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Data Article

NMR and MS data for novel bioactive constituents from *Pugionium cornutum* L. Gaertn

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ABSTRACT

The data presented in this article are associated with the research article entitled "**Bioactive Constituents Study of *Pugionium cornutum* L. Gaertn on Intestinal Motility**" [1]. The aim of this data was to provide the 1D, 2D NMR and MS spectrum of novel bioactive compounds from *P. cornutum*.

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Specifications Table

Subject	Chemistry
Specific subject area	Natural products research
Type of data	Figure
How data were acquired	First, the compounds were isolated from the 70% ethanol-water extract of <i>P. cornutum</i> roots. Then the samples were dissolved in DMSO- <i>d</i> ₆ or CD ₃ OD before NMR test.
Data format	Raw and Analyzed
Parameters for data collection	The NMR spectra data parameters including chemical shift δ -values and coupling constant (<i>J</i>). The MS spectra data parameters including <i>m/z</i> .
Description of data collection	NMR spectra data of novel compounds from the roots of <i>P. cornutum</i> were recorded on a Bruker Advance-500 spectrometer using standard Bruker pulse programs (Bruker, Karlsruhe, Germany). Chemical shifts were shown as δ -values with reference to tetramethylsilane (TMS) as an internal standard. Positive- and negative-ion HRESI-TOF-MS of novel compounds from <i>P. cornutum</i> roots were recorded on an Agilent Technologies 6520 Accurate-Mass Q-ToF LC/MS spectrometer.
Data source location	Tianjin State Key Laboratory of Modern Chinese Medicine, Tianjin, China
Data accessibility	Data is with the article [1]
Related research article	W. Shi, J. Ruan, Y. Guo, Z. Ding, J. Yan, L. Qu, C. Zheng, Y. Zhang, T. Wang, Bioactive constituents study of <i>Pugionium cornutum</i> L. Gaertn on intestinal motility, <i>Fitoerapia</i> , 138 (2019) 104291.

Value of the Data

- NMR and MS data of novel compounds is important for elucidating their chemical structures.
- NMR and MS data of novel thiohydantoin derivatives is useful for the elucidating their chemical analogues.
- This information will allow comparisons across different thiohydantoin derivatives and other new compounds from *Pugionium* species or Cruciferae family plant sources.

1. Data

The dataset contains raw analysis data obtained through the chemistry research of the dried roots of Mongolian medicinal and edible plant, *Pugionium cornutum* L. Gaertn. Information about the eight new compounds pugnornols A (**1**), B (**2**), C (**3**), and D (**4**), pugnornosides A (**5**), B (**6**), C (**7**), and D (**8**) has been presented in Figs. 1–56.

2. Experimental design, materials, and methods

2.1. Study area description

Pugionium cornutum L. Gaertn (PC), Cruciferae family, is widely distributed in the Badain Jaran Desert, Kubuqi Desert, Mu Us Desert, Horqin sandy land, and Hulunbuir sandy land [2]. As the traditional arenicolous Mongolian medicine, PC has accumulated rich experience and knowledge of promoting gastrointestinal motility and improving indigestion [3]. Previously, a further phytochemical investigation on the 70% ethanol-water extract of the roots of *P. cornutum* led to the isolation and characterization of eight compounds that have been not reported previously. Among them, **1–4** were rare naturally occurring thiohydantoin derivatives. Herein, their structure characterization has been identified by various spectrometry methods including NMR and MS spectra.

2.2. Plant material

The roots of *Pugionium cornutum* L. Gaertn were collected from Alxa Youqi, Inner Mongolia Autonomous region, China, and identified by Dr. Li Tianxiang (Experiment Teaching Department,

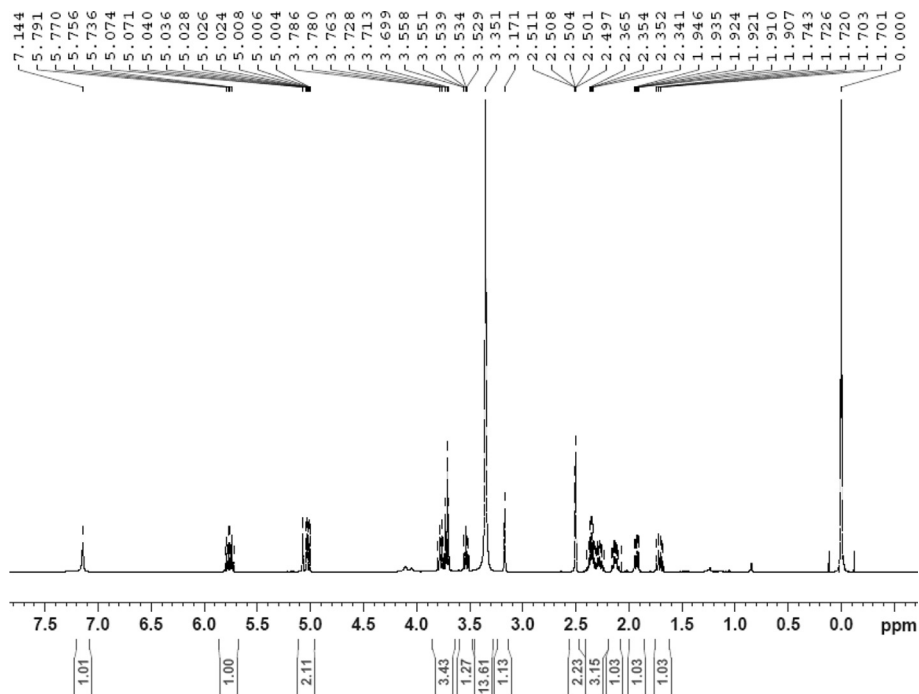


Fig. 1. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound 1.

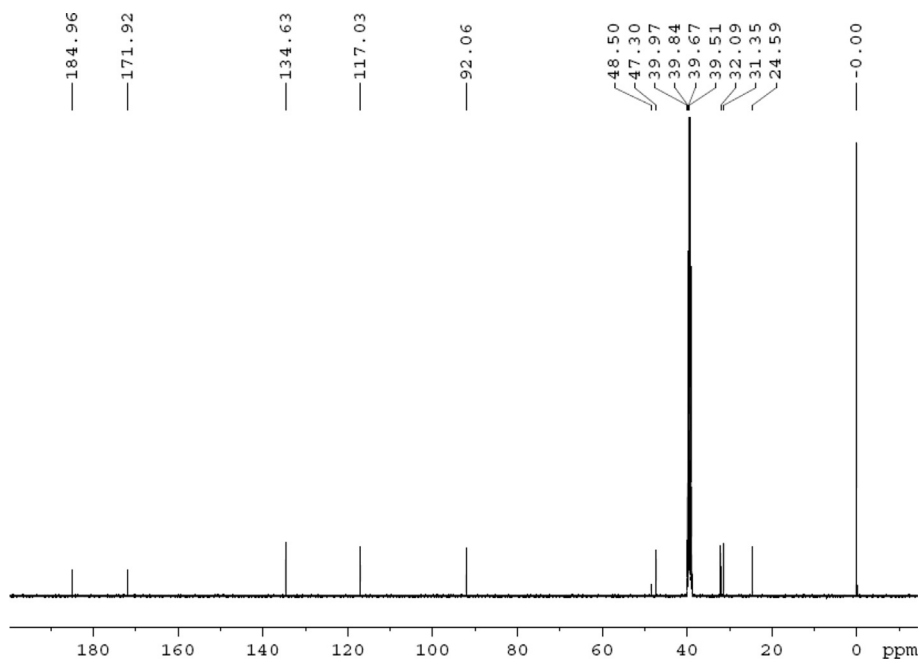


Fig. 2. ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$) spectrum of compound 1.

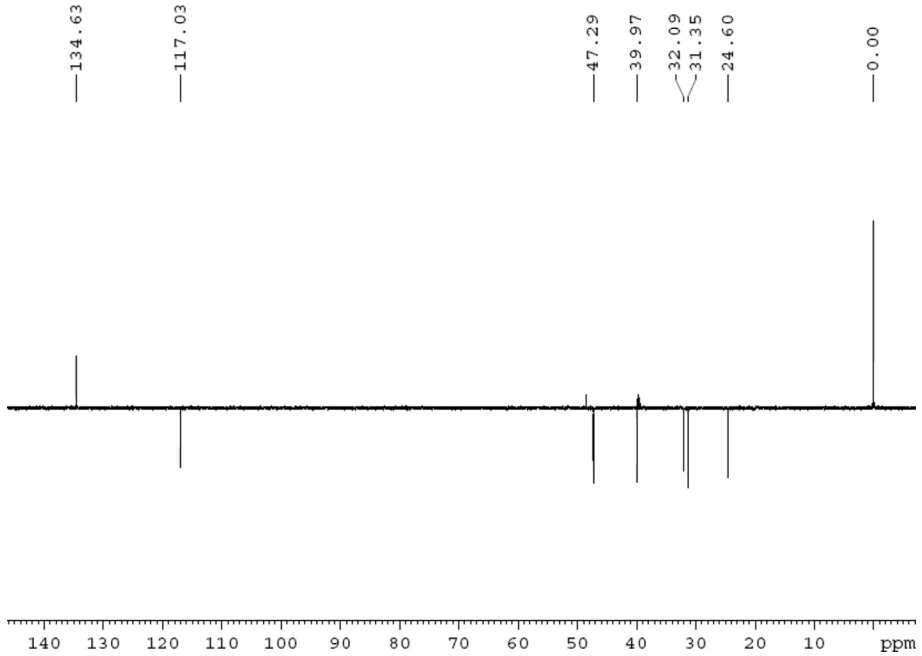


Fig. 3. DEPT 135 (DMSO- d_6) spectrum of compound 1.

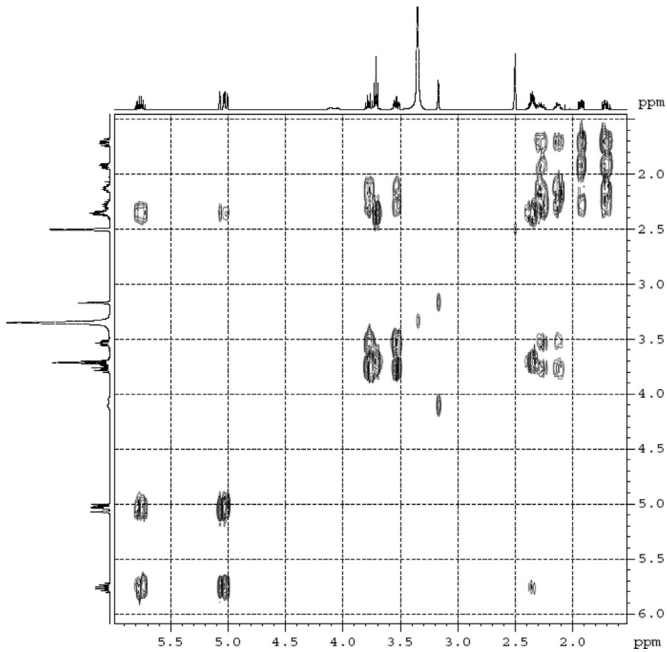


Fig. 4. ^1H - ^1H COSY (DMSO- d_6) spectrum of compound 1.

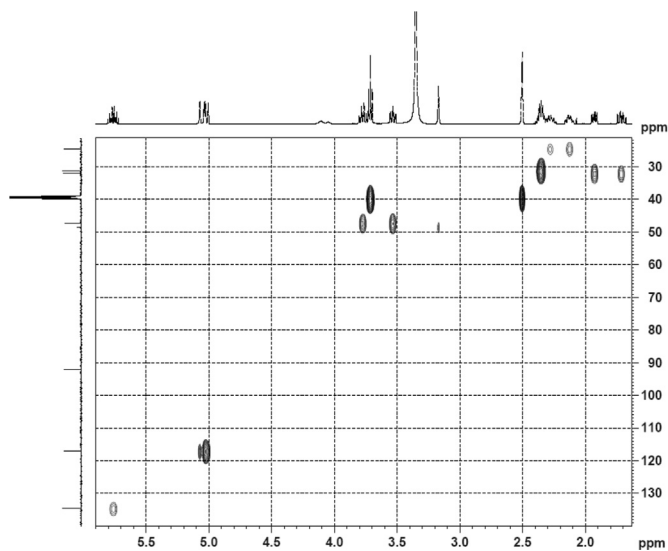


Fig. 5. HSQC (DMSO- d_6) spectrum of compound 1.

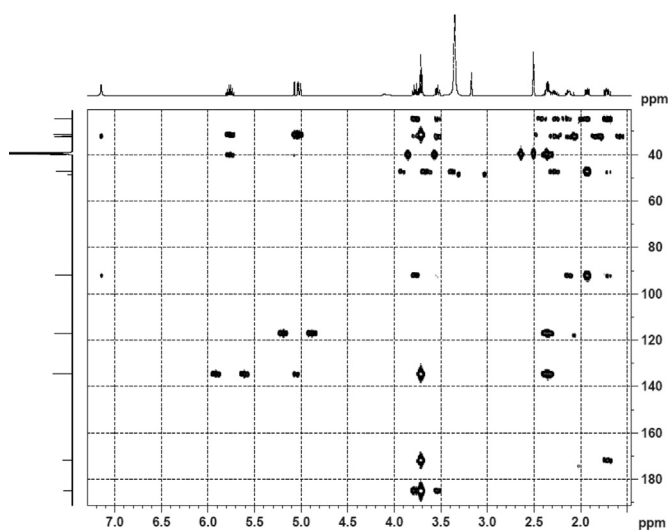


Fig. 6. HMBC (DMSO- d_6) spectrum of compound 1.

