



Data Article

Electrospray ionization mass spectrometry and nuclear magnetic resonance spectroscopy data for anacardic acid derivatives

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ABSTRACT

The data presented here are related to the research paper entitled "Janohigenins: Long-chain anacardic acid derivatives with neuroprotective activity from *Ophiopogon japonicus* seeds" (Ohta et al., 2021). In this data article, we provide electrospray ionization mass spectrometry (ESIMS) and 1D and 2D nuclear magnetic resonance (NMR) spectroscopy data of four new anacardic acid derivatives, janohigenins isolated from the seeds of *Ophiopogon japonicus*.

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Specifications Table

Subject	chemistry
Specific subject area	natural products
Type of data	Figure
How the data were acquired	ESIMS: Thermo Fisher Scientific LTQ Orbitrap XL mass spectrometer; NMR spectroscopy: JEOL Lambda500.
Data format	Raw Analyzed
Parameters for data collection	HPLC-PDA: JASCO LC-2000 instrument equipped with a JASCO MD-2015 multiwavelength detector, mobile phase CH ₃ CN containing 0.1% trifluoroacetic acid, flow rate 0.6 mL/min. MS: Thermo Fisher Scientific LTQ Orbitrap XL mass spectrometer, Source type ESI, MS scan range <i>m/z</i> 50–2000, resolution 60,000. MS/MS: auto mode, scan range <i>m/z</i> 50–2000. NMR: 500 MHz JEOL Lambda500 spectrometer, operating temp 25 °C.
Description of data collection	The new anacardic acid derivatives were purified by column chromatography and HPLC. The purified compounds were analyzed by ESIMS and NMR spectroscopy.
Data source location	Hiroshima University, Higashi-Hiroshima, Japan
Data accessibility	Data are available within this article and as a supplementary file
Related research article	S. Ohta, M. Takeda, E. Ohta, T. Nehira, H. Ômura, M. M. Uy, Y. Ishihara, Janohigenins: Long-chain anacardic acid derivatives with neuroprotective activity from <i>Ophiopogon japonicus</i> seeds, <i>Phytochemistry</i> , 191 (2021) 112904. https://doi.org/10.1016/j.phytochem.2021.112904

Value of the Data

- The data presents ESIMS and NMR spectroscopic data of anacardic acid derivatives having rare side chains with lengths of C₂₁–C₂₅ and can be used by other researchers.
- Information about the spectroscopic data provided for anacardic acid derivatives can be useful for spectral analysis and structural elucidation of other anacardic acid derivatives.
- This data could be a benchmark for other researchers in elucidating the structures of anacardic acid derivatives.

1. Data Description

The present data set focuses on characterization of the anacardic acid derivatives in our reference [1]. The article contains information on the electrospray ionization mass spectrometry (ESIMS) and 1D and 2D nuclear magnetic resonance (NMR) spectroscopic data of the anacardic acid derivatives, janohigenins **1–4** isolated from the seed of *Ophiopogon japonicus* (Fig. 1). The HPLC-photodiode array (PDA) profiles of a mixture of janohigenins **1–4** obtained from the EtOAc-soluble fraction are given in Fig. 2. The negative ion mode high resolution (HR) ESIMS data of **1–4** are shown in Figs. 3a, 4a, 5a, and 6a, respectively. The product ion spectra (ESI-MS/MS) of **1–4** are shown in Figs. 3b, 4b, 5b, and 6b, respectively. The ¹H NMR spectra of **1–4** are shown in Figs. 3c, 4c, 5c, and 6c, respectively. The ¹H–¹H COSY spectra of **1–4** are shown in Figs. 3d, 4d, 5d, and 6d, respectively. The ¹H–¹³C heteronuclear multiple quantum correlation (HMQC) spectra of **1–4** are shown in Figs. 3e, 4e, 5e, and 6e, respectively. The ¹H–¹³C heteronuclear multiple-bond correlation (HMBC) spectra of **1–4** are shown in Figs. 3f, 4f, 5f, and 6f, respectively. Analyses of the ESIMS and NMR spectra of **1–4** are described in the reference [1]. It has been reported that **3** and **4** showed neuroprotective activity against rotenone-induced cellular damage in SH-SY5Y human neuroblastoma cells [1].

