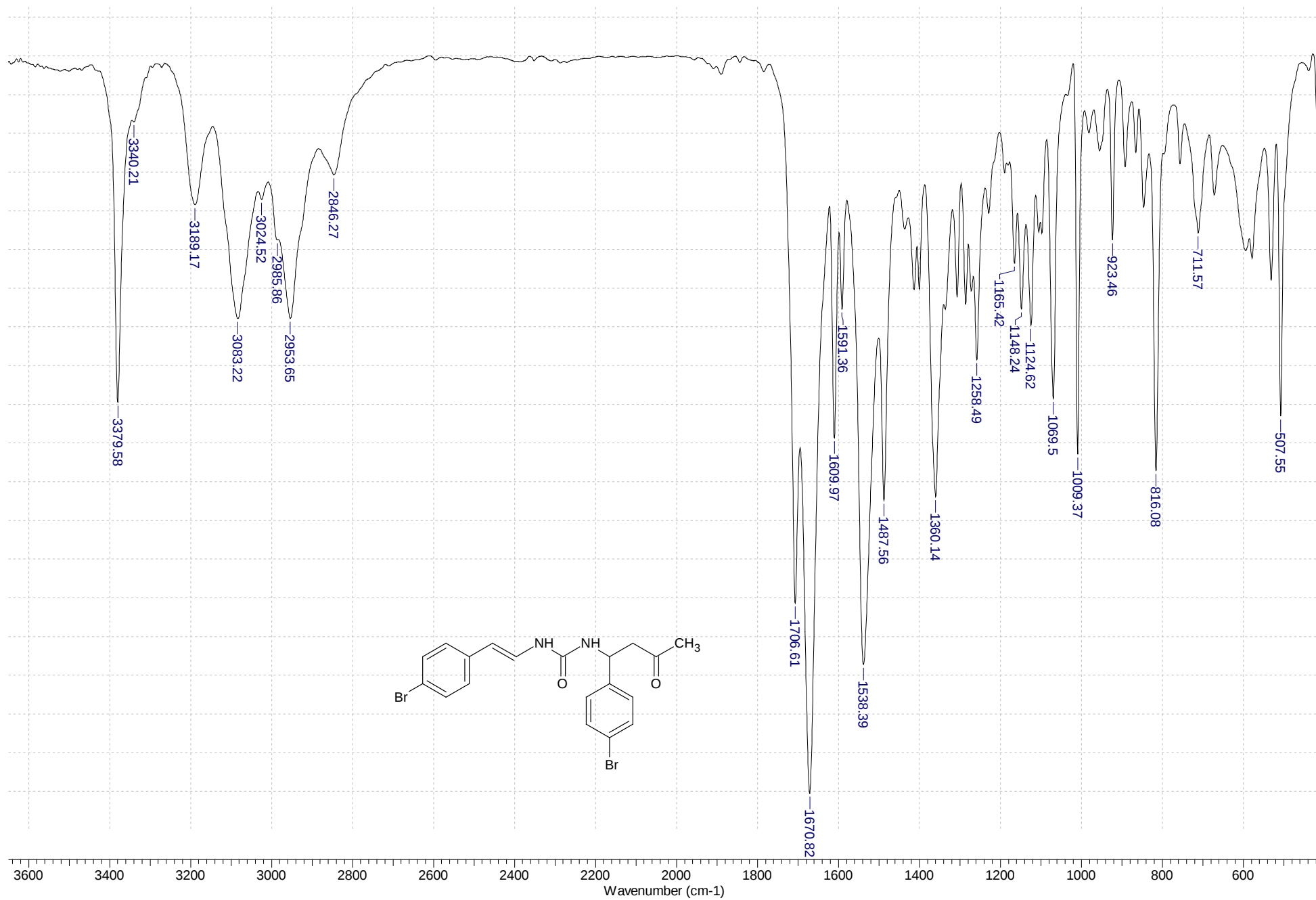
IR spectrum of compound **10e** (KBr).



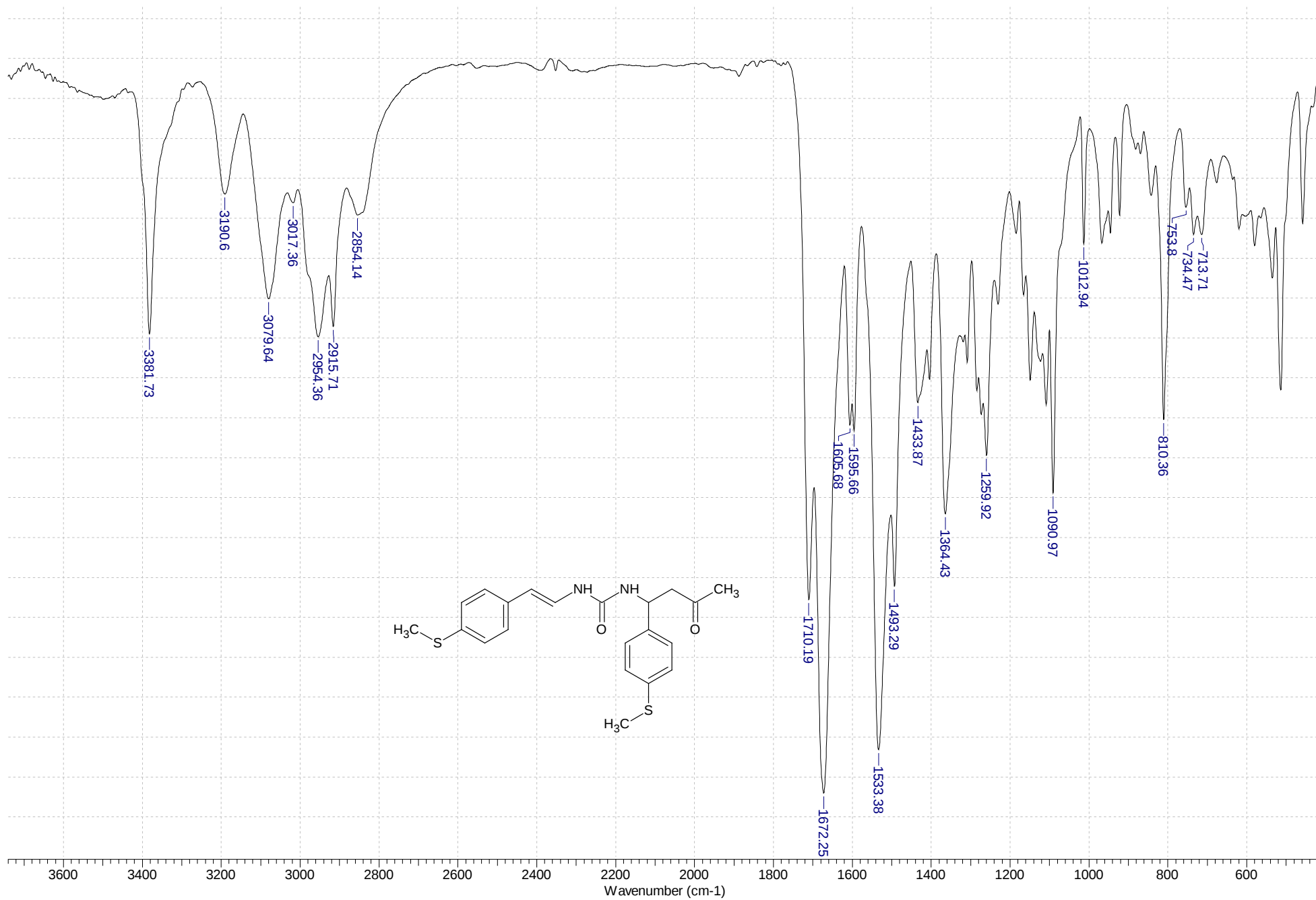
IR spectrum of compound **10f** (KBr).



