

SYNTHESIS AND CHARACTERIZATION OF NEW RACEMIC SECONDARY ALCOHOL WITH 5-BENZYL-4-(4-METHYLPHENYL)-3-SULFANYL-1,2,4-TRIAZOLE STRUCTURE

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Introduction

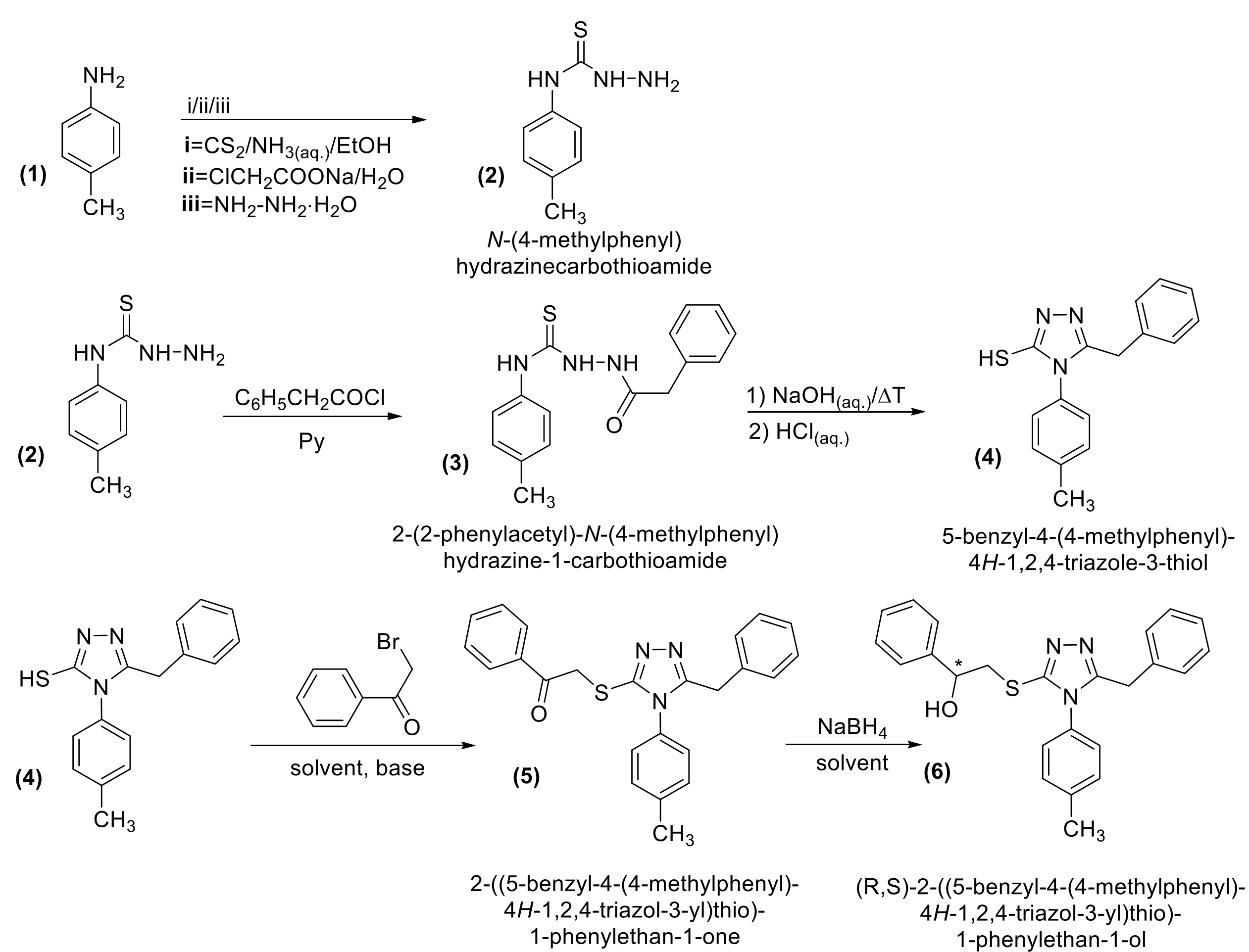
Compounds containing the 1,2,4-triazole ring have a variety of biological activities e. g. anti-inflammatory, anti-HIV, anticonvulsant and antifungal [1-4].