Tianjin University of Traditional Chinese Medicine). The voucher specimen was deposited at the Academy of Traditional Chinese Medicine of Tianjin University of TCM.

2.3. NMR and MS spectrum of the isolates

The dried roots of PC were refluxed with 70% ethanol-water for three times. Evaporation of the solvent under pressure provided a 70% ethanol-water extract, which was partitioned with H₂O and

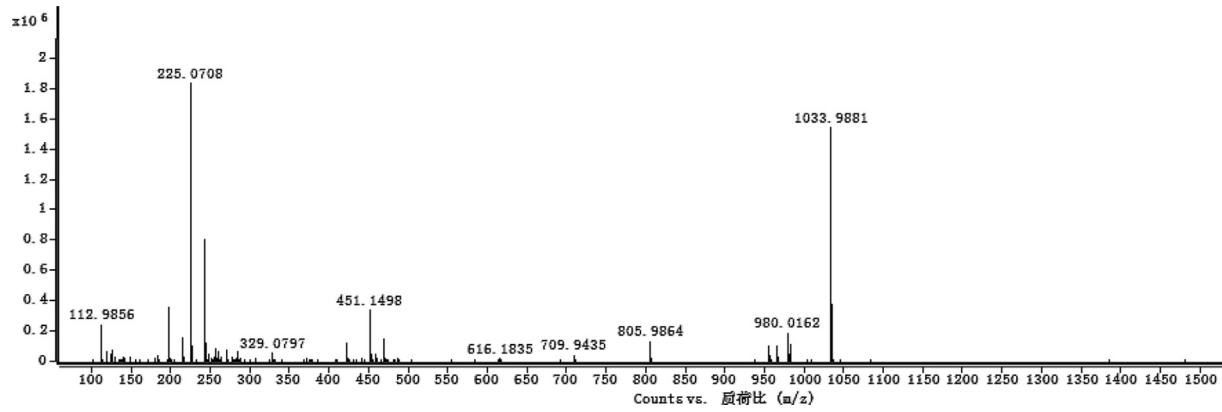


Fig. 7. HRESI-TOF-MS spectrum of compound 1.

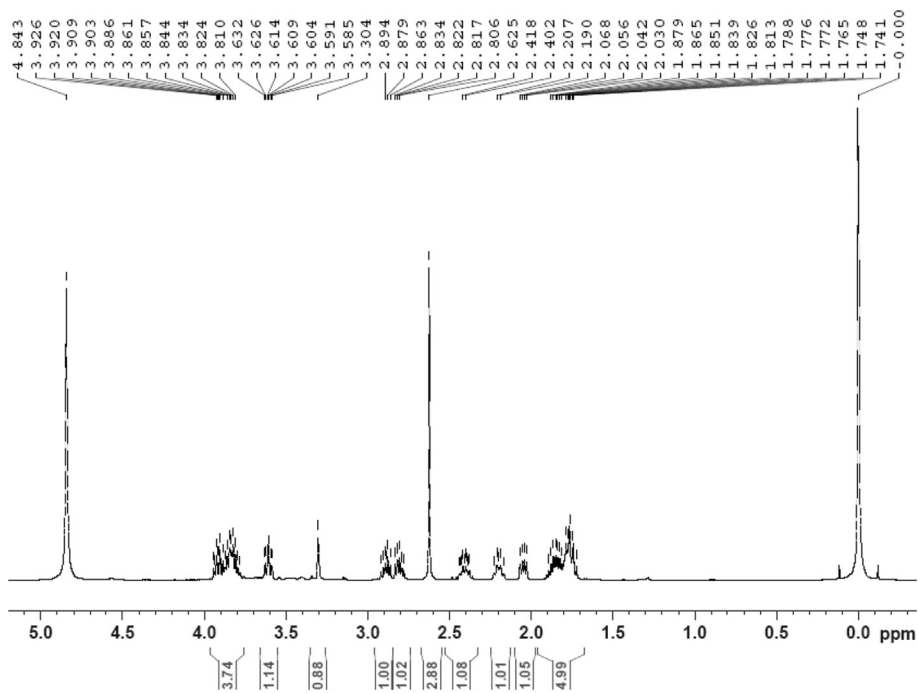


Fig. 8. ^1H NMR (500 MHz, CD_3OD) spectrum of compound 2.

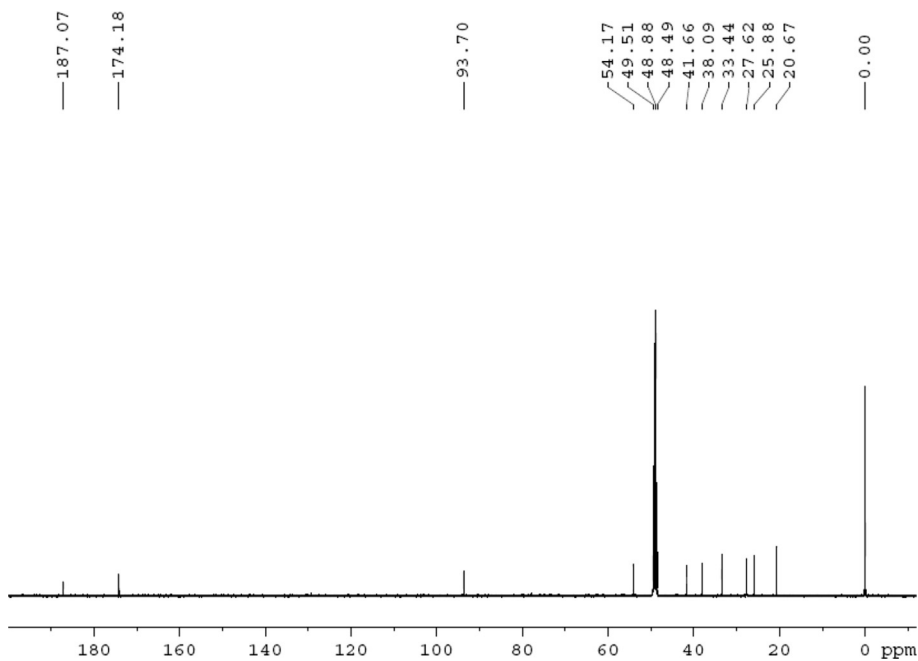


Fig. 9. ^{13}C NMR (125 MHz, CD_3OD) spectrum of compound 2.

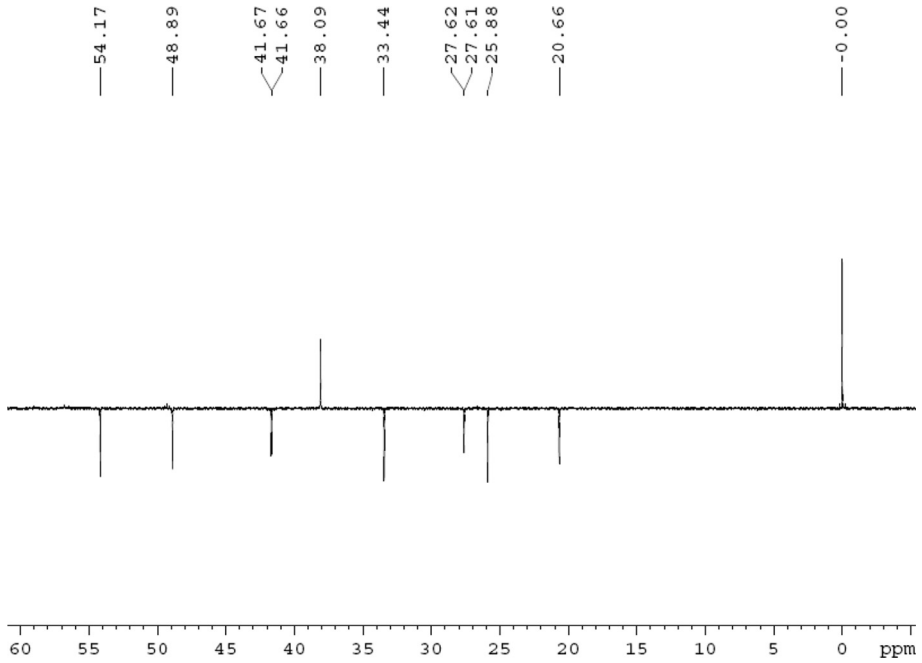


Fig. 10. DEPT 135 (CD_3OD) spectrum of compound 2.

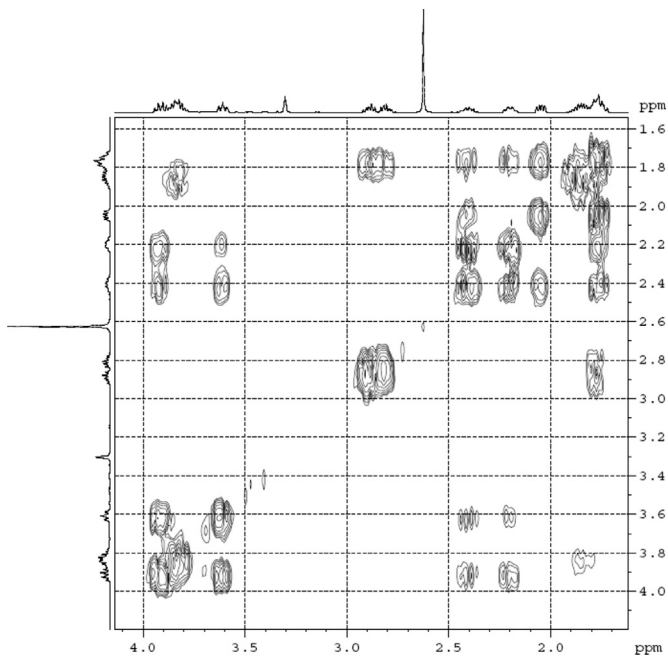


Fig. 11. ^1H ^1H COSY (CD_3OD) spectrum of compound 2.

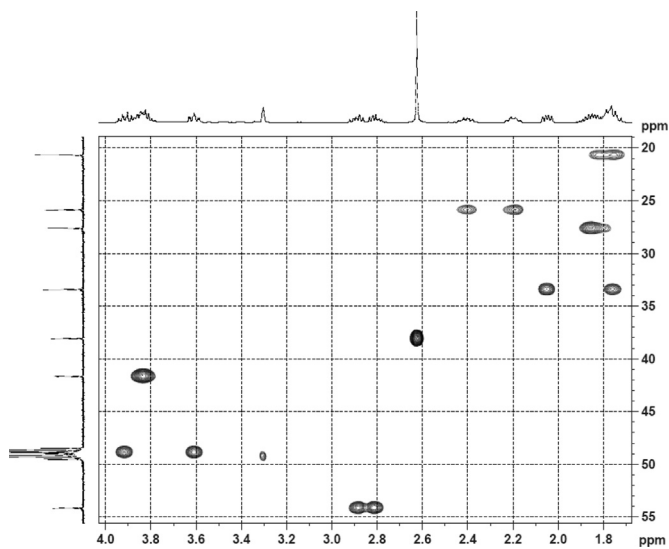


Fig. 12. HSQC (CD₃OD) spectrum of compound 2.

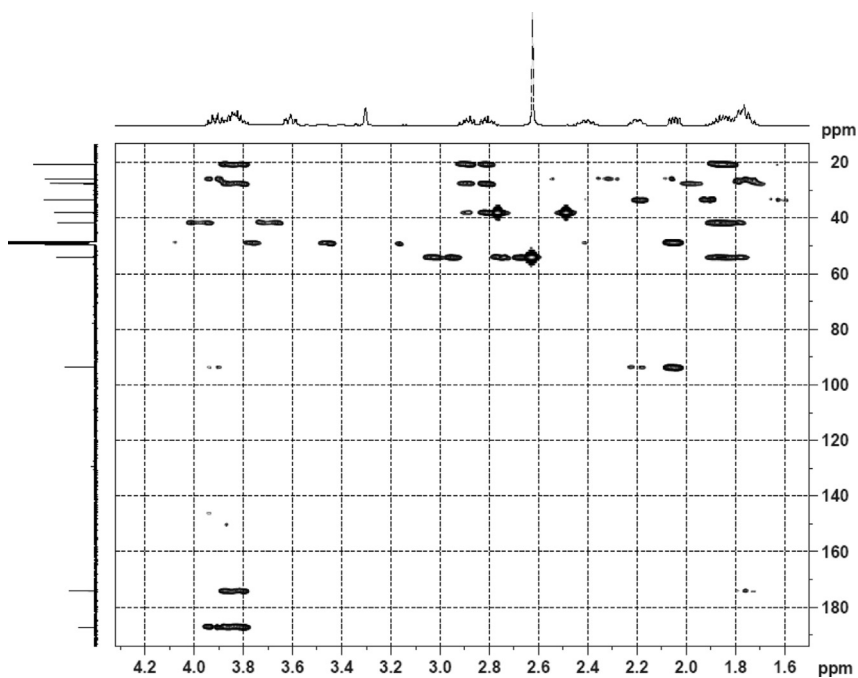


Fig. 13. HMBC (CD₃OD) spectrum of compound 2.