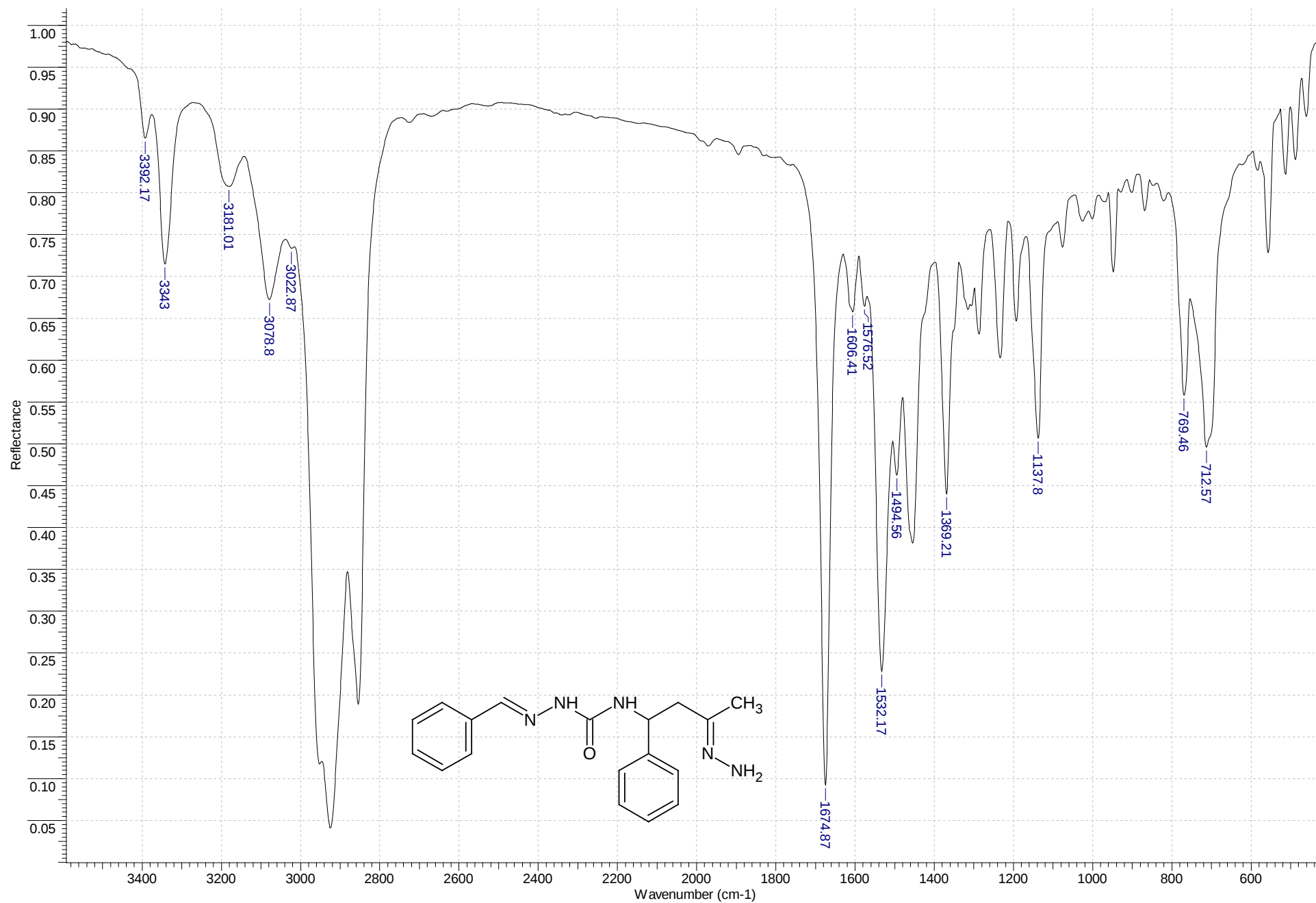
IR spectrum of compound **10g** (KBr).



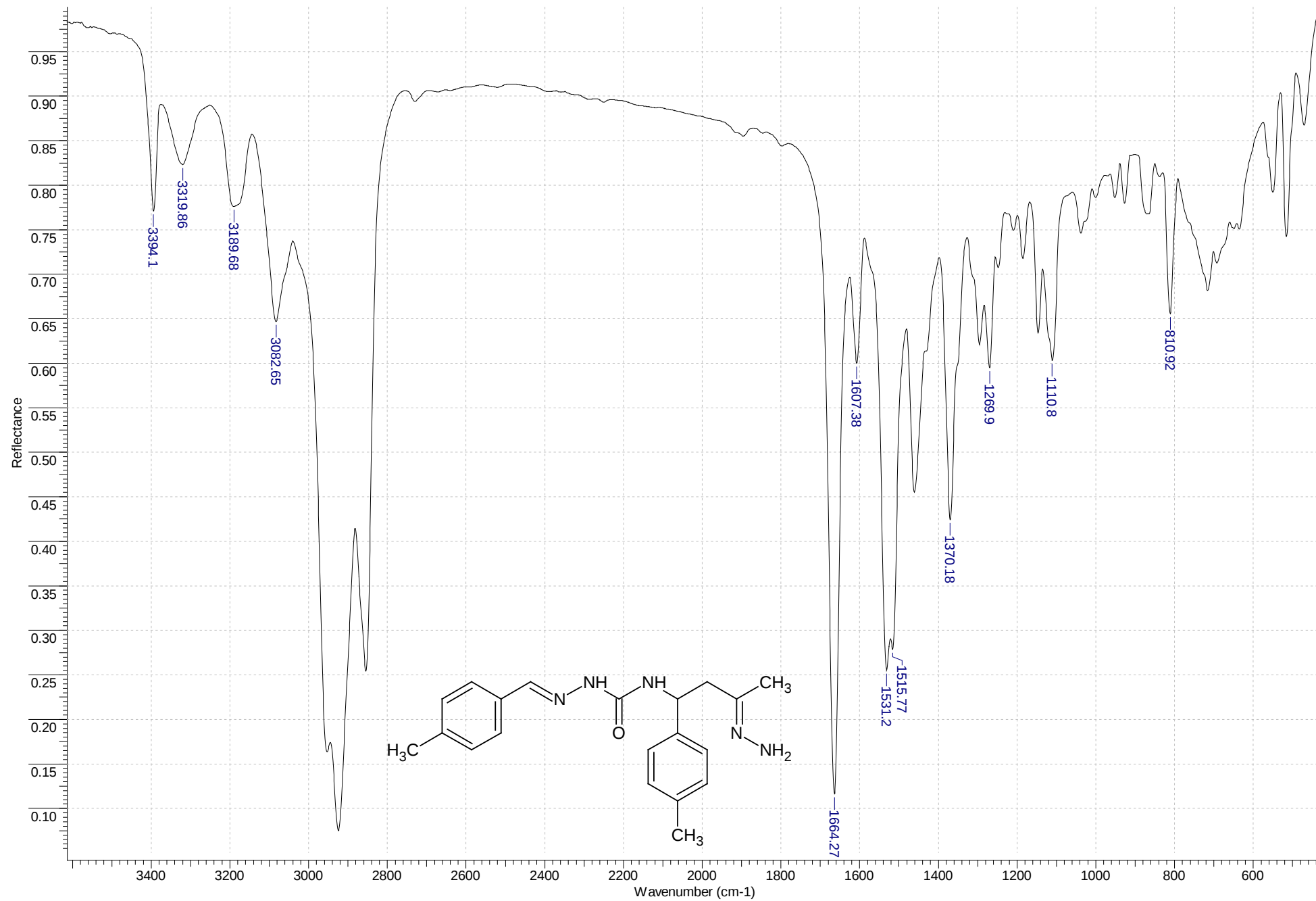
IR spectrum of compound **10h** (KBr).



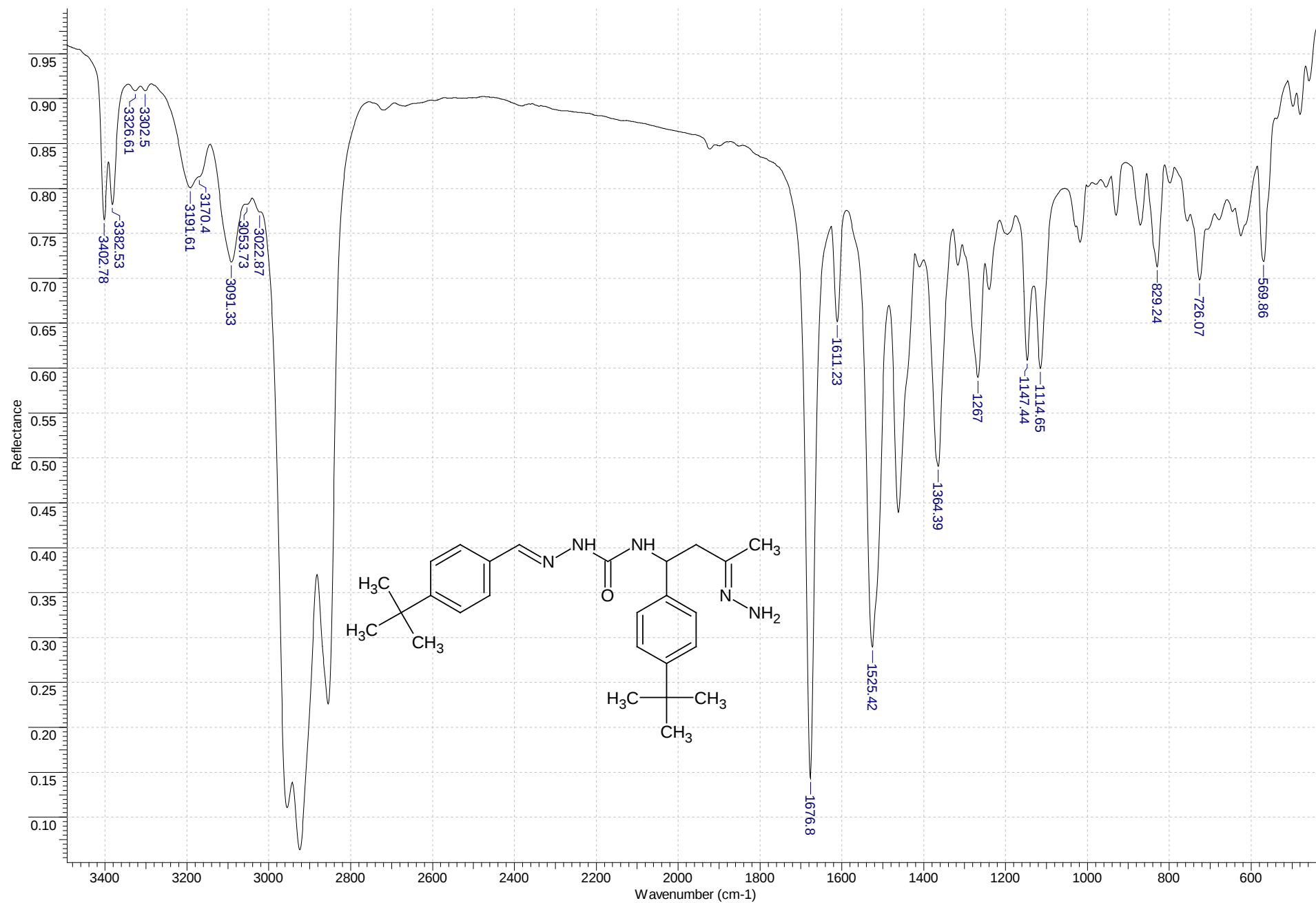
IR spectrum of compound **14a** (Nujol).