Aim of the study

The 5-benzyl-4-(4-methylphenyl)-4*H*-1,2,4-triazole-3-thiol (**4**) was synthesized using modified procedures from literature [1-4]. Then the compound (**4**) was alkylated with 2-bromo-1-phenylethan-1-one in the presence of caesium carbonate, thus resulting the 2-((5-benzyl-4-(4-methylphenyl)-4*H*-1,2,4-triazol-3-yl)thio)-1-phenylethan-1-one (**5**). The ketone was reduced non-selectively, using sodium borohydride, to the corresponding secondary racemic alcohol (**6**).

All the synthesized compounds were characterized by m. p., IR, 1D and 2D NMR spectroscopy.

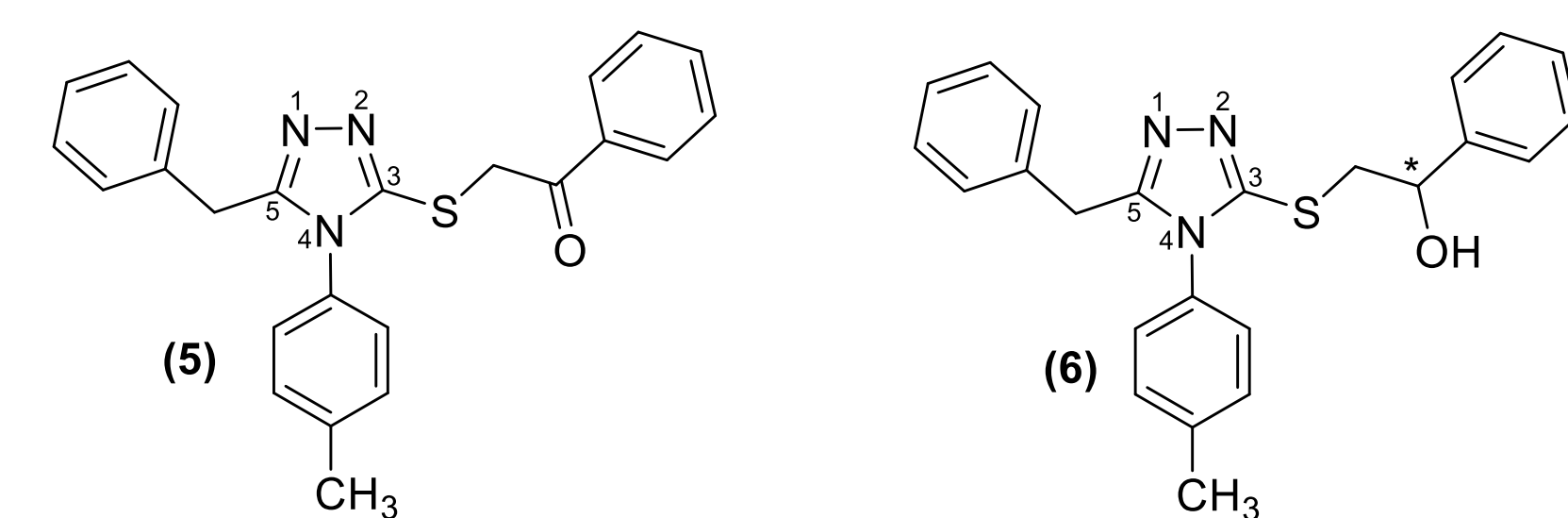
Results and discussion



Scheme 1. Synthetic route for the racemic alcohol

Table 1. Physico-chemical characterization of compounds

Compound	m.p. (°C)
(4)	183-185
(5)	110-112
(6)	118-120



(5) ¹H NMR (300MHz, CDCl₃) δ(ppm): 8,00 (d, 2H); 7,59 (t, 1H); 7,47 (t, 2H); 7,21 – 7,15 (m, 5H); 6,98 – 6,95 (m, 2H); 6,87 (d, 2H); 4,90 (s, 2H); 3,99 (s, 2H, C₆H₅-CH₂); 2,90 (s, 3H, -CH₃);

¹³C NMR (75MHz, CDCl₃) δ(ppm): 193,4 (C=O); 155,3 (5-C); 151,6 (3-C); 135,7; 135,4; 134,0 (CH); 130,5 (CH); 130,3; 128,9 (CH); 128,77 (CH); 128,73 (CH); 128,6 (CH); 127,2 (CH); 127,0 (CH); 41,3 (-CH₂-S); 31,6 (-CH₂-H₅C₆); 21,4 (-CH₃).