EtOAc to gain H₂O layer and EtOAc layer partition, respectively. The H₂O layer partition was subjected to D101 macroporous resin CC and eluted with H₂O, 95% EtOH, and acetone, successively. Then 95% EtOH eluate and EtOAc layer partition was isolated by silica gel, Sephadex LH-20 chromatography columns and preparative HPLC. As results, four new rare thiohydantoin derivatives pugcornols A (1), B

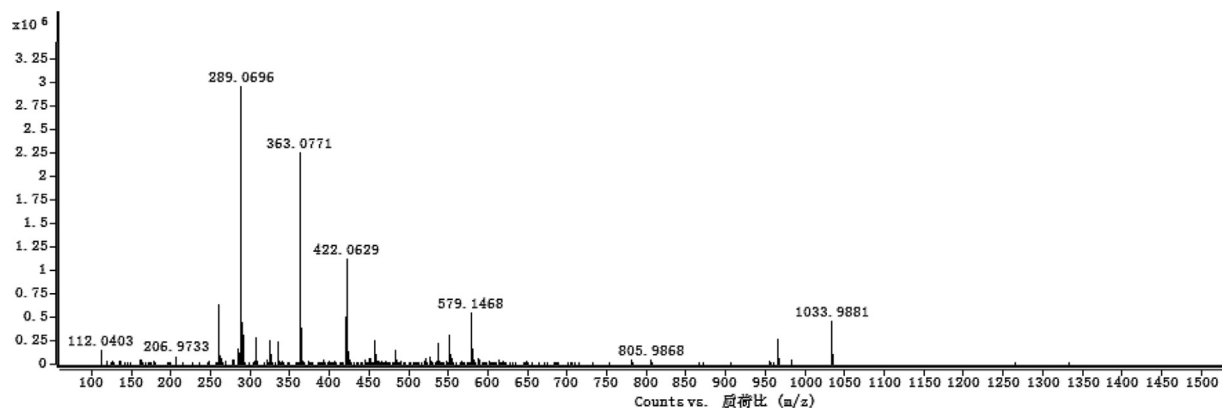


Fig. 14. HRESI-TOF-MS spectrum of compound 2.

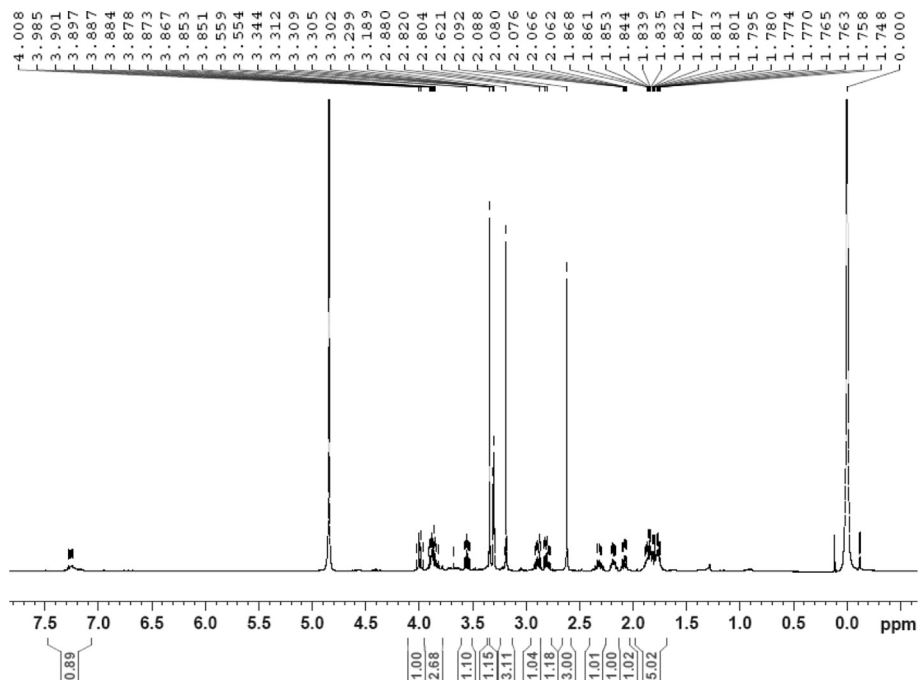


Fig. 15. ^1H NMR (500 MHz, CD_3OD) spectrum of compound 3.

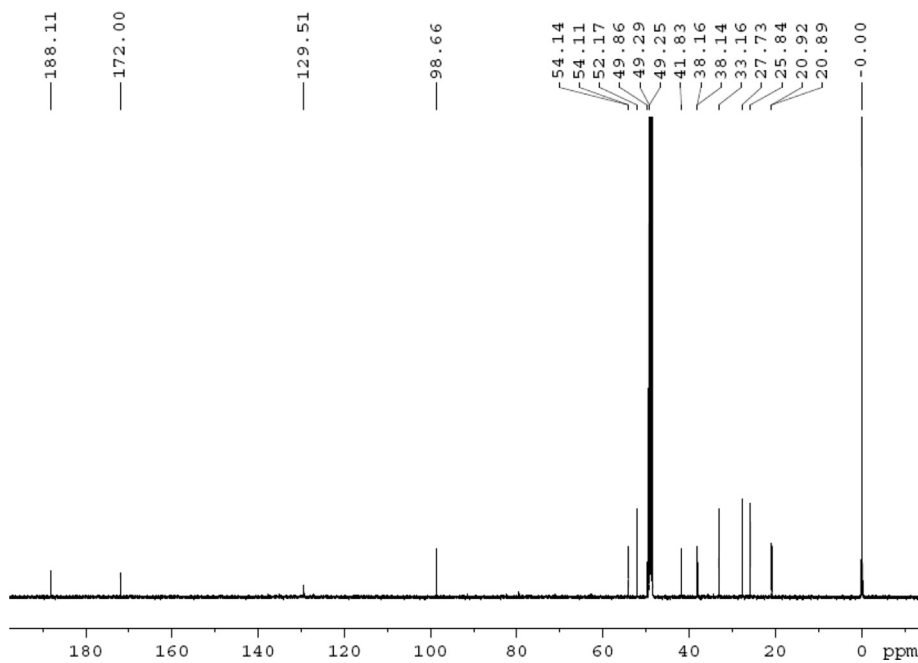


Fig. 16. ^{13}C NMR (125 MHz, CD_3OD) spectrum of compound 3.

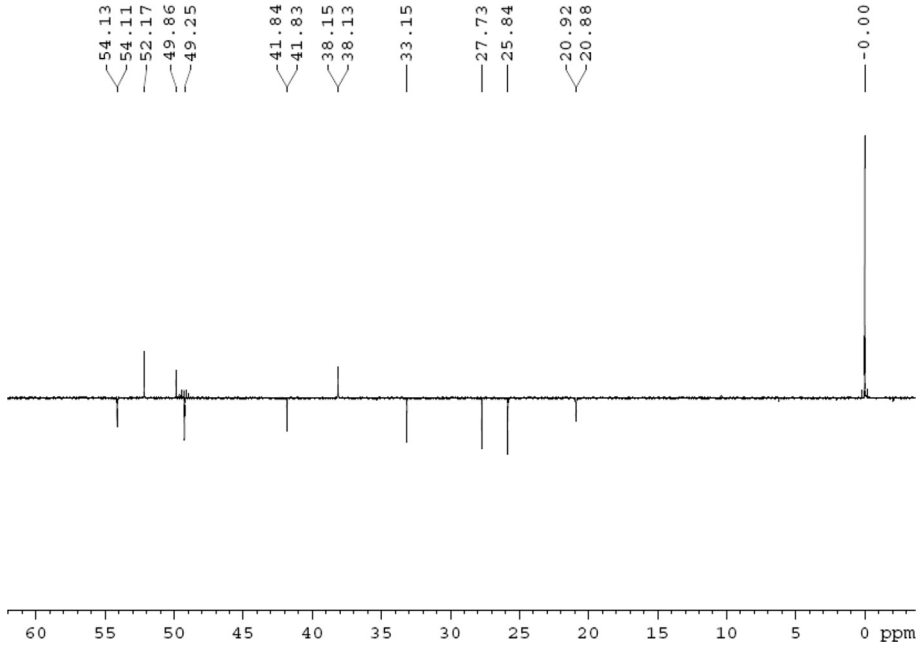


Fig. 17. DEPT 135 (CD_3OD) spectrum of compound 3.

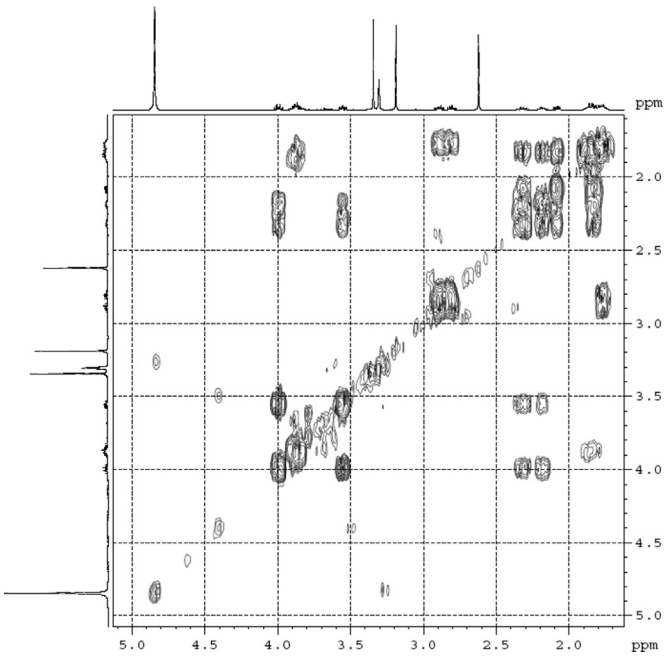


Fig. 18. ^1H - ^1H COSY (CD_3OD) spectrum of compound 3.

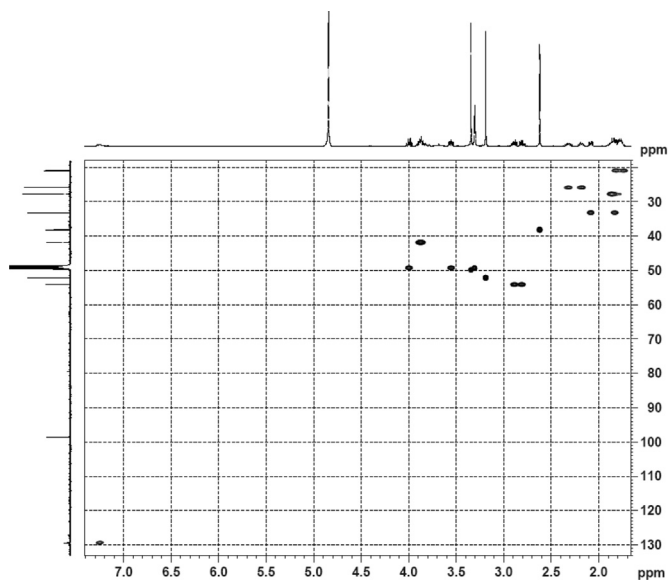


Fig. 19. HSQC (CD₃OD) spectrum of compound 3.

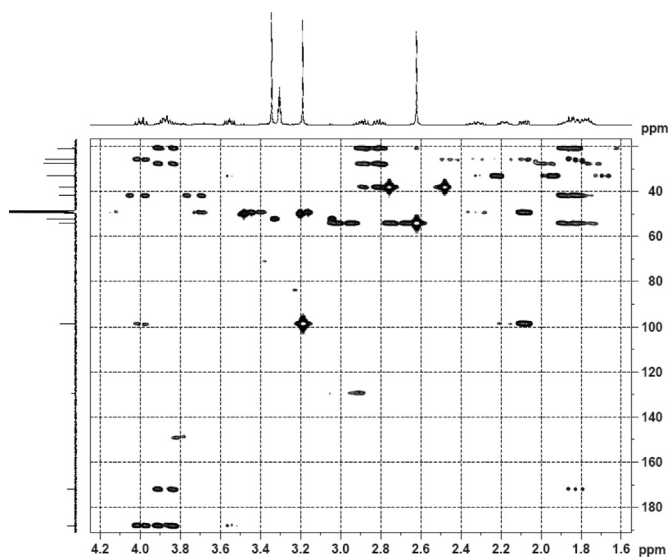


Fig. 20. HMBC (CD₃OD) spectrum of compound 3.

(2), C (3), and D (4) [4–7], two new glucosinolates pugcornosides A (5) [8–10], B (6) [10], two new others pugcornosides C (7) [11], D (8) were obtained and identified by using NMR, MS, ECD technologies, as well as chemical reaction.

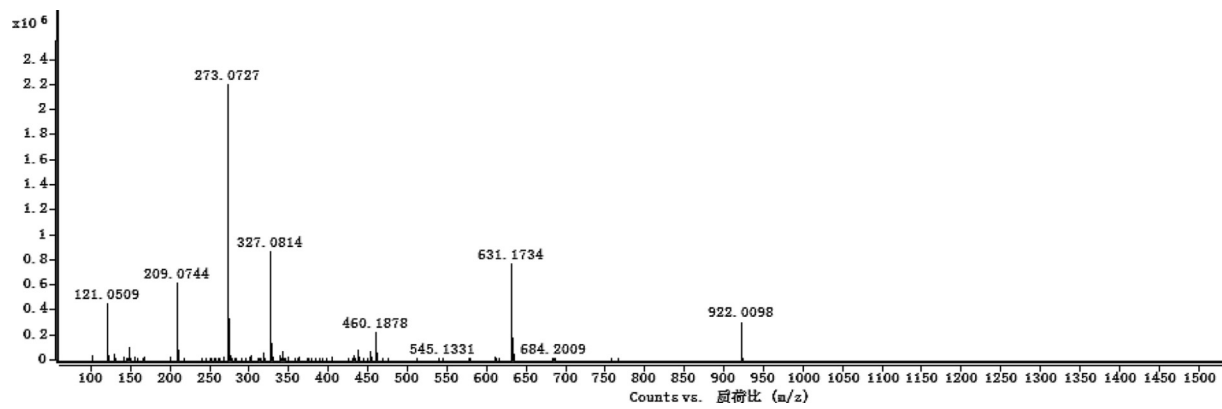


Fig. 21. HRESI-TOF-MS spectrum of compound 3.