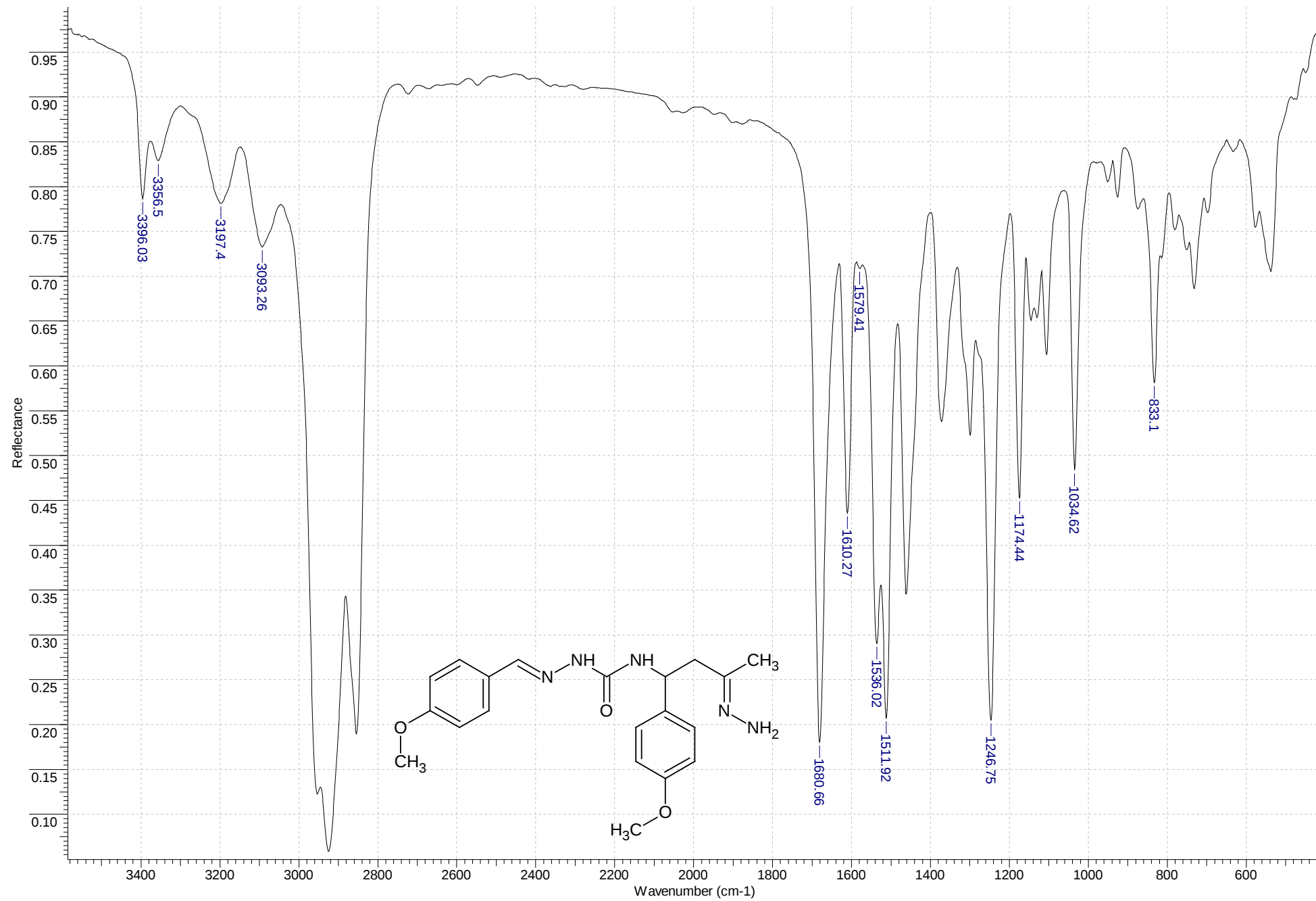
IR spectrum of compound **14b** (Nujol).



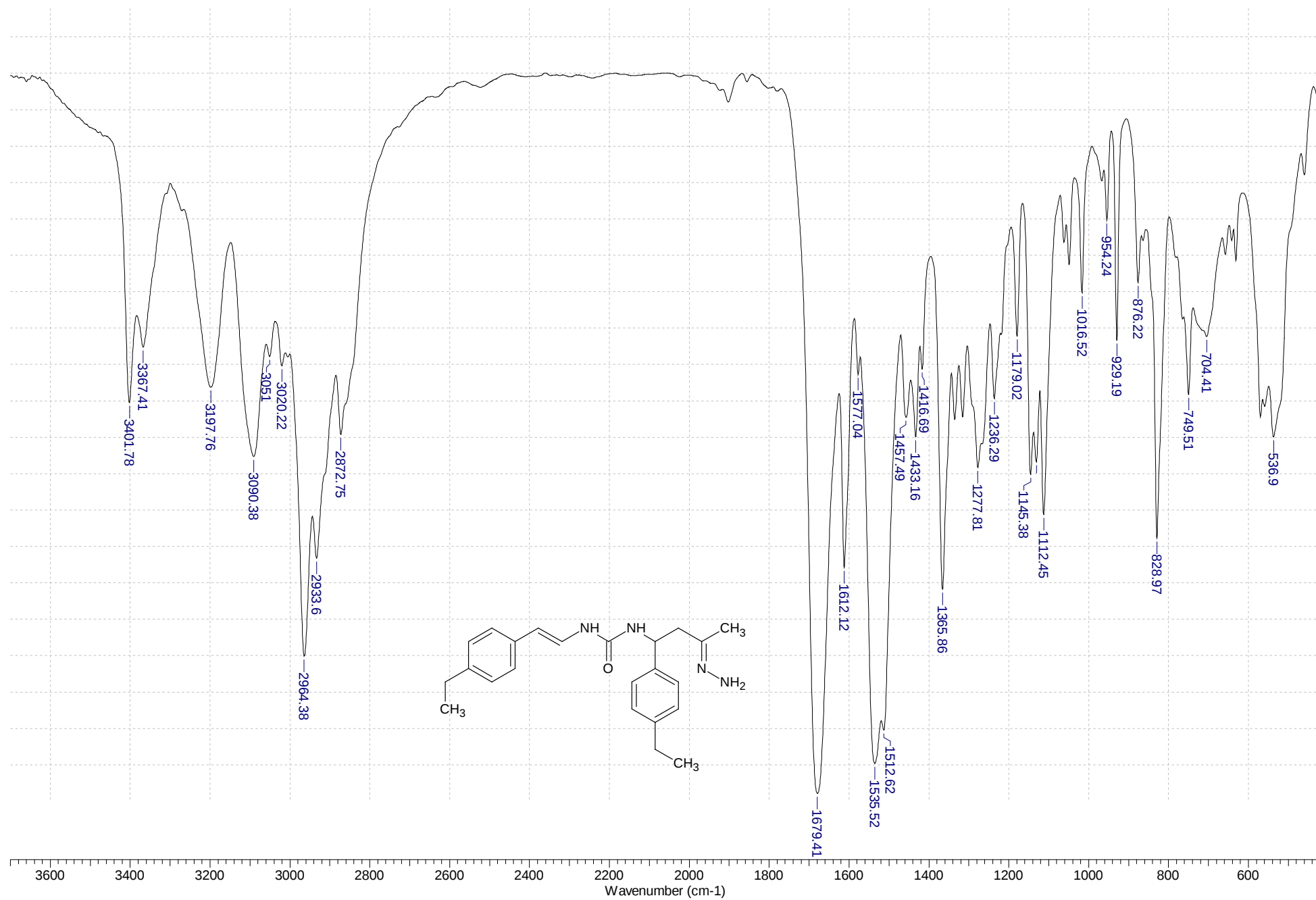
IR spectrum of compound **14c** (Nujol).