(6) ¹H NMR (300MHz, CDCl₃) δ(ppm): 7,44 (d, 2H); 7,36 – 7,16 (m, 8H); 6,99 – 6,96 (m, 2H); 6,87 (d, 2H); 5,31 (s, -OH); 5,13 (dd, 1H, -CH-OH); 3,98 (s, 2H, C₆H₅-CH₂); 3,51 – 3,55 (m, 2H, S-CH₂); 2,41 (s, 3H, -CH₃);

¹³C NMR (75MHz, CDCl₃) δ(ppm): 155,3 (5-C); 153,3 (3-C); 143,2; 140,6; 135,5 (CH); 130,4; 128,8 (CH); 128,7 (CH); 128,6 (CH); 127,8 (CH); 127,1 (CH); 126,0 (CH); 74,2 (-CH-OH); 41,7 (-CH₂-S); 31,7 (-CH₂-H₅C₆); 21,4 (-CH₃).

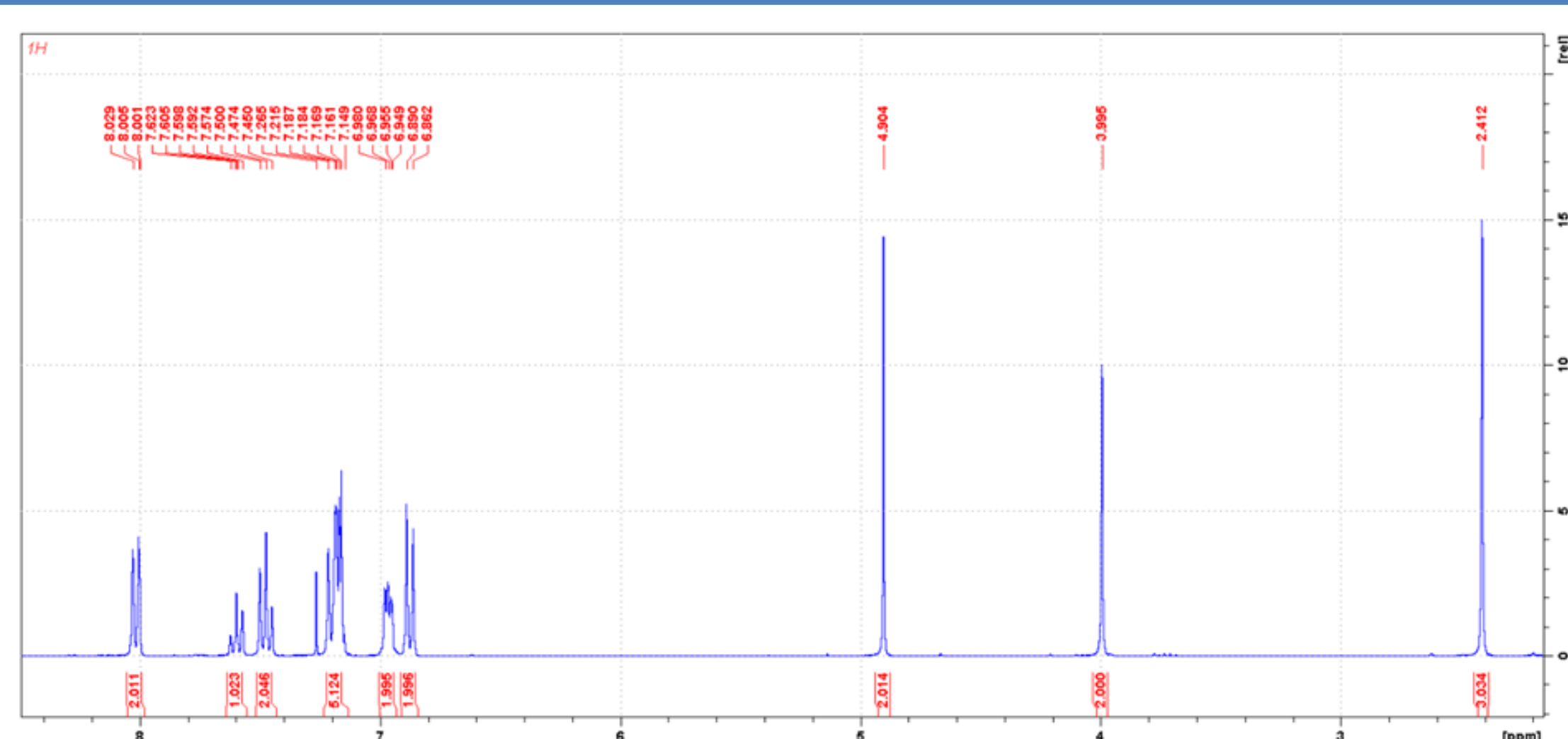


Figure 1. ¹H NMR spectrum of compound (5)