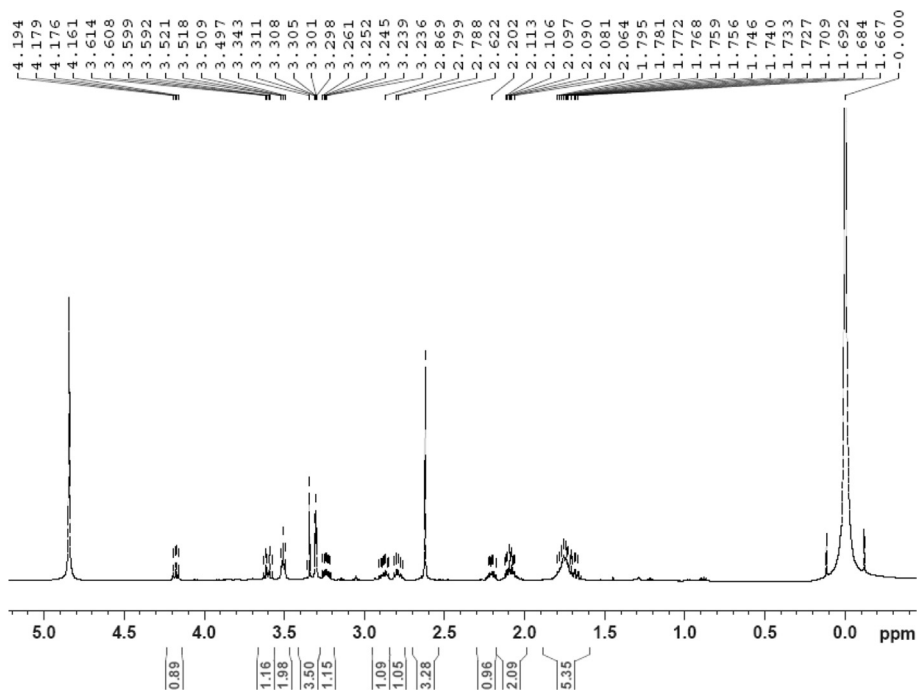


Fig. 22. ^1H NMR (500 MHz, CD_3OD) spectrum of compound 4.

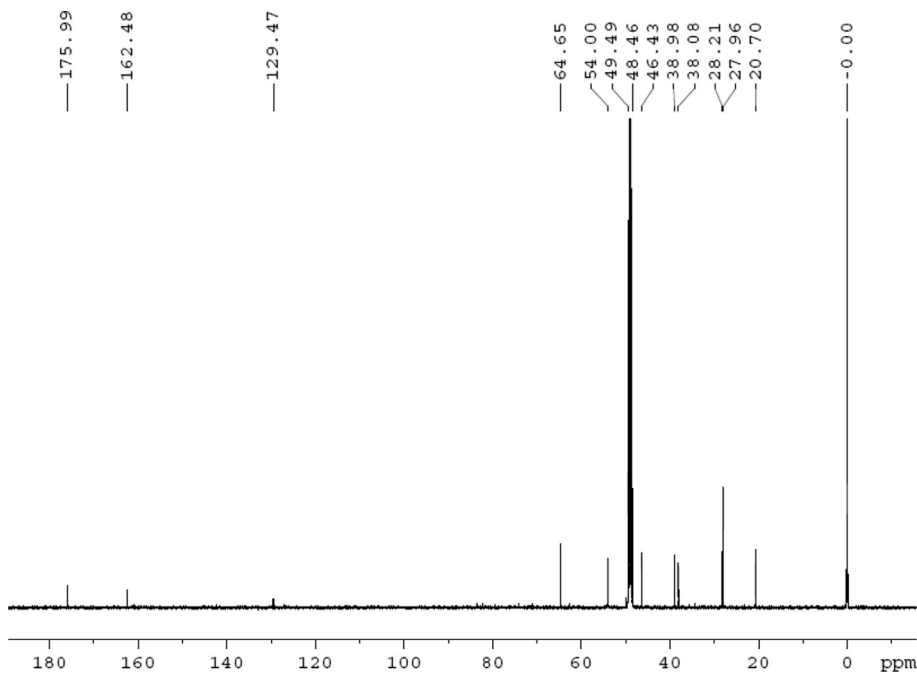


Fig. 23. ^{13}C NMR (125 MHz, CD_3OD) spectrum of compound 4.

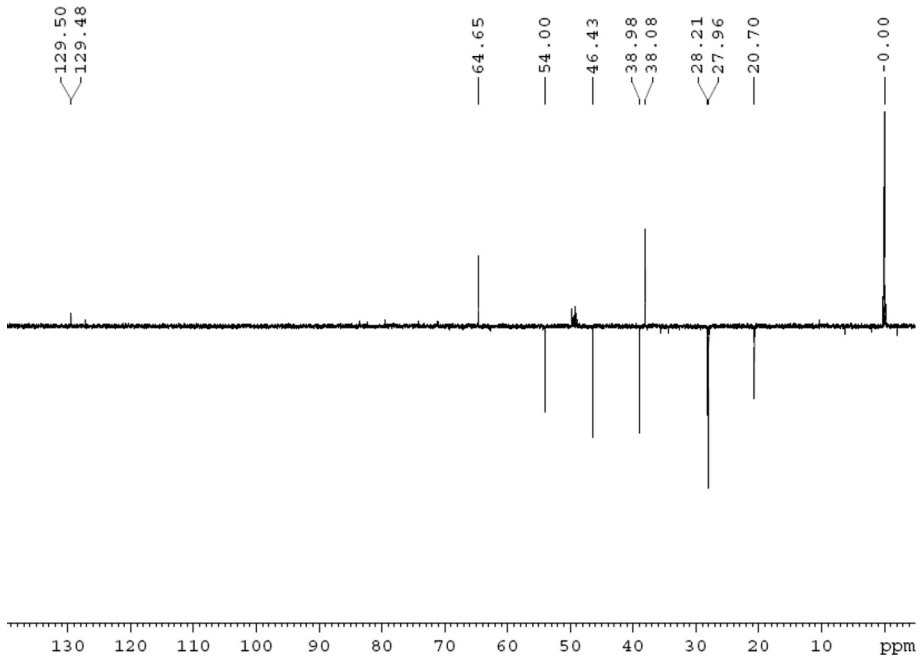


Fig. 24. DEPT 135 (CD₃OD) spectrum of compound 4.

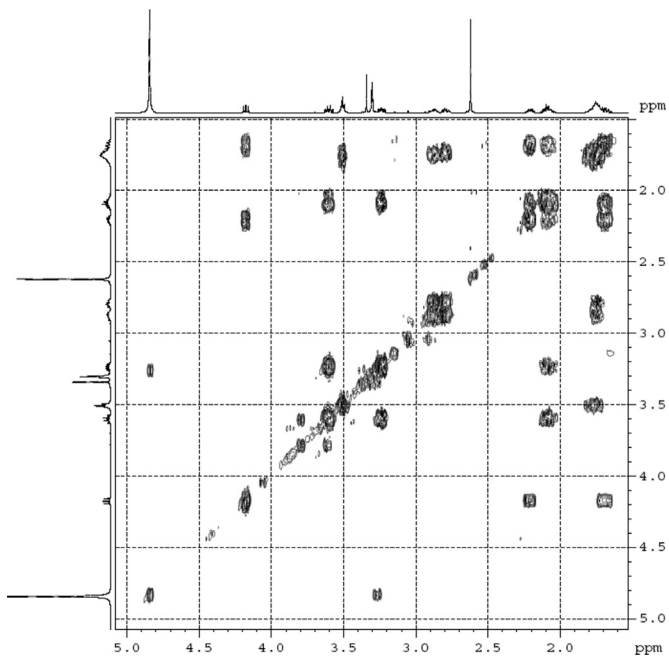


Fig. 25. ¹H ¹H COSY (CD₃OD) spectrum of compound 4.

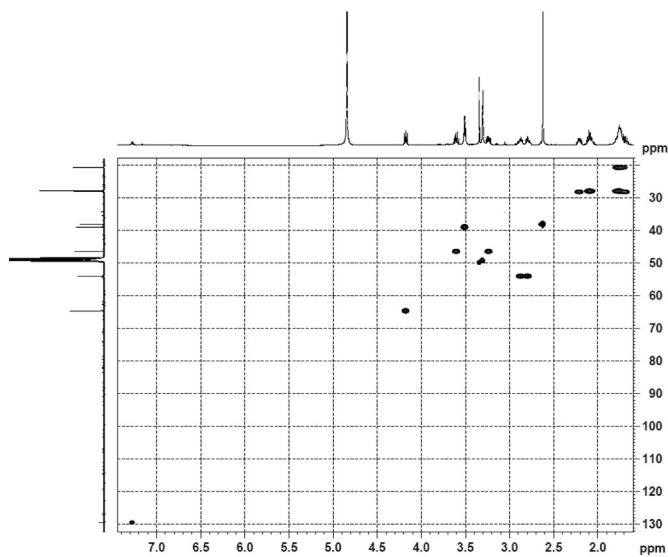


Fig. 26. HSQC (CD₃OD) spectrum of compound 4.

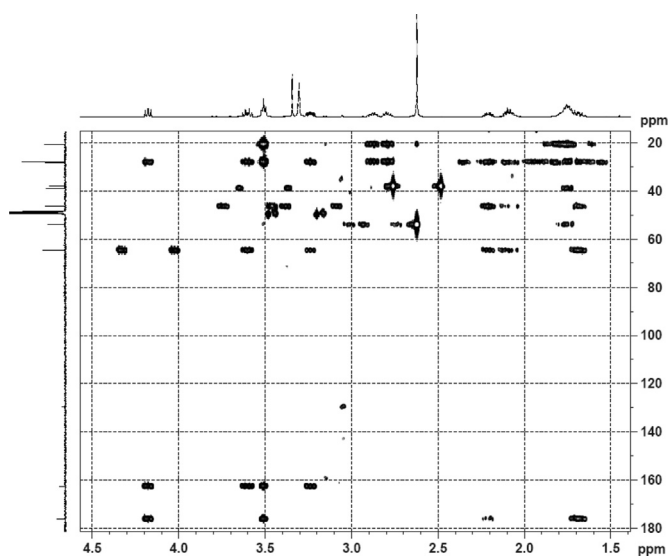


Fig. 27. HMBC (CD₃OD) spectrum of compound 4.

2.3.1. Pugcornol A (**1**)

Colorless oil; $[\alpha]_D^{25}$ -31.3 (conc. 0.12, MeOH); CD (conc. 0.0015 M, MeOH) mdeg (λ_{nm}): -1.39 (235), -3.33 (256), -3.60 (269); CD (conc. 0.0015 M, CH₃CN) mdeg (λ_{nm}): -1.21 (229), -2.83 (245), -4.52 (267); UV λ_{max} (MeOH) nm ($\log \epsilon$): 269 (3.81); IR ν_{max} (KBr) cm^{-1} : 3364, 2943, 1750, 1424, 1347, 1225,

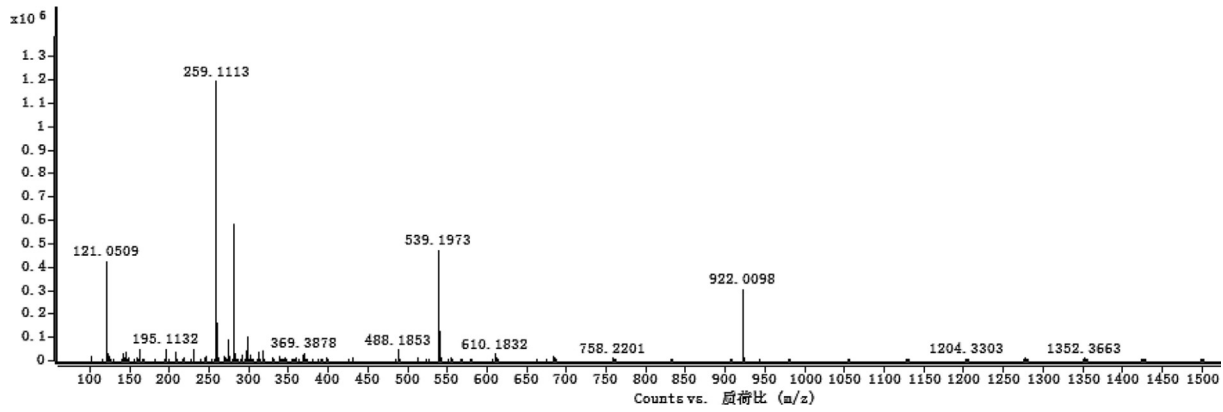


Fig. 28. HRESI-TOF-MS spectrum of compound 4.