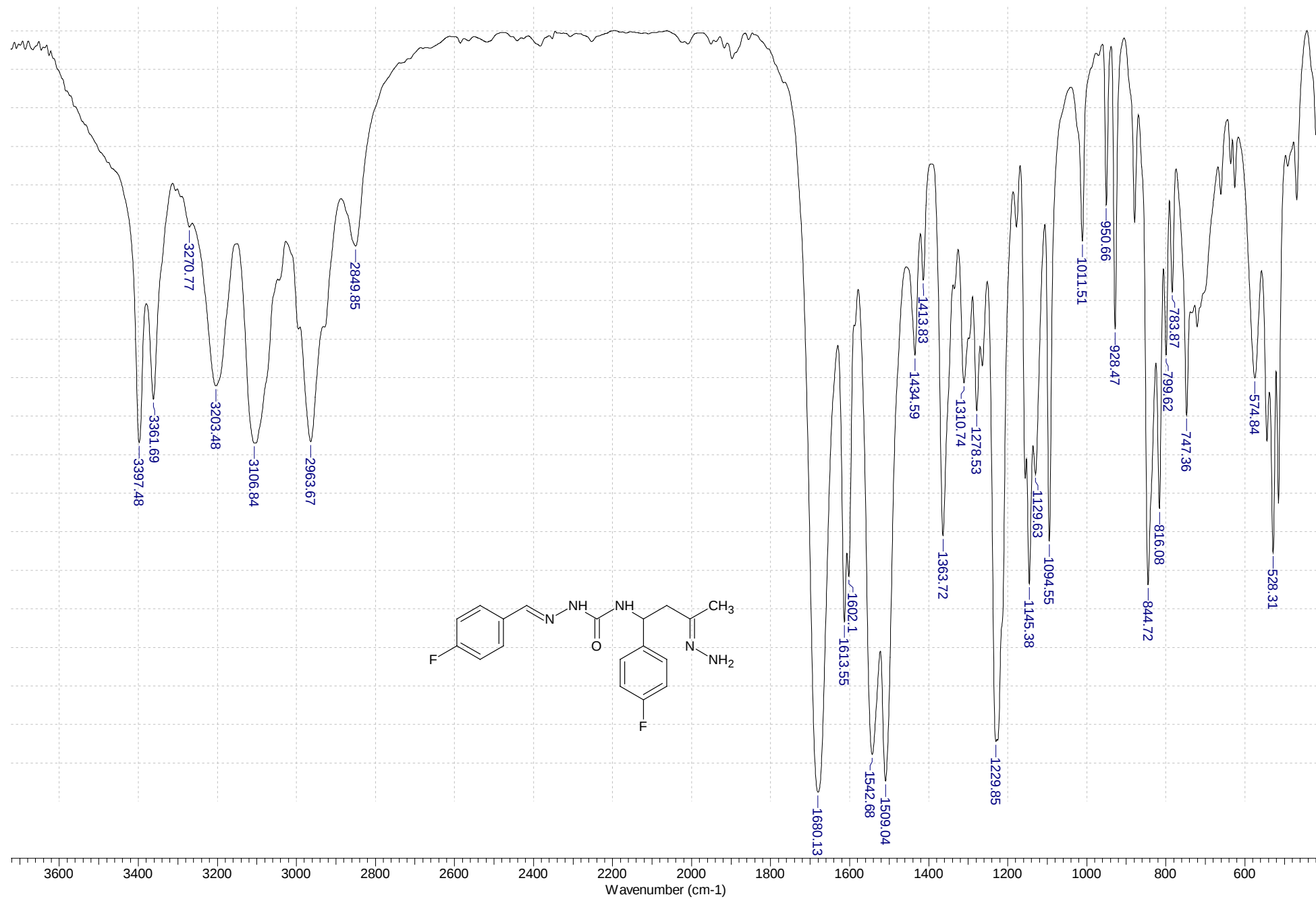
IR spectrum of compound **14d** (Nujol).



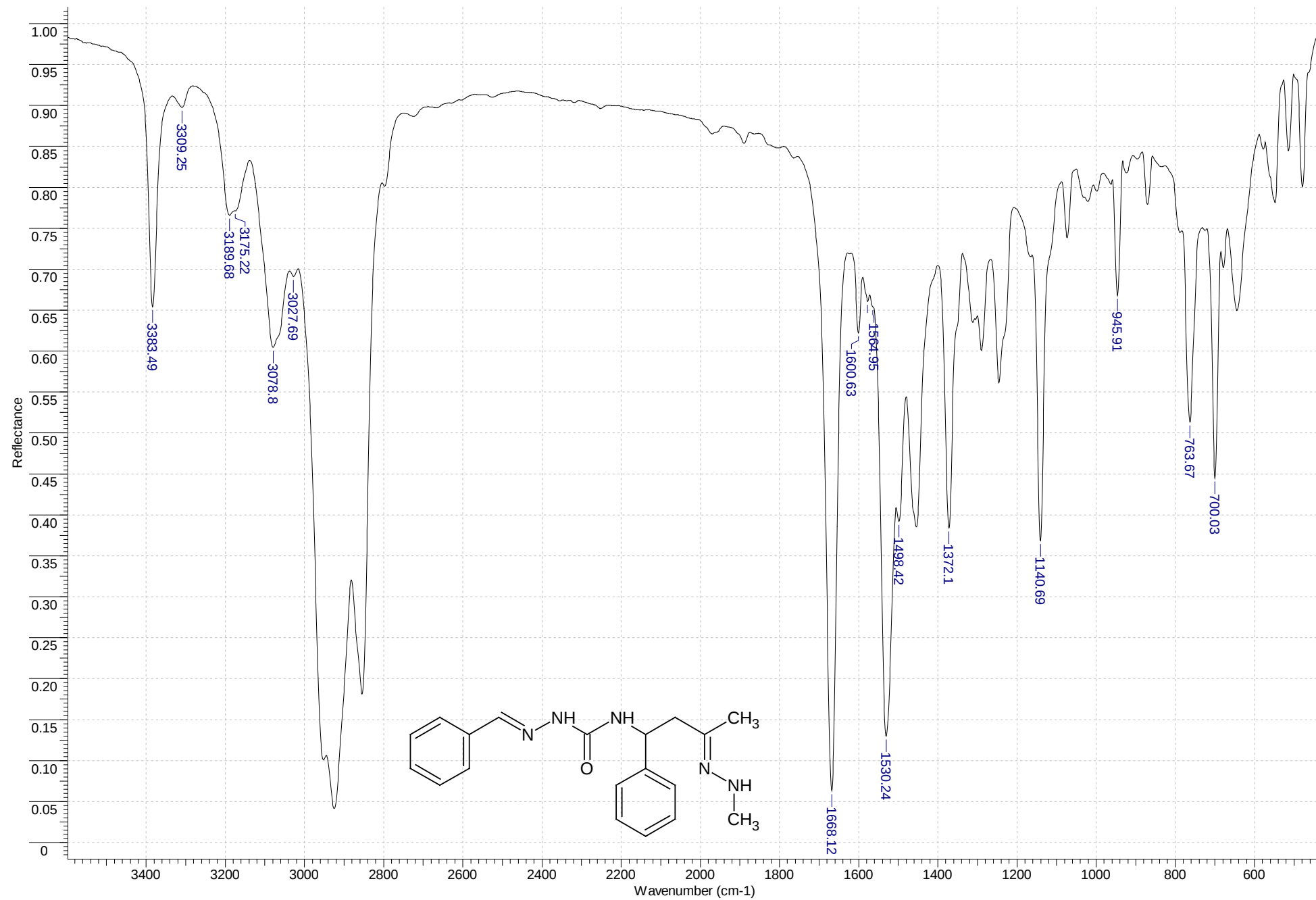
IR spectrum of compound **14e** (KBr).