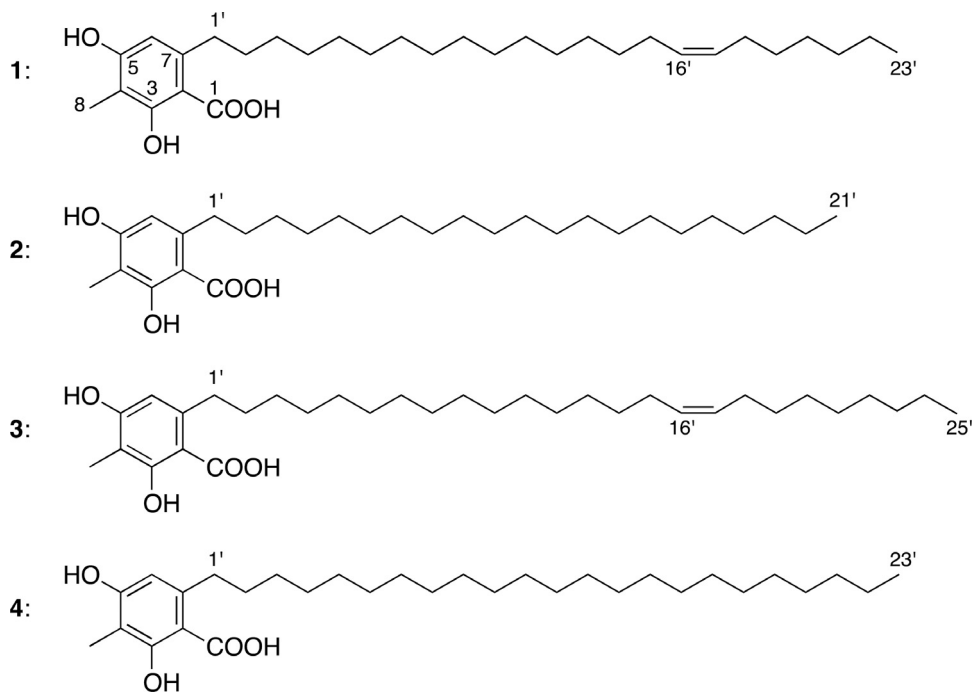


Fig. 1. Structures of janohigenins isolated from the seed of *Ophiopogon japonicus* modified from the Fig. 1 of the related research article [1].