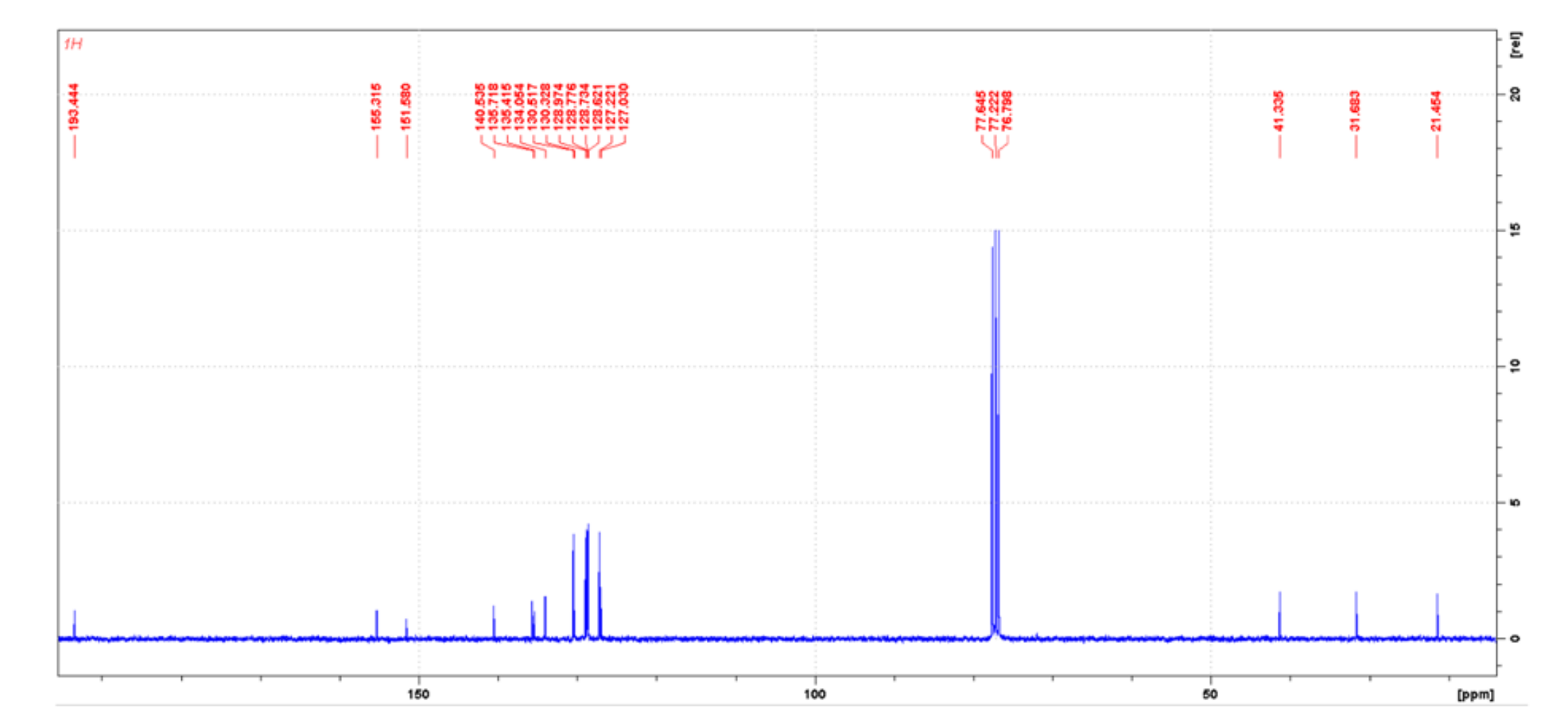


Figure 2. ¹³C NMR spectrum of compound (5)