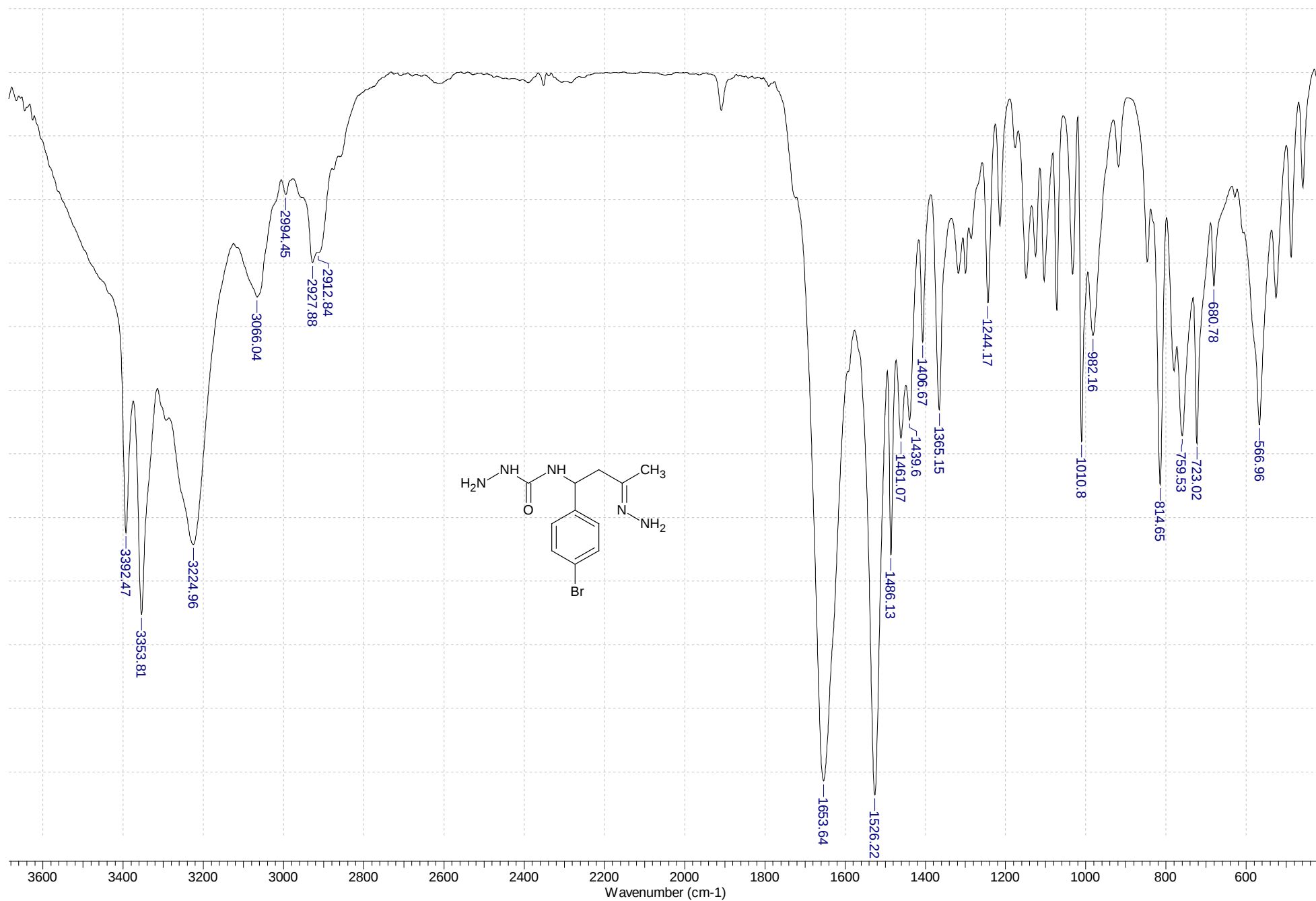
IR spectrum of compound **14f** (KBr).



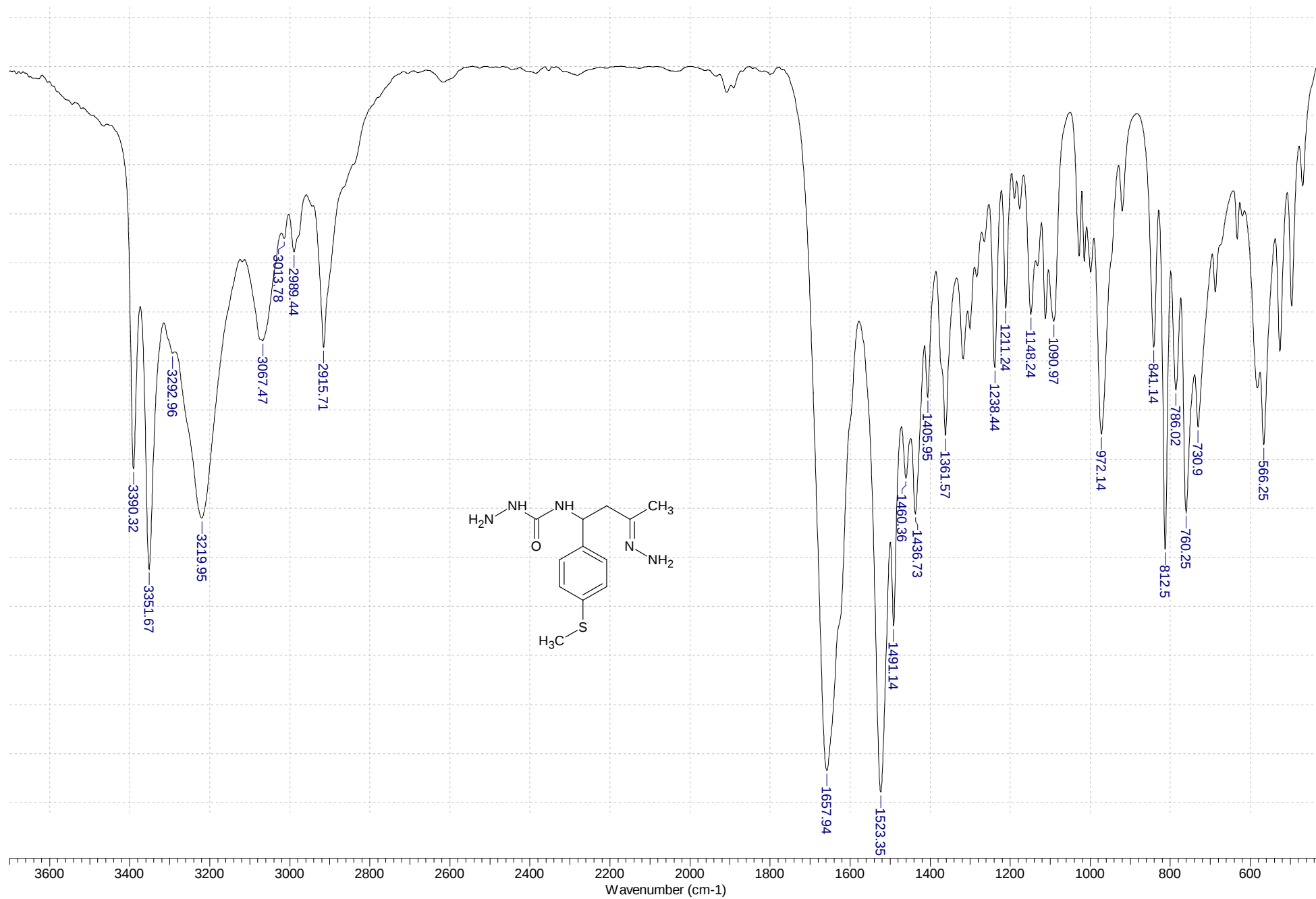
IR spectrum of compound 15 (Nujol).