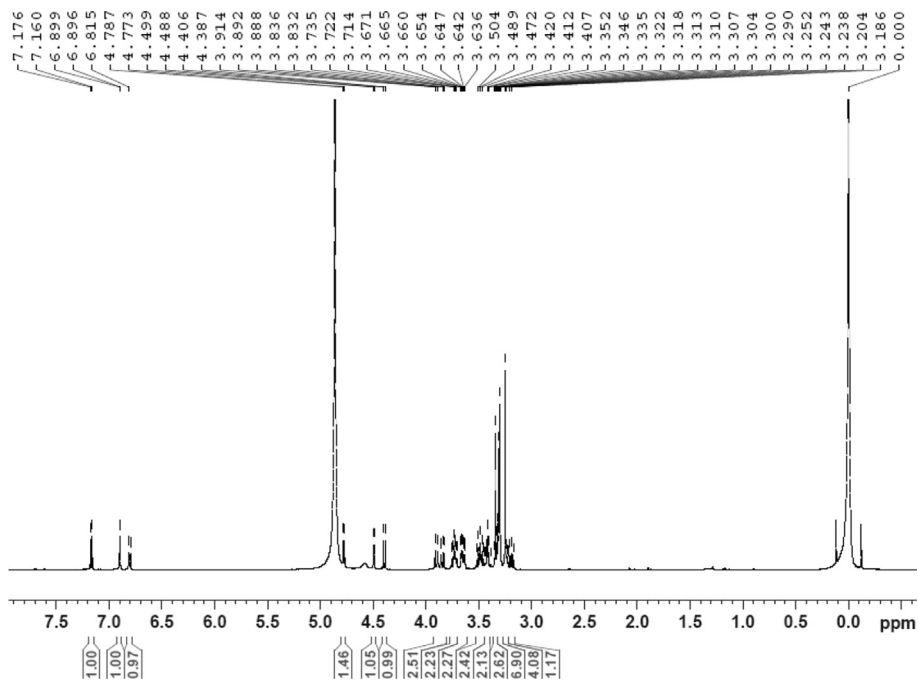


Fig. 29. ^1H NMR (500 MHz, CD_3OD) spectrum of compound 5.

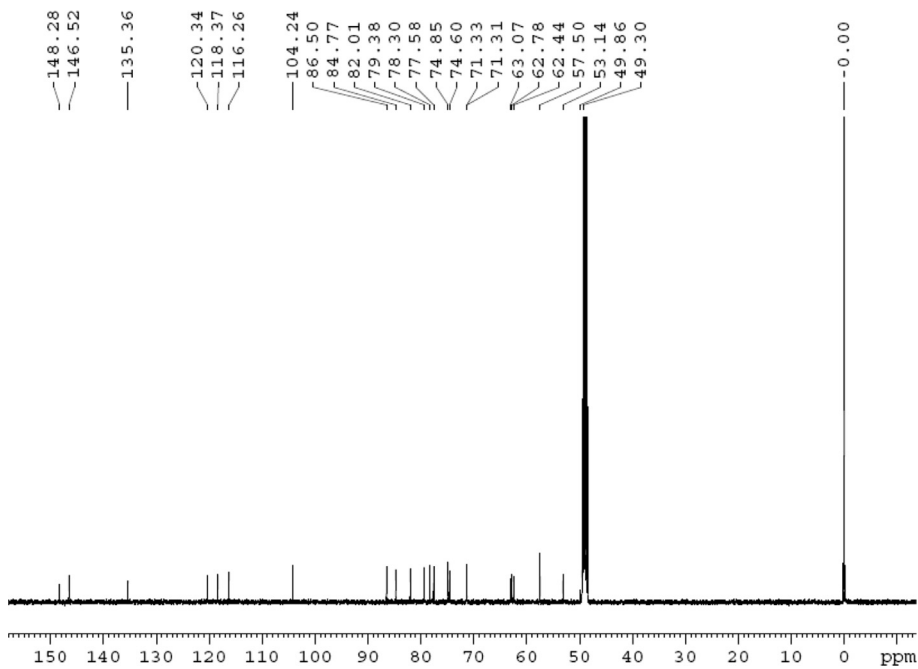


Fig. 30. ^{13}C NMR (125 MHz, CD_3OD) spectrum of compound 5.

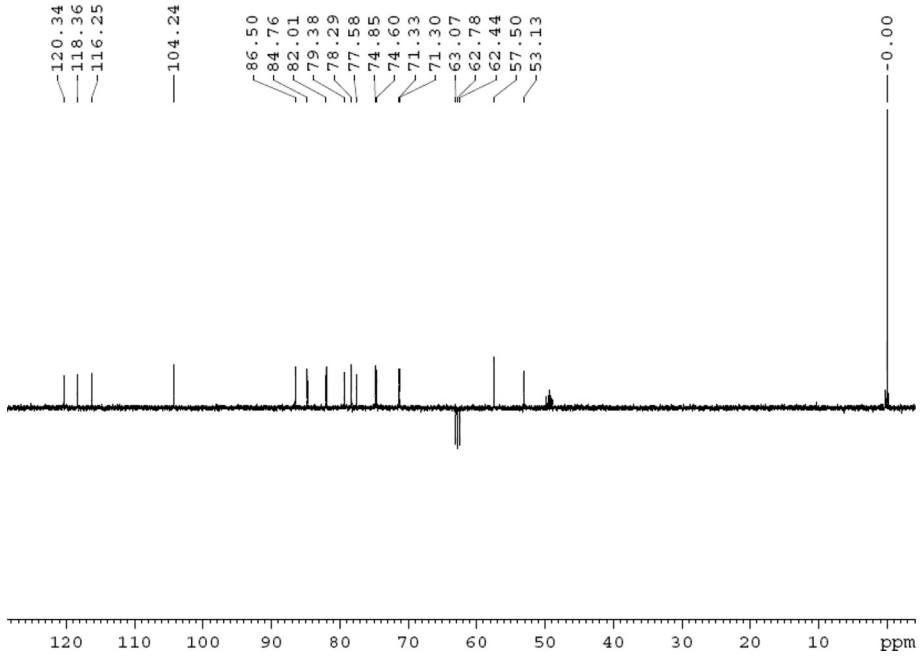


Fig. 31. DEPT 135 (CD_3OD) spectrum of compound 5.

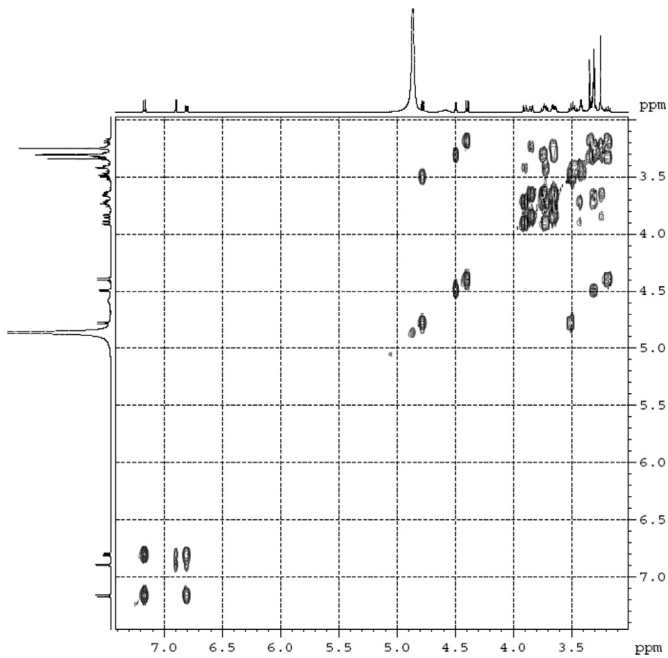


Fig. 32. ^1H - ^1H COSY (CD_3OD) spectrum of compound 5.

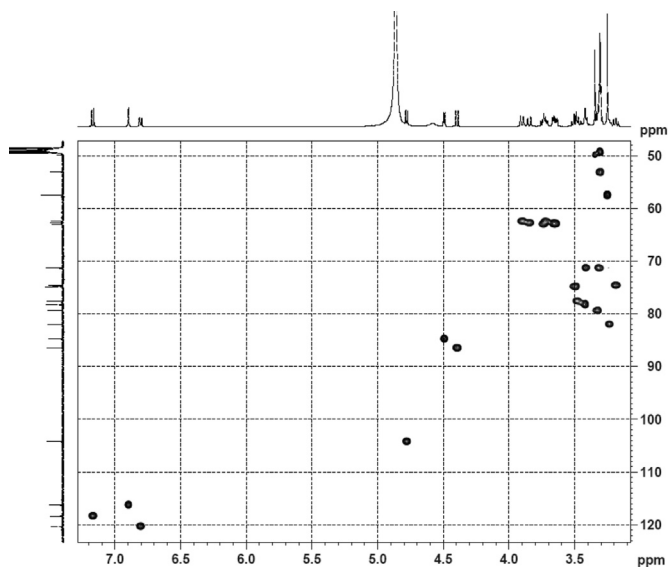


Fig. 33. HSQC (CD₃OD) spectrum of compound 5.

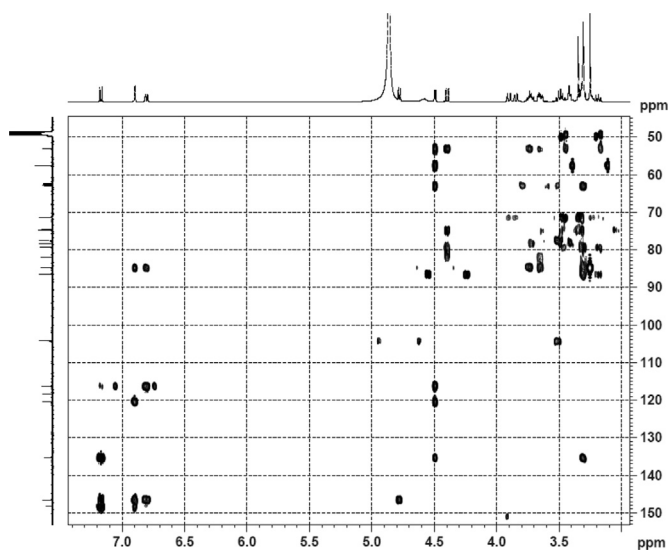


Fig. 34. HMBC (CD₃OD) spectrum of compound 5.

1124, 1061, 1025; ¹H NMR (DMSO-*d*₆, 500 MHz) and ¹³C NMR (DMSO-*d*₆, 125 MHz) data see Figs. 1–6. HRESI-TOF-MS data see Fig. 7.

2.3.2. *Pugcornol B* (2)

Colorless oil; $[\alpha]_D^{25}$ -39.3 (conc. 0.12, MeOH); CD (conc. 0.0015 M, MeOH) mdeg (λ_{nm}): -1.30 (223), -5.34 (270); CD (conc. 0.0015 M, CH₃CN) mdeg (λ_{nm}): -1.56 (223), -6.80 (252), -4.81 (272); UV λ_{max}

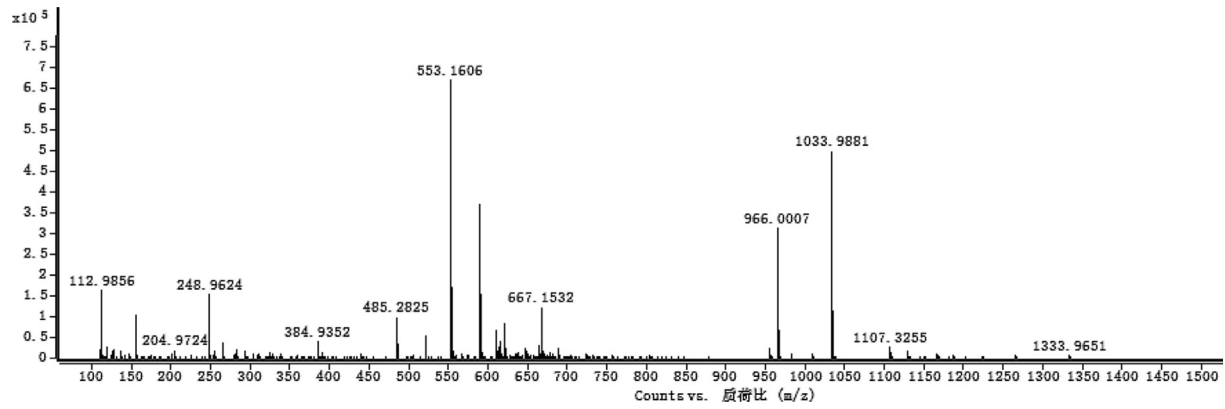


Fig. 35. HRESI-TOF-MS spectrum of compound 5.

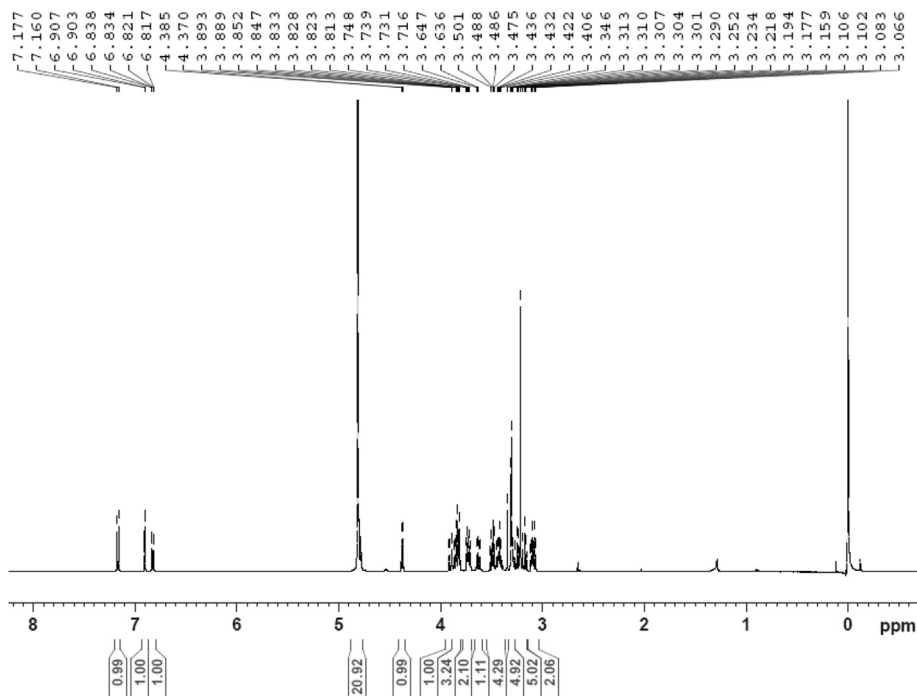


Fig. 36. ¹H NMR (500 MHz, CD₃OD) spectrum of compound 6.