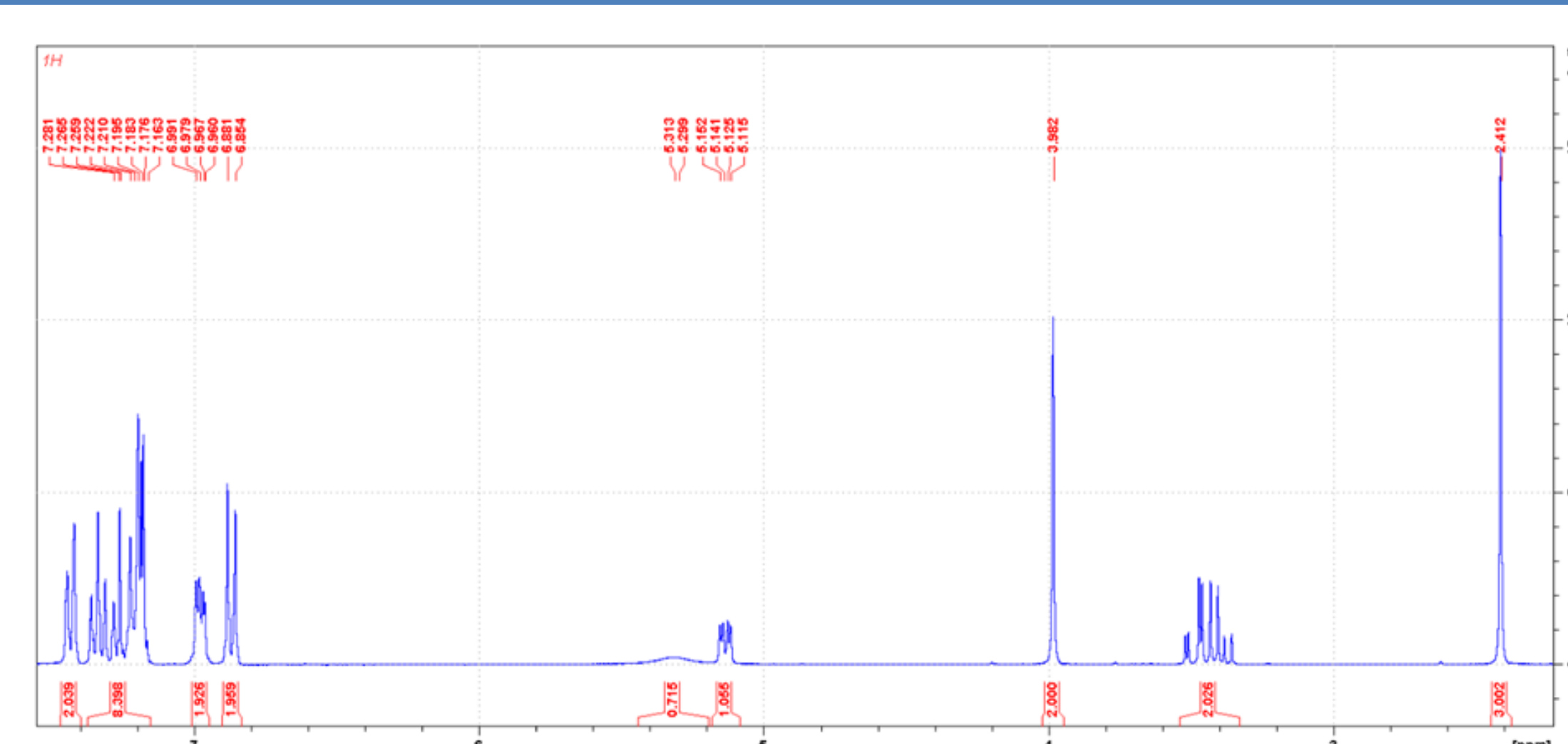


Figure 3. ¹H NMR spectrum of compound (6)