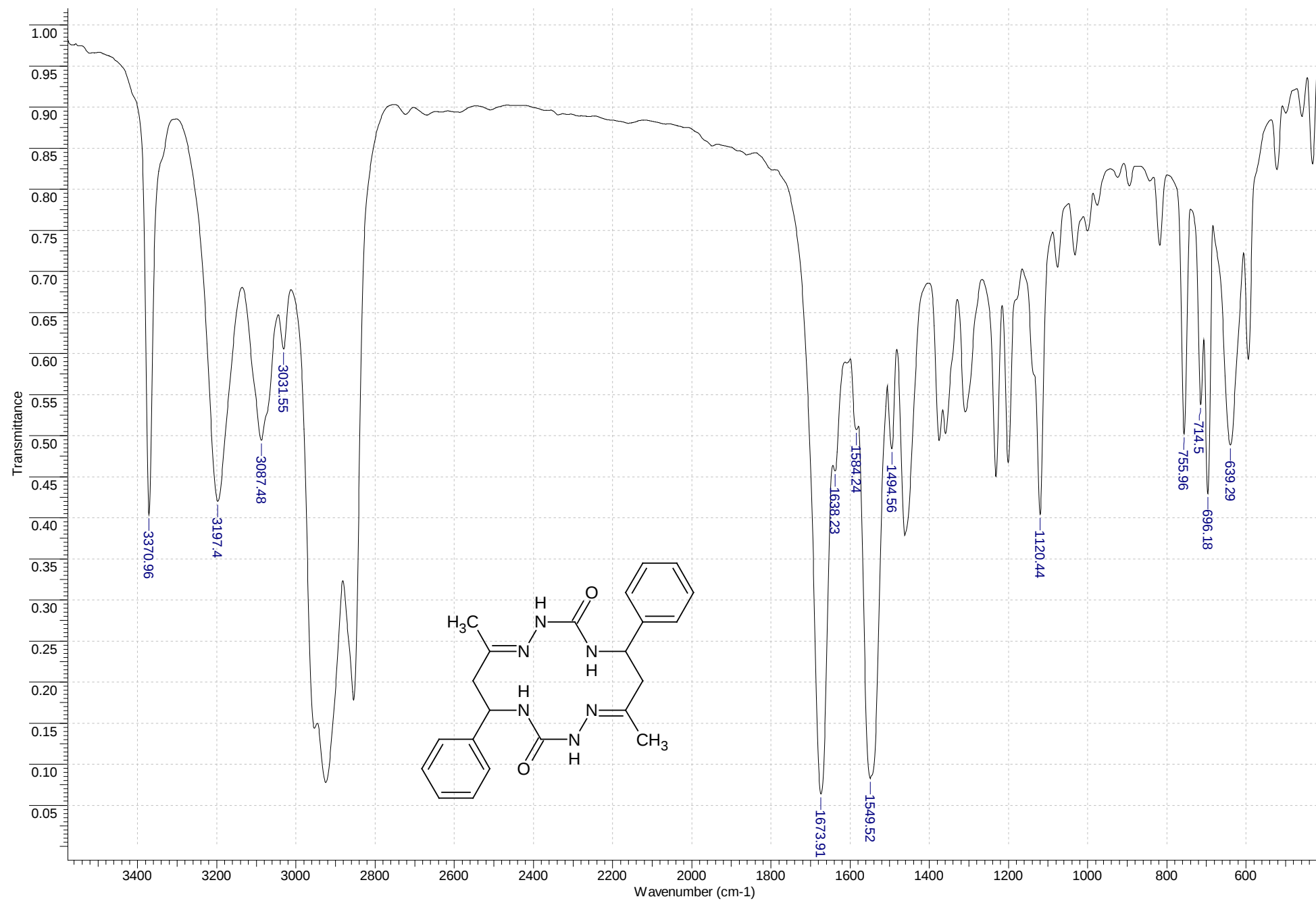
IR spectrum of compound **16a** (KBr).



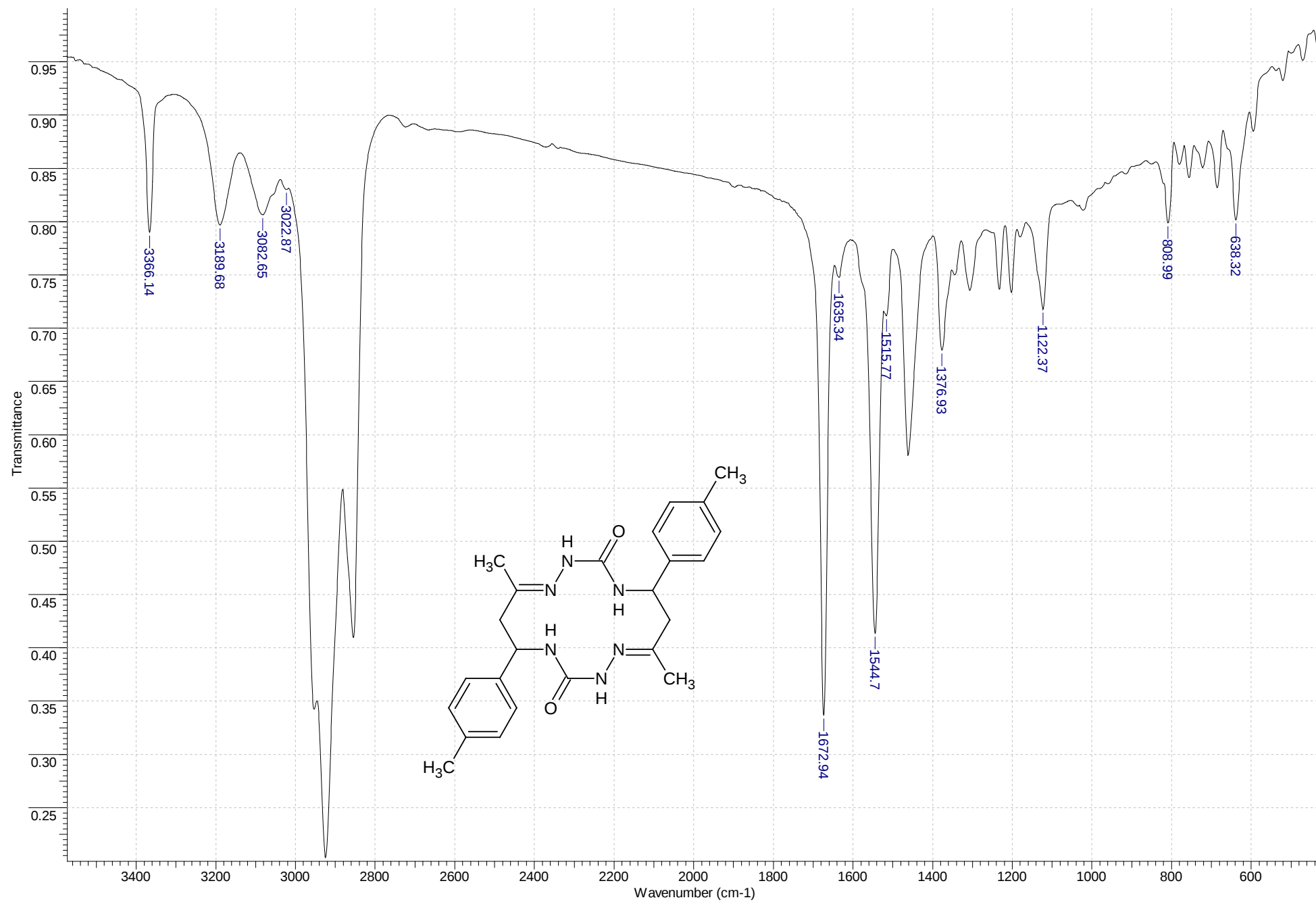
IR spectrum of compound **16b** (KBr).



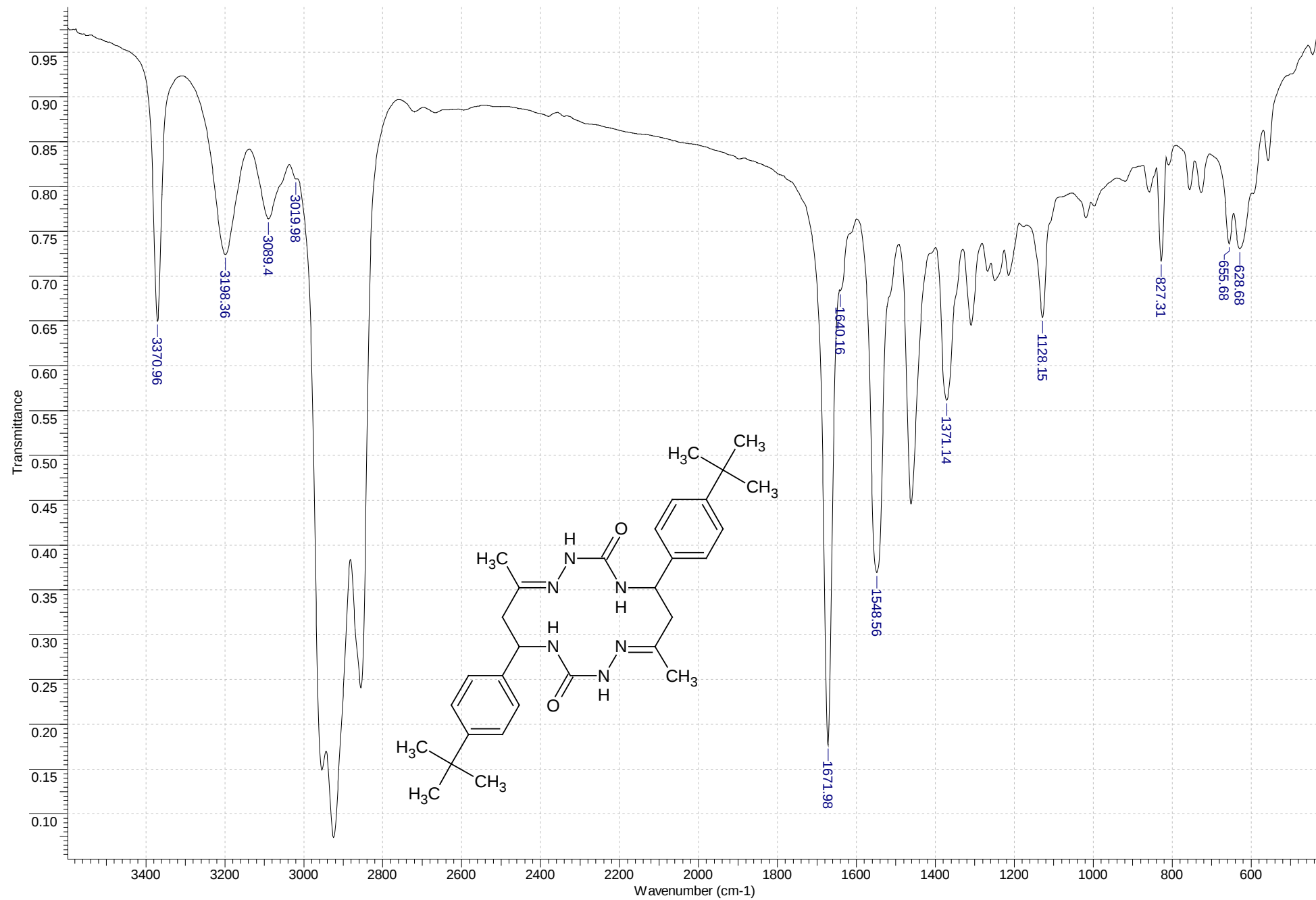
IR spectrum of compound **17a** (Nujol).