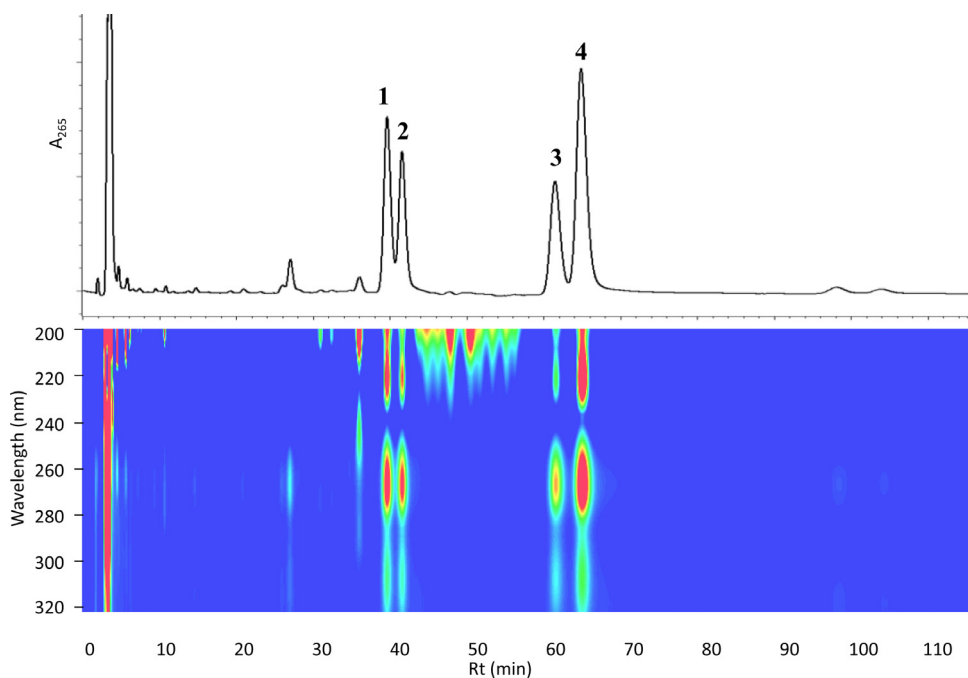


Fig. 2. HPLC-PDA profiles of a mixture of janohigenins modified from the Fig. 3 of the related research article [1].

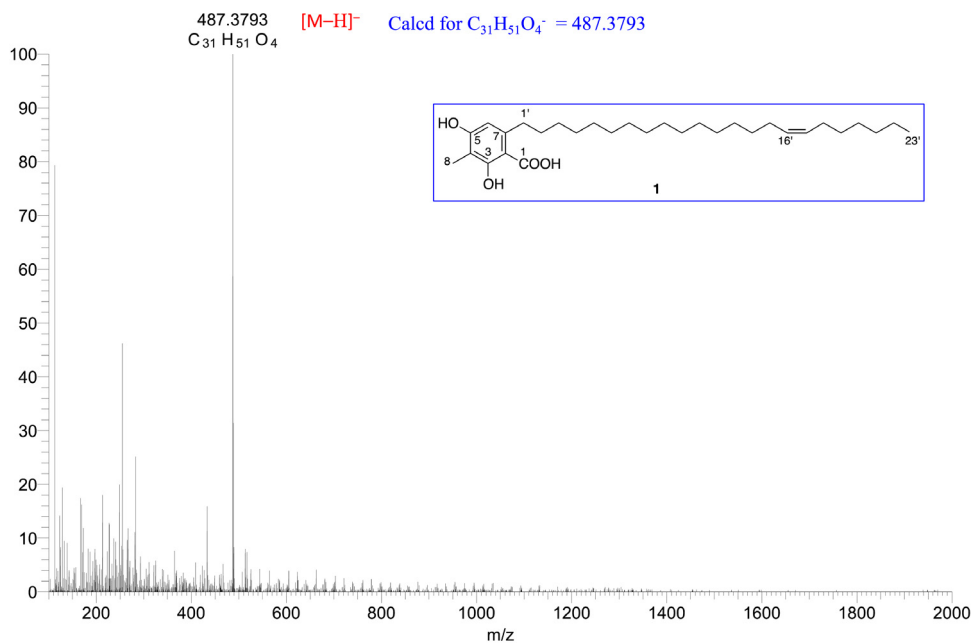


Fig. 3a. Negative ion mode HRESIMS of janhigenin 23-1 (1).