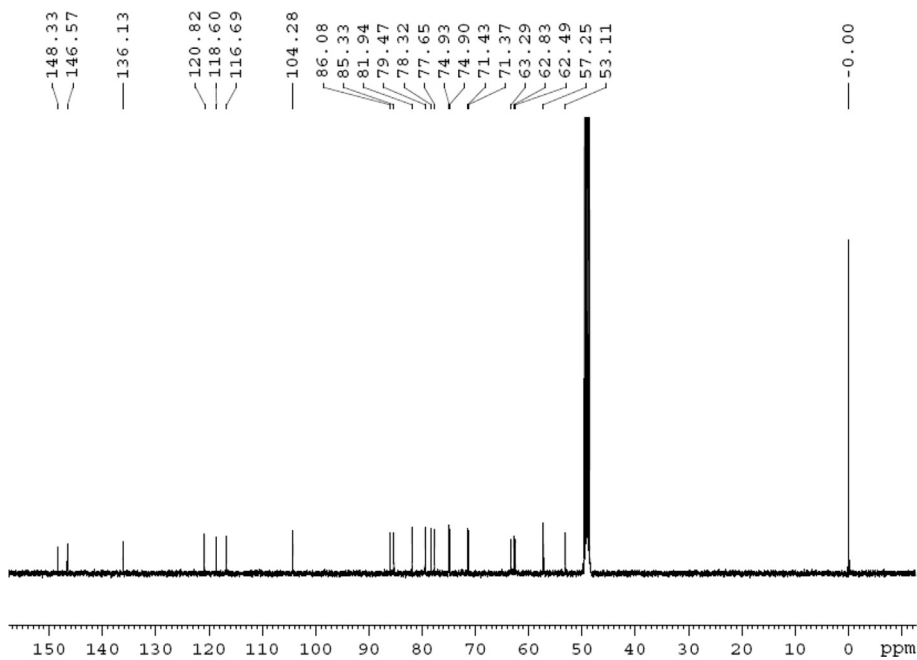


Fig. 37. ¹³C NMR (125 MHz, CD₃OD) spectrum of compound 6.

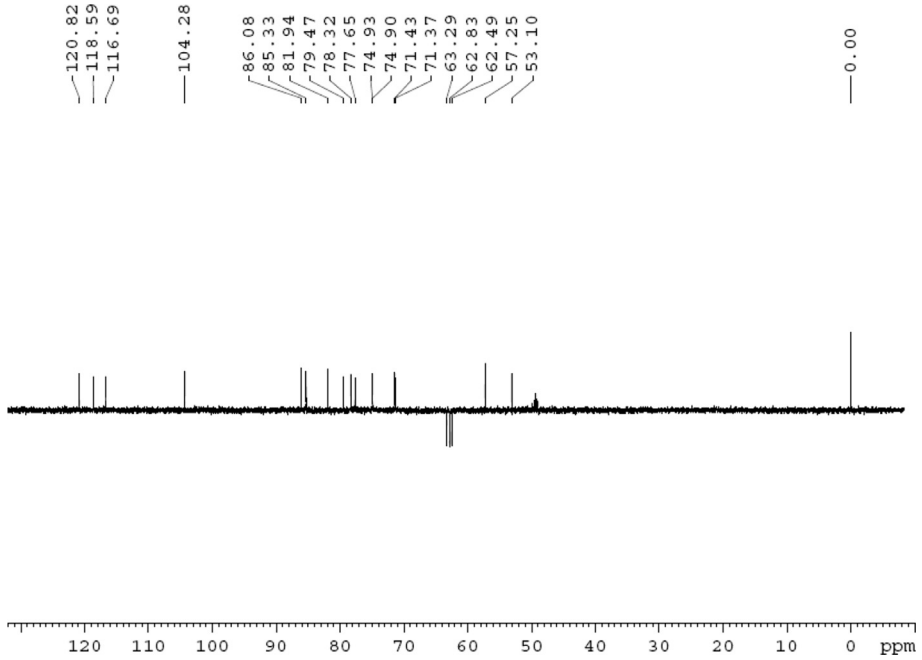


Fig. 38. DEPT 135 (CD₃OD) spectrum of compound 6.

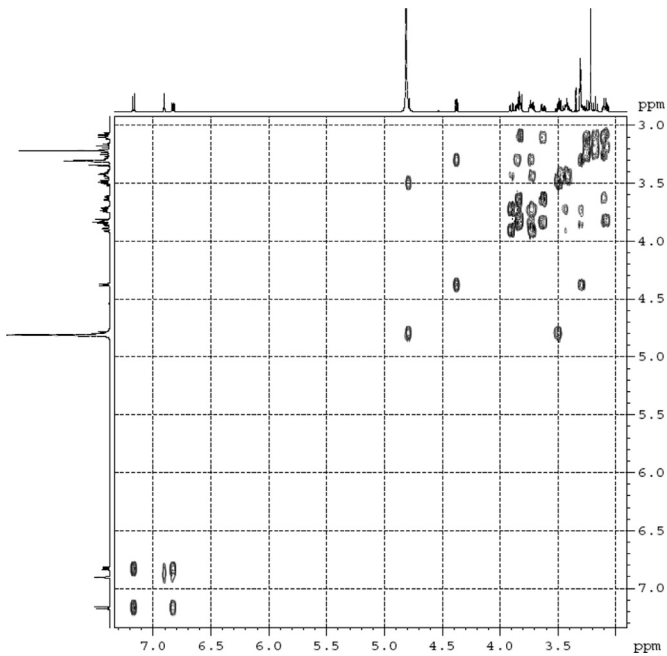


Fig. 39. ¹H-¹H COSY (CD₃OD) spectrum of compound 6.

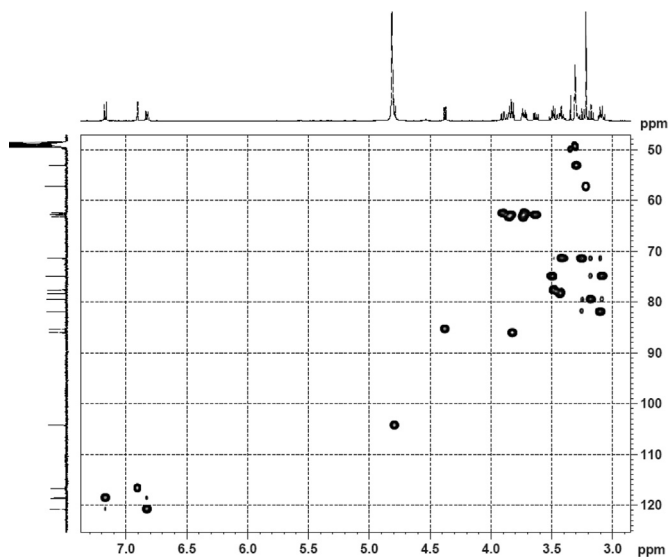


Fig. 40. HSQC (CD₃OD) spectrum of compound 6.

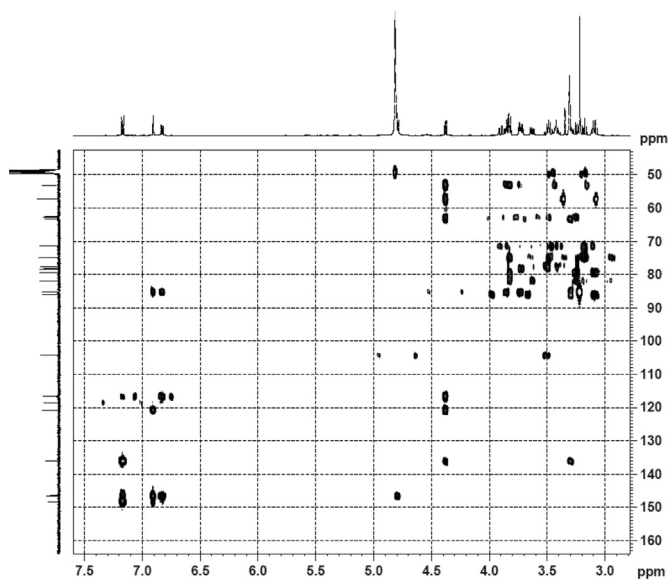


Fig. 41. HMBC (CD₃OD) spectrum of compound 6.

(MeOH) nm ($\log \epsilon$): 277 (3.96); IR ν_{\max} (KBr) cm^{-1} : 3355, 2941, 1750, 1428, 1346, 1236, 1133, 1049, 1010; ^1H NMR (CD₃OD, 500 MHz) and ^{13}C NMR (CD₃OD, 125 MHz) data Figs. 8–13. HRESI-TOF-MS data see Fig. 14.

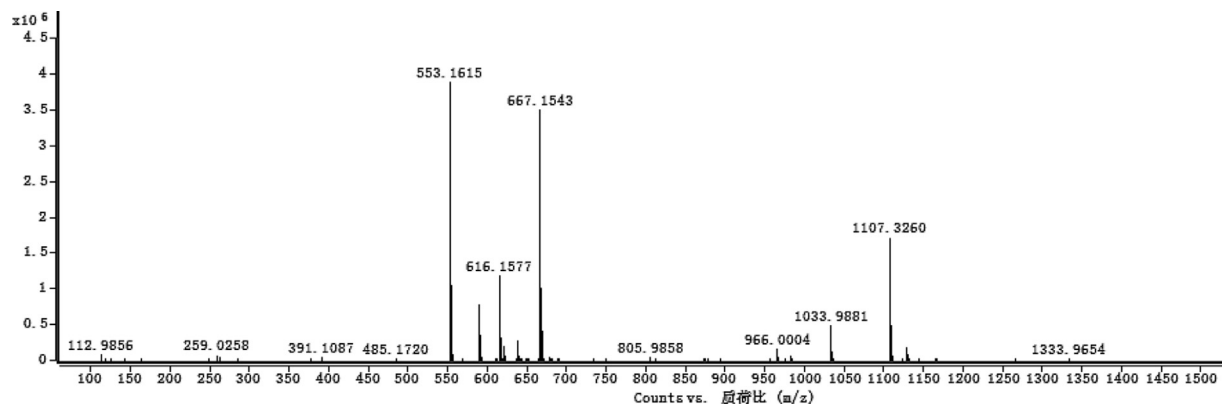


Fig. 42. HRESI-TOF-MS spectrum of compound 6.

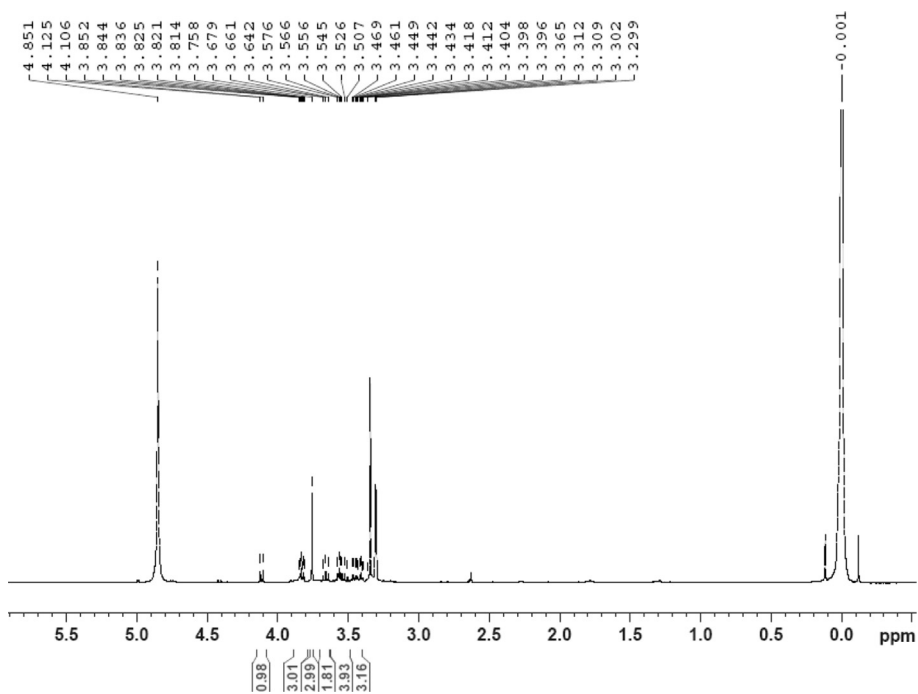


Fig. 43. ¹H NMR (500 MHz, CD₃OD) spectrum of compound 7.

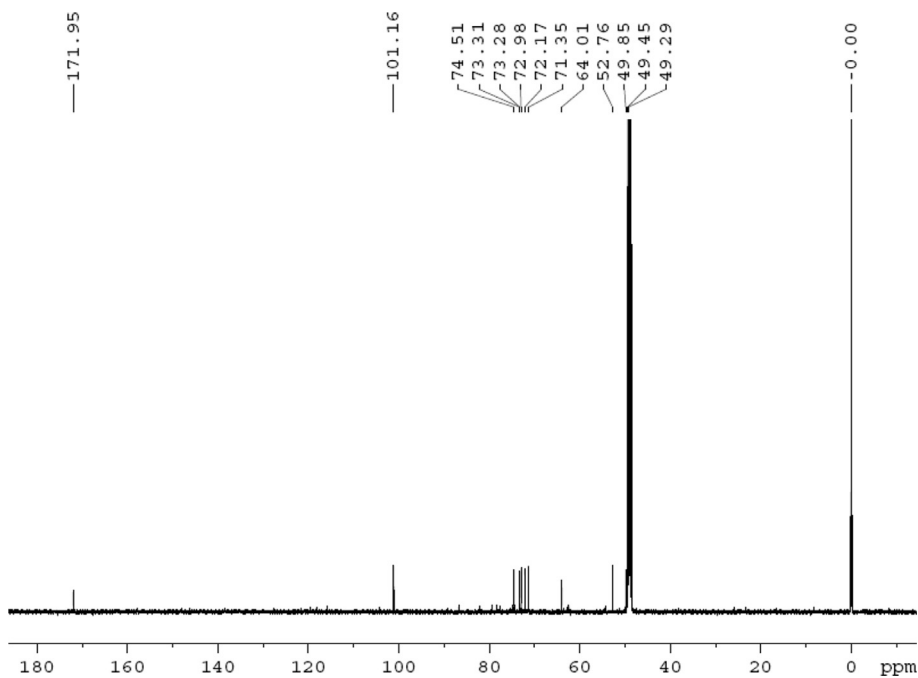


Fig. 44. ¹³C NMR (125 MHz, CD₃OD) spectrum of compound 7.