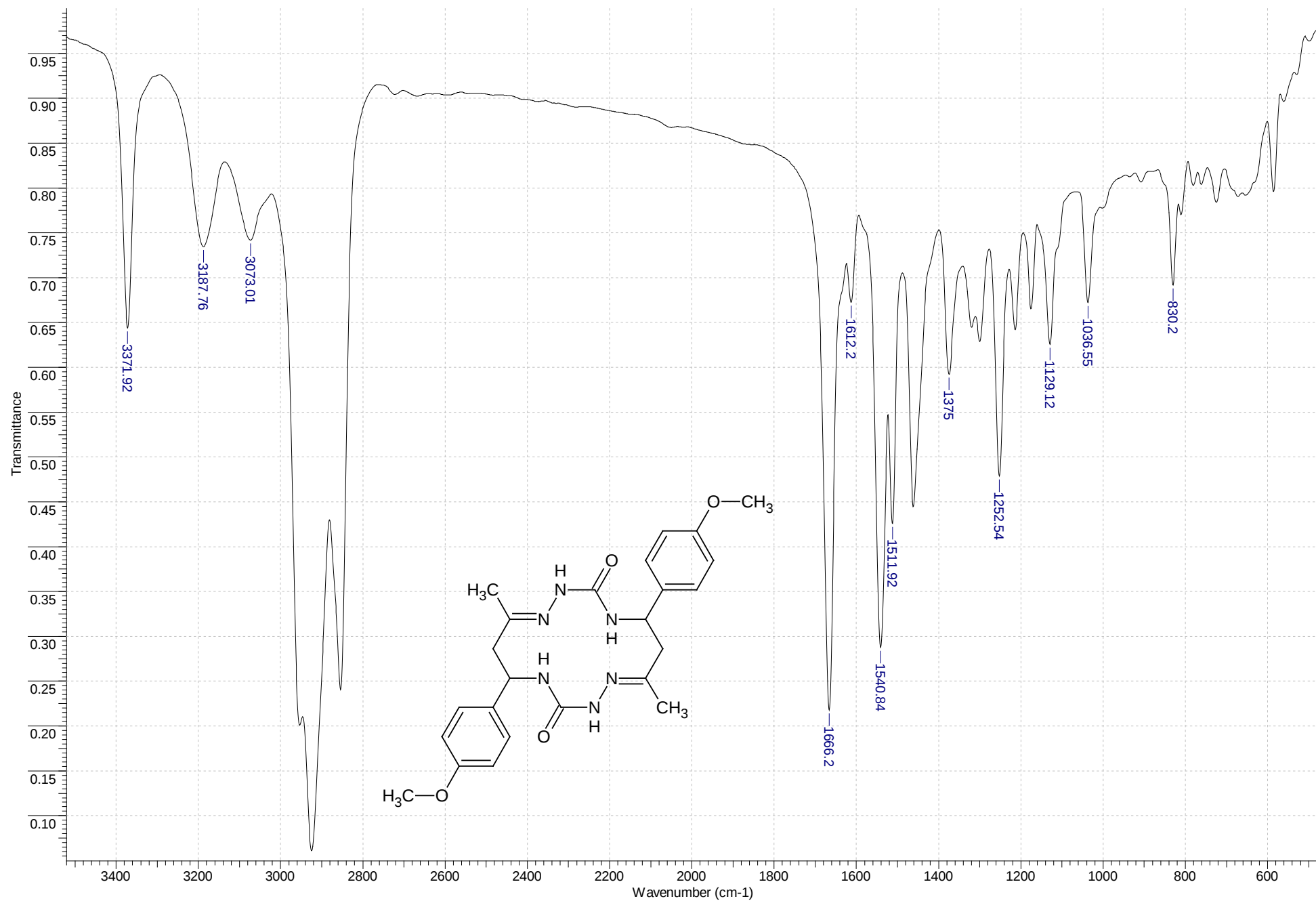
IR spectrum of compound **17b** (Nujol).



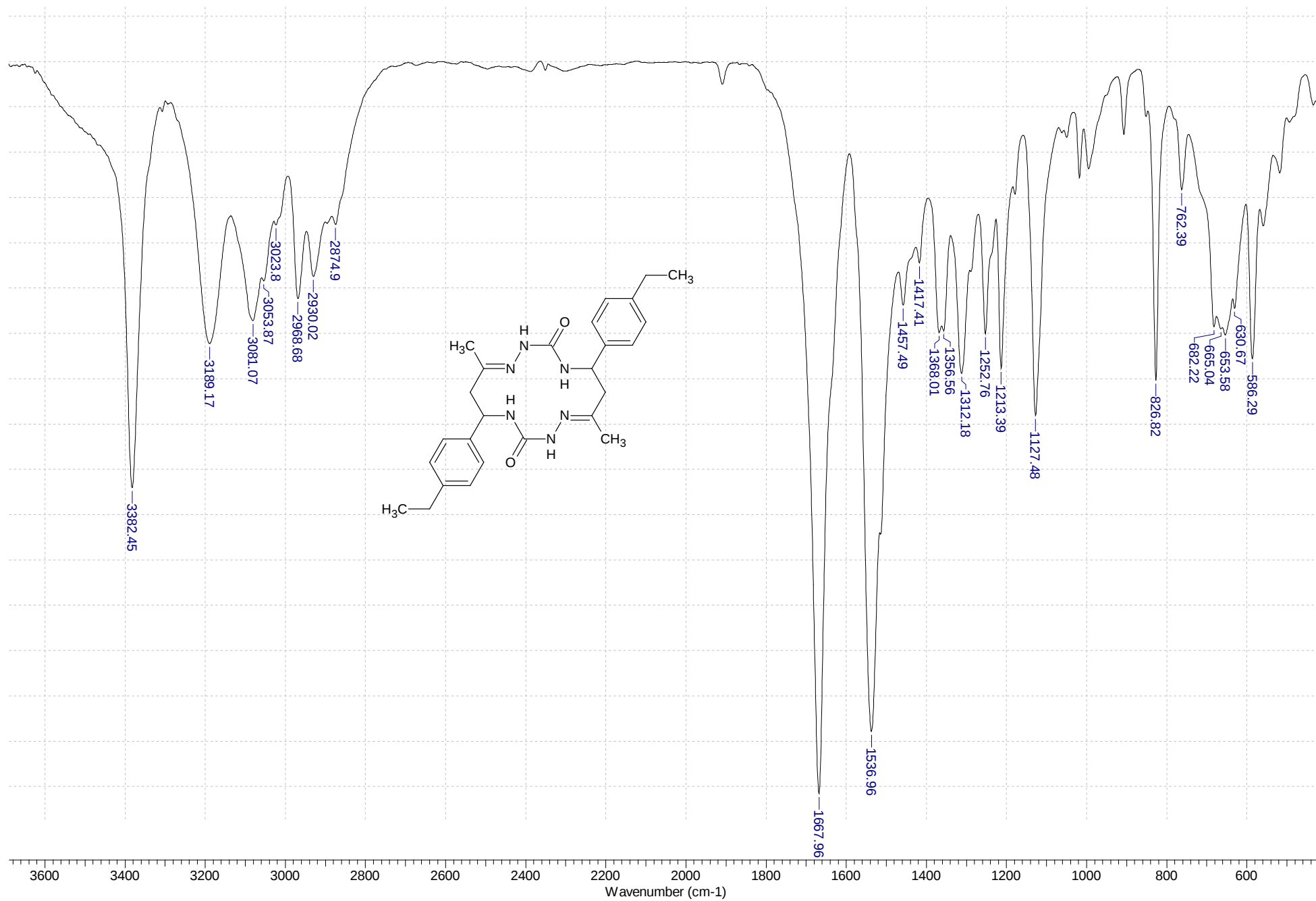
IR spectrum of compound **17c** (Nujol).