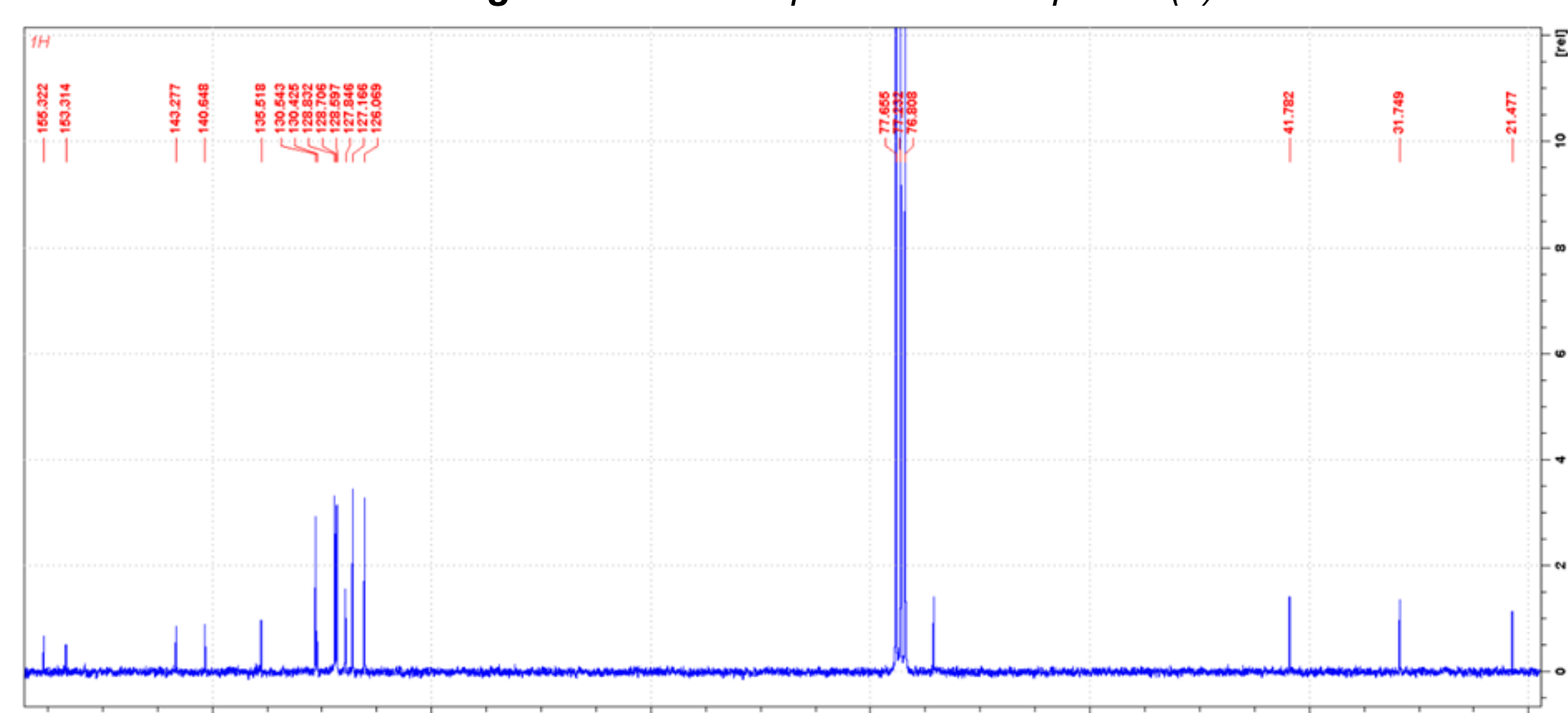


Figure 4. ¹³C NMR spectrum of compound (6)

Conclusions

- Two novel compounds: the ketone 2-((5-benzyl-4-(4-methylphenyl)-4*H*-1,2,4-triazol-3-yl)thio)-1-phenylethan-1-one and the racemic secondary alcohol (R,S)-2-((5-benzyl-4-(4-methylphenyl)-4*H*-1,2,4-triazol-3-yl)thio)-1-phenylethan-1-ol have been successfully synthesized starting from 5-benzyl-4-(4-methylphenyl)-4*H*-1,2,4-triazole-3-thiol.
- The chemical structures have been confirmed by 1D NMR (¹H and ¹³C), 2D NMR (¹H-¹H COSY, ¹H-¹³C HSQC and HMBC, ¹H-¹⁵N HMBC) spectra.

References:

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- [2] Zoumpoulakis, P.; Camoutsis, C.; Pairs, G.; Soković, M.; Glamočlija, J.; Potamitis, C.; Pitsas, A. *Bioorganic and Medicinal Chemistry*, **2012**, *20*(4), 1569-1583;
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Acknowledgements:

This work was supported by a grant of the Ministry of Research, Innovation and Digitization, CNCS/CCCDI – UEFISCDI, project number PN-III-P2-2.1-PED-2019-3414, within PNCDI III.