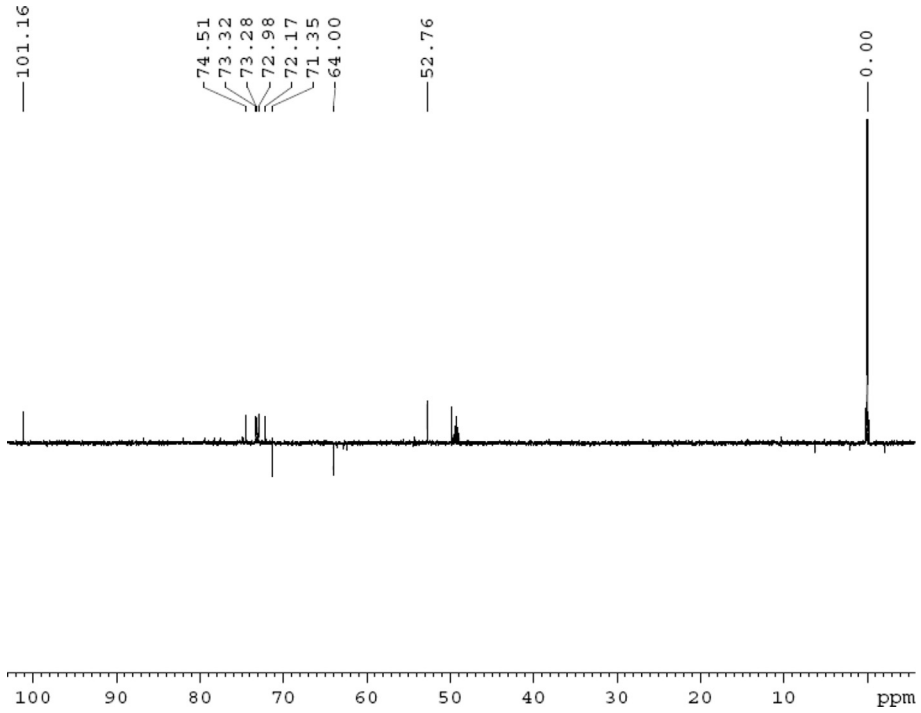


Fig. 45. DEPT 135 (CD₃OD) spectrum of compound 7.

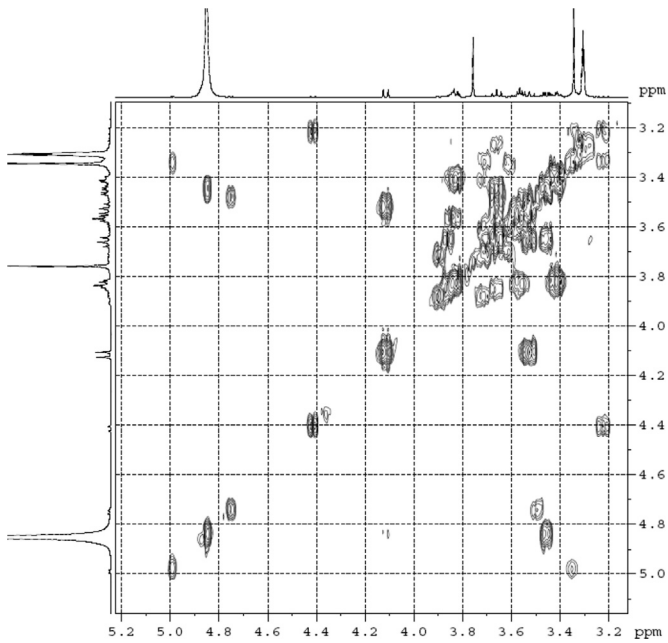


Fig. 46. ¹H-¹H COSY (CD₃OD) spectrum of compound 7.

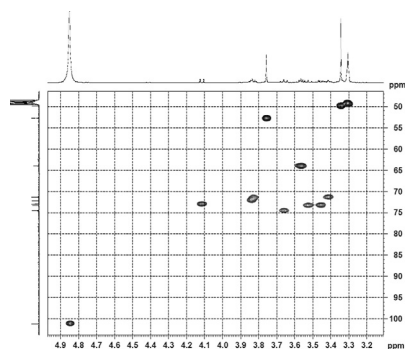


Fig. 47. HSQC (CD₃OD) spectrum of compound 7.

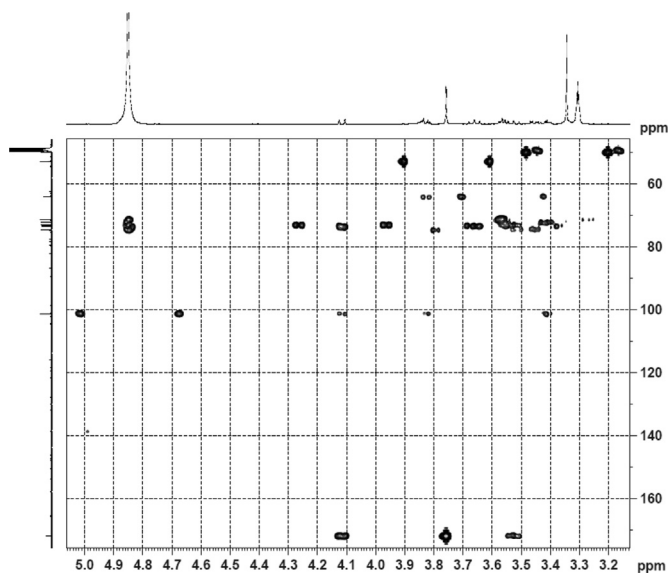


Fig. 48. HMBC (CD₃OD) spectrum of compound 7.

2.3.3. Pugcornol C (**3**)

Colorless oil; $[\alpha]_D^{25}$ -36.1 (conc. 0.14, MeOH); CD (conc. 0.0015 M, MeOH) mdeg (λ_{nm}): -0.34 (228), -2.60 (253), -2.99 (269); CD (conc. 0.0015 M, CH₃CN) mdeg (λ_{nm}): -1.26 (231), -3.91 (250), -5.16 (267); UV λ_{max} (MeOH) nm (log ϵ): 249 (3.50), 268 (3.58); IR ν_{max} (KBr) cm^{-1} : 3365, 2935, 1749, 1419, 1347, 1236, 1133, 1050, 1015; ¹H NMR (CD₃OD, 500 MHz) and ¹³C NMR (CD₃OD, 125 MHz) data Figs. 14–20. HRESI-TOF-MS data see Fig. 21.

2.3.4. Pugcornol D (**4**)

Colorless oil; $[\alpha]_D^{25}$ -64.1 (conc. 0.13, MeOH); CD (conc. 0.0015 M, MeOH) mdeg (λ_{nm}): -35.54 (214), $+14.06$ (238), $+0.12$ (263); CD (conc. 0.0015 M, CH₃CN) mdeg (λ_{nm}): -33.13 (212), 12.47 (238), $+0.23$ (262); UV λ_{max} (MeOH) nm (log ϵ): 214 (3.44), 267 (2.38); IR ν_{max} (KBr) cm^{-1} : 3420, 2943, 1765, 1701,

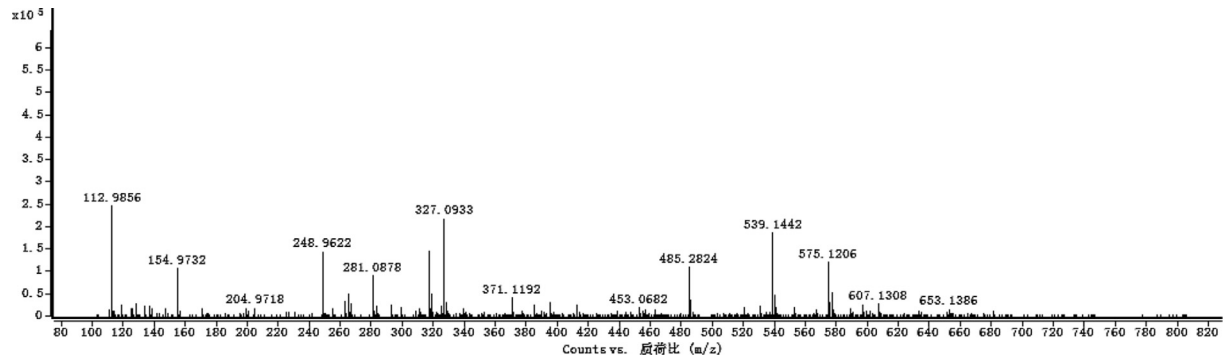


Fig. 49. HRESI-TOF-MS spectrum of compound 7.

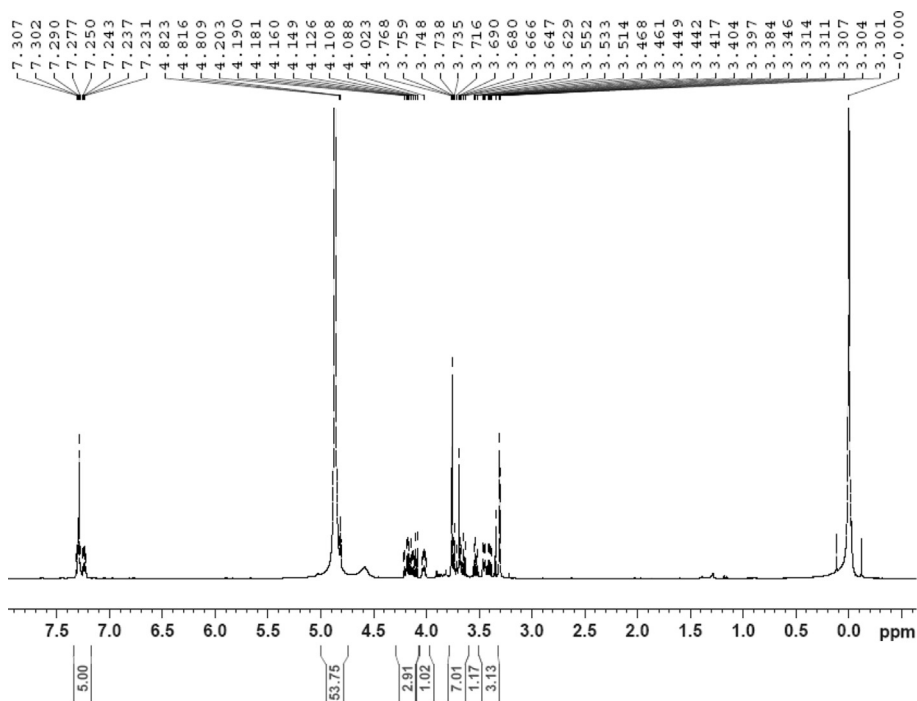


Fig. 50. ^1H NMR (500 MHz, CD_3OD) spectrum of compound 8.

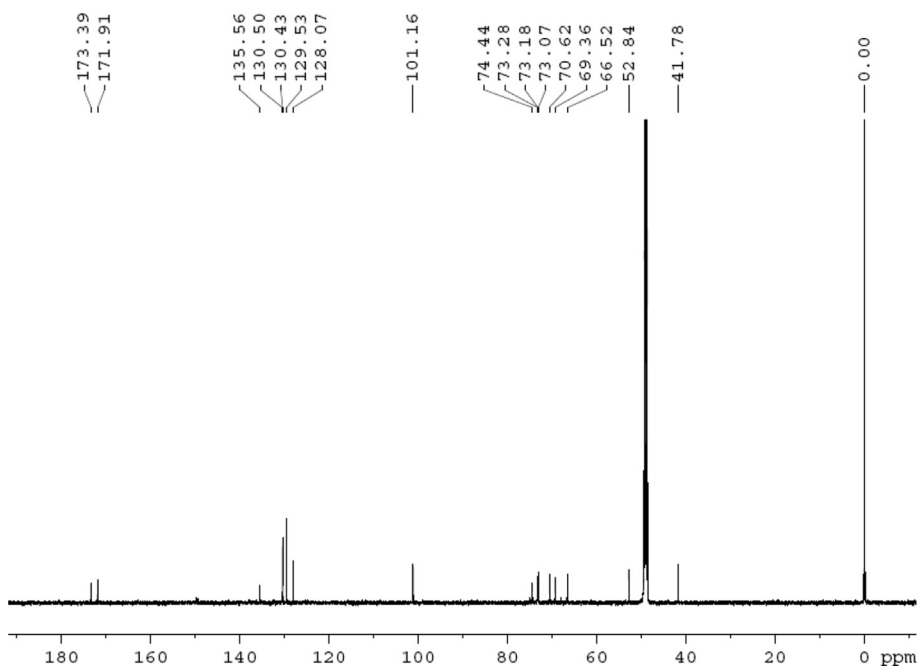


Fig. 51. ^{13}C NMR (125 MHz, CD_3OD) spectrum of compound 8.