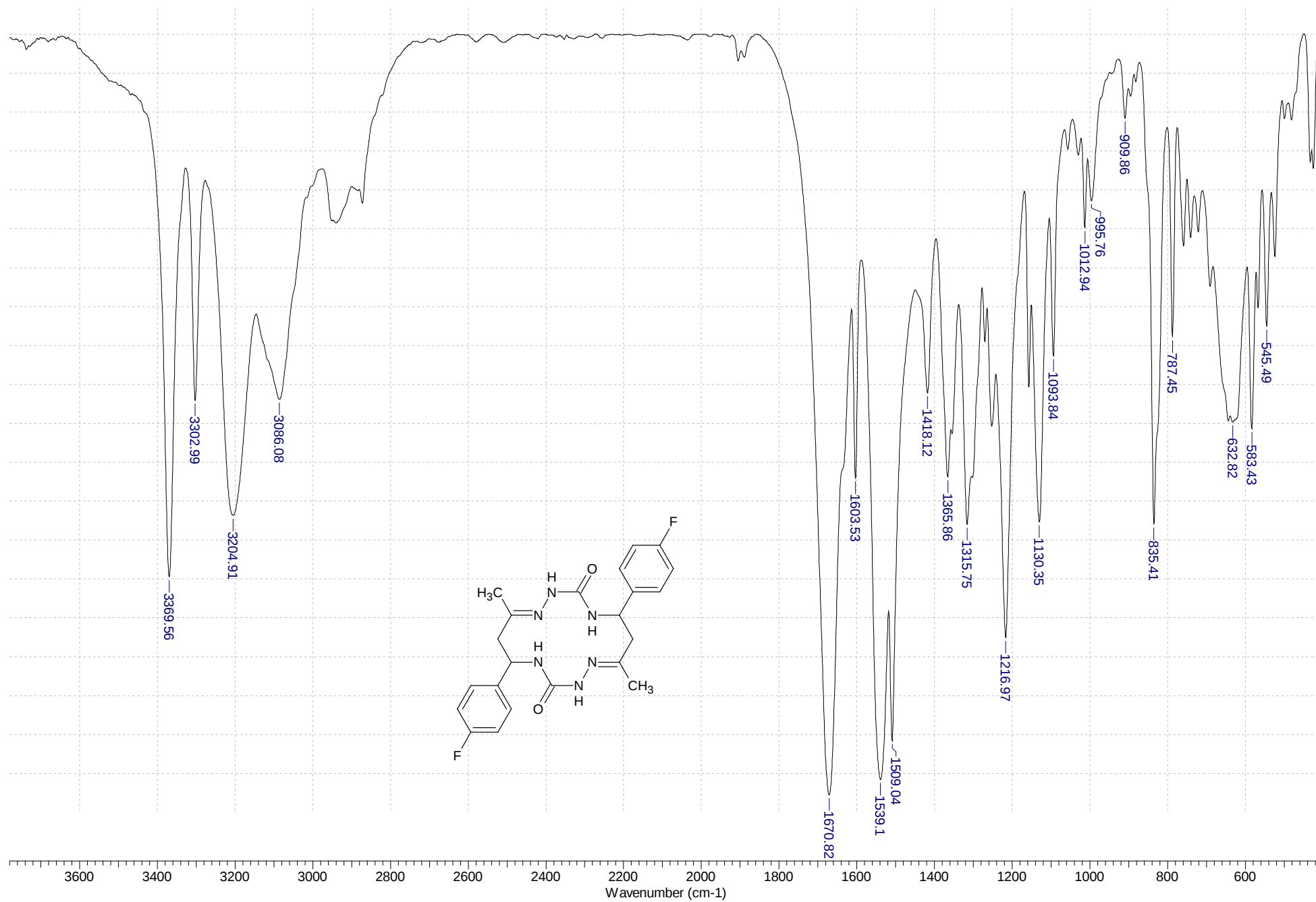
IR spectrum of compound **17d** (Nujol).



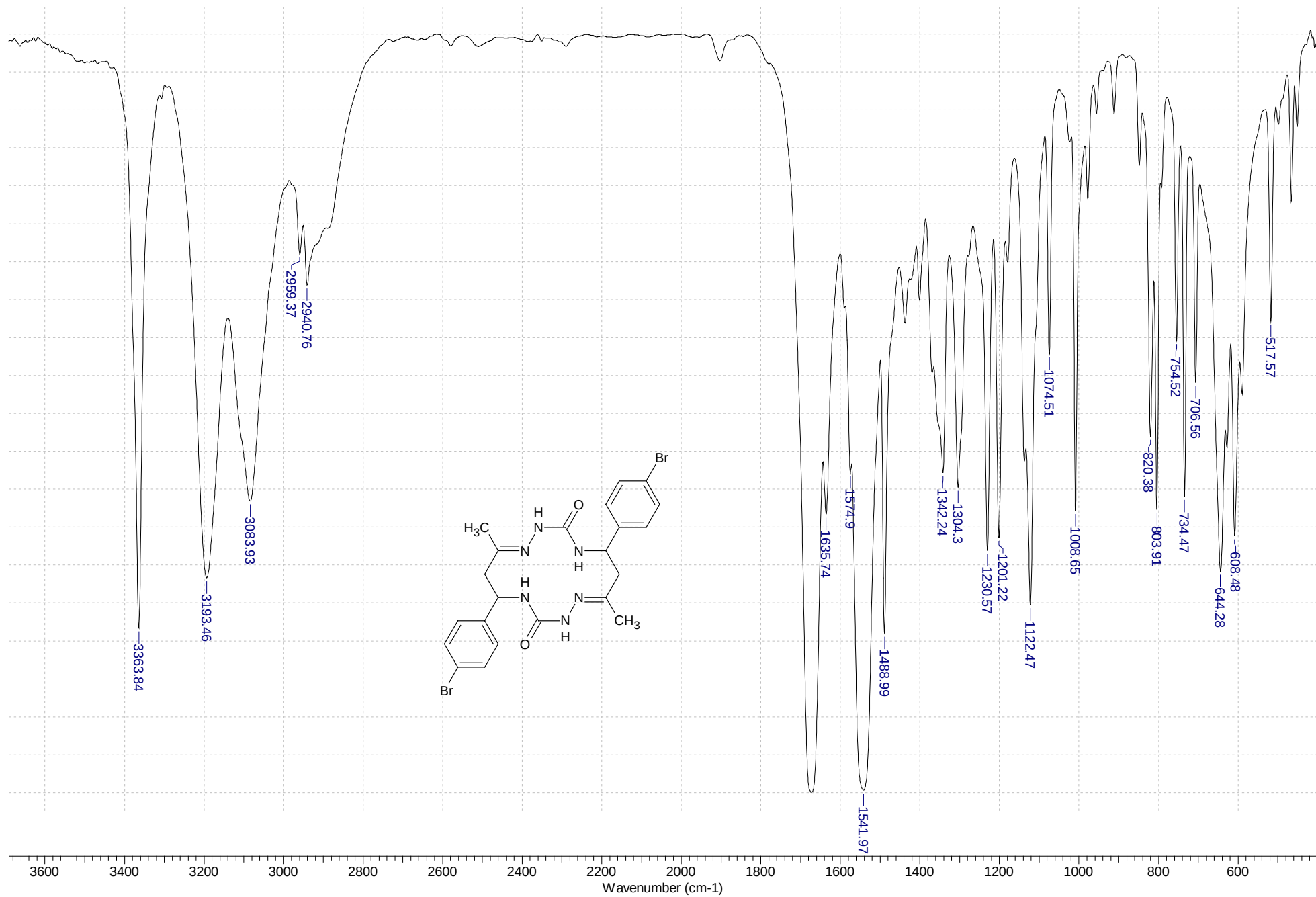
IR spectrum of compound **17e** (KBr).



IR spectrum of compound **17f** (KBr).



IR spectrum of compound **17g** (KBr).



IR spectrum of compound **17h** (KBr).