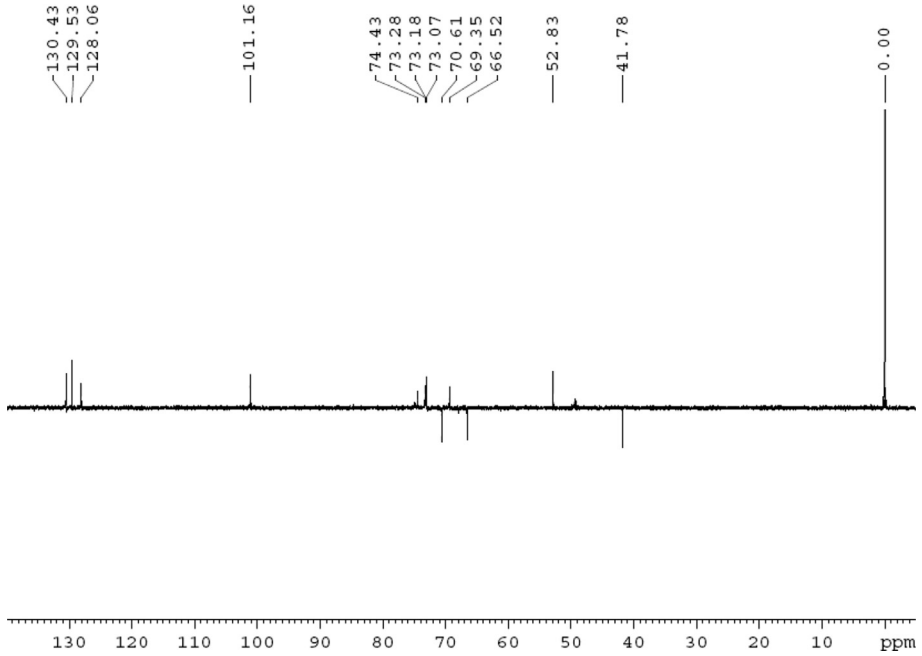


Fig. 52. DEPT 135 (CD₃OD) spectrum of compound 8.

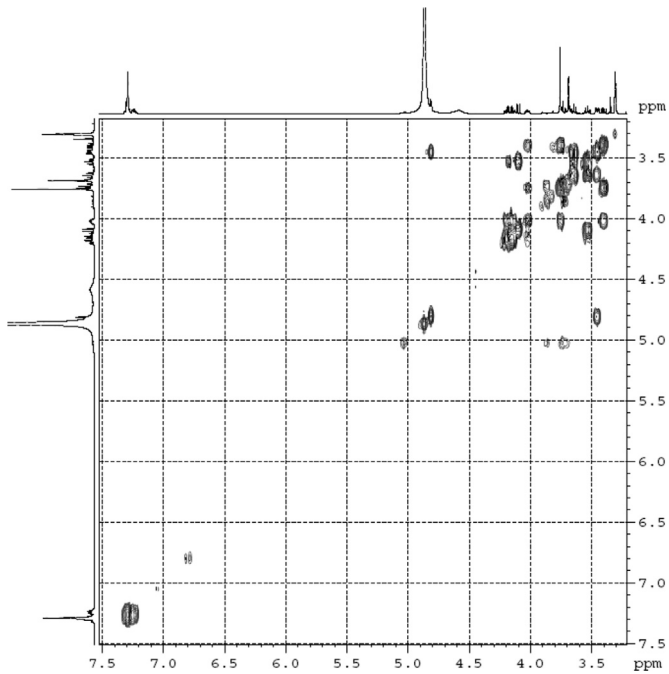


Fig. 53. ¹H-¹H COSY (CD₃OD) spectrum of compound 8.

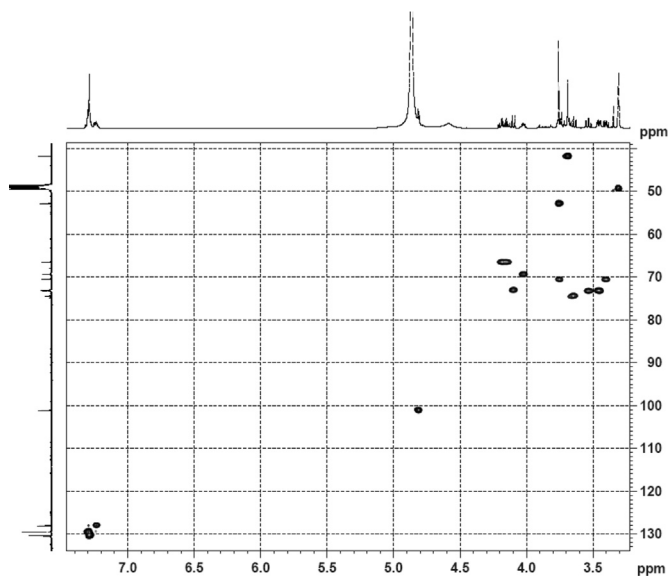


Fig. 54. HSQC (CD₃OD) spectrum of compound 8.

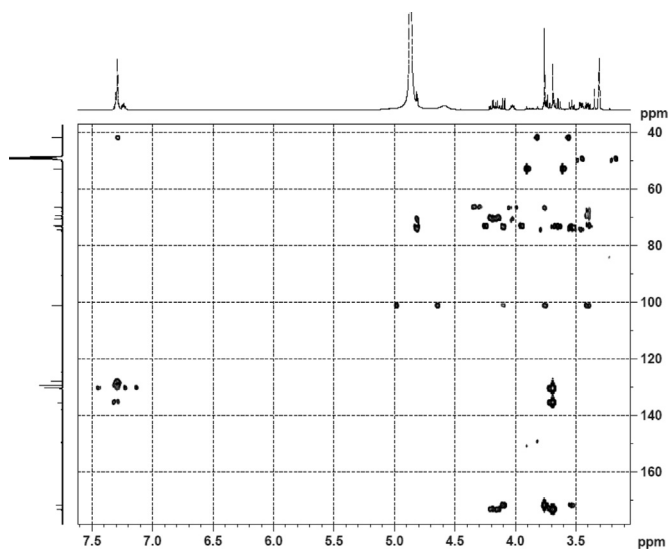


Fig. 55. HMBC (CD₃OD) spectrum of compound 8.

1447, 1419, 1361, 1014; ¹H NMR (CD₃OD, 500 MHz) and ¹³C NMR (CD₃OD, 125 MHz) data Figs. 22–27. HRESI-TOF-MS data see Fig. 28.

2.3.5. Pugcornoside A (5)

White powder; [α]_D²⁵ –23.2 (conc. 0.11, MeOH); UV λ_{\max} (MeOH) nm (log ϵ): 223 (3.61), 277 (3.18); IR ν_{\max} (KBr) cm⁻¹: 3365, 2922, 1646, 1558, 1507, 1457, 1277, 1070; ¹H NMR (CD₃OD, 500 MHz) and ¹³C NMR (CD₃OD, 125 MHz) data Figs. 29–34. HRESI-TOF-MS data see Fig. 35.

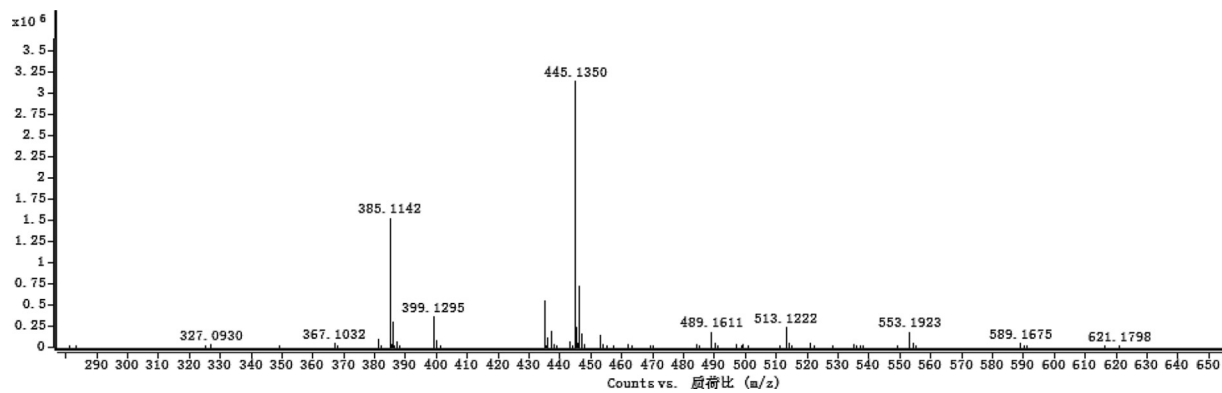


Fig. 56. HRESI-TOF-MS spectrum of compound **8**.

2.3.6. *Pugcornoside B (6)*

White powder; $[\alpha]_D^{25} -52.6$ (conc. 0.13, MeOH); UV λ_{\max} (MeOH) nm (log ϵ): 220 (3.65), 277 (3.18); IR ν_{\max} (KBr) cm^{-1} : 3366, 2918, 1600, 1507, 1456, 1436, 1278, 1045, 1072; ^1H NMR (CD_3OD , 500 MHz) and ^{13}C NMR (CD_3OD , 125 MHz) data Figs. 36–41. HRESI-TOF-MS data see Fig. 42.

2.3.7. *Pugcornoside C (7)*

White powder; $[\alpha]_D^{25} +6.3$ (conc. 0.12, MeOH); UV λ_{\max} (MeOH) nm (log ϵ): 266 (2.94); IR ν_{\max} (KBr) cm^{-1} : 3386, 2391, 1734, 1652, 1107, 1044; ^1H NMR (CD_3OD , 500 MHz) and ^{13}C NMR (CD_3OD , 125 MHz) data Figs. 43–48. HRESI-TOF-MS data see Fig. 49.

2.3.8. *Pugcornoside D (8)*

White powder; $[\alpha]_D^{25} +28.1$ (conc. 0.12, MeOH); UV λ_{\max} (MeOH) nm (log ϵ): 205 (3.72), 262 (2.77); IR ν_{\max} (KBr) cm^{-1} : 3420, 2933, 1733, 1635, 1466, 1339, 1267, 1154, 1112, 1047; ^1H NMR (CD_3OD , 500 MHz) and ^{13}C NMR (CD_3OD , 125 MHz) data Figs. 50–55. HRESI-TOF-MS data see Fig. 56.

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

- [1] W. Shi, J. Ruan, Y. Guo, Z. Ding, J. Yan, L. Qu, C. Zheng, Y. Zhang, T. Wang, Bioactive Constituents Study of *Pugionium Cornutum* L. Gaertn on Intestinal Motility, *Fitoerapia* (2019) (be accepted).
- [2] Q. Wang, R.J. Abbott, Q.S. Yu, K. Lin, J.Q. Liu, Pleistocene climate change and the origin of two desert plant species, *Pugionium cornutum* and *Pugionium dolabratum* (Brassicaceae), in northwest China, *New Phytol.* 199 (2013) 277–287.
- [3] H. Li, C. Li, C. Zhang, B. Chen, L. Hui, Y. Shen, Compositional and gastrointestinal prokinetic studies of *Pugionium* (L.), *Food Chem.* 186 (2015) 285–291.
- [4] X.C. Li, D. Ferreira, Y. Ding, Determination of absolute configuration of natural products: theoretical calculation of electronic circular dichroism as a tool, *Curr. Org. Chem.* 14 (2010) 1678–1697.
- [5] G. Mazzeo, E. Santoro, A. Andolfi, A. Cimmino, P. Troselj, A.G. Petrovic, S. Superchi, A. Evidente, N. Berova, Absolute configurations of fungal and plant metabolites by chiroptical methods. ORD, ECD, and VCD studies on phyllostin, scytolide, and oxysporone, *J. Nat. Prod.* 76 (2013) 588–599.
- [6] J. Xu, S. Li, X. Su, J. Ma, F. Liu, L. Tong, D. Lee, Y. Ohizumi, M. Tuerhong, Y. Guo, Diterpenoids from *Callicarpa kwangtungensis* and their NO inhibitory effects, *Fitoterapia* 113 (2016) 151–157.
- [7] Y. Zhang, Y. Guo, X. Li, J. Ruan, T. Wang, J. Li, L. Han, H. Yu, T. Wang, Rearranged oleanane type saponins, astraisoolesaponins A₁–A₃ and B, from the stems of *Astragalus membranaceus* (Fisch.) Bge. var. *mongholicus* (Bge.) Hsiao, *Tetrahedron* 72 (2016) 7008–7013.
- [8] C.F. Shaw, J. Eldridge, M.P. Cancro, ^{13}C NMR studies of aurothioglucoase: ligand exchange and redox disproportionation reactions, *J. Inorg. Biochem.* 14 (1981) 267–274.
- [9] A.N. Ngane, M. Lavault, D. Séraphin, A. Landreau, P. Richomme, Three 1-thio-beta-D-glucopyranosides from the seeds of *Afrotyrax lepidophyllus* mildbr, *Carbohydr. Res.* 341 (2006) 2799–2802.
- [10] X.X. Huang, S. Liu, L.L. Lou, Q.B. Liu, C.C. Zhou, L.Z. Li, Y. Peng, S.J. Song, Phenylpropanoids from *Crataegus pinnatifida* and their chemotaxonomic importance, *Biochem. Syst. Ecol.* 54 (2014) 208–212.
- [11] Y. Zhang, X. Li, J. Ruan, Y. Dong, J. Hao, E. Liu, L. Han, X. Gao, T. Wang, Oleanane type saponins from the stem of *Astragalus membranaceus* (Fisch.) Bge. var. *mongholicus* (Bge.) Hisao, *Fitoterapia* 109 (2016) 99–105.