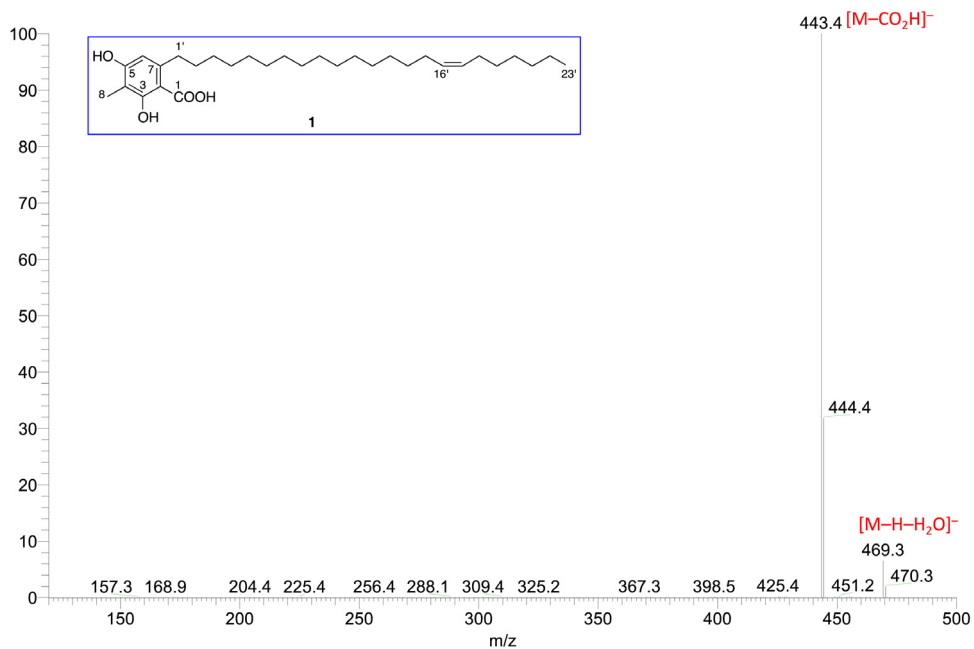


Fig. 3b. Product ion spectrum (ESI-MS/MS: precursor ion: m/z 487 [M-H]⁻) of janhigenin 23-1 (1).

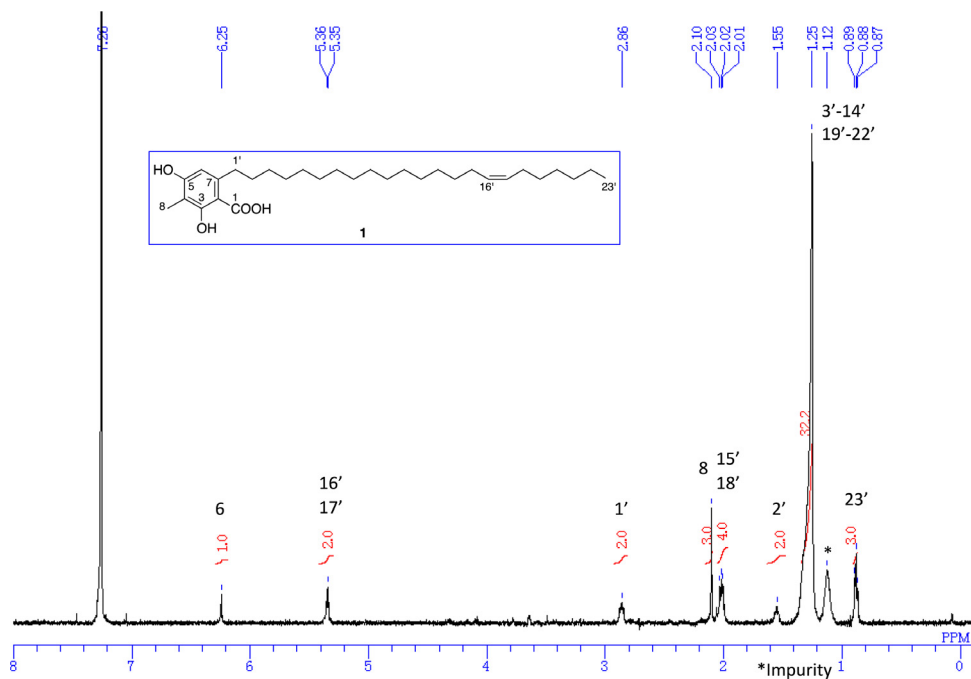


Fig. 3c. ^1H NMR spectrum of janothigenin 23-1 (1) (500 MHz, CDCl_3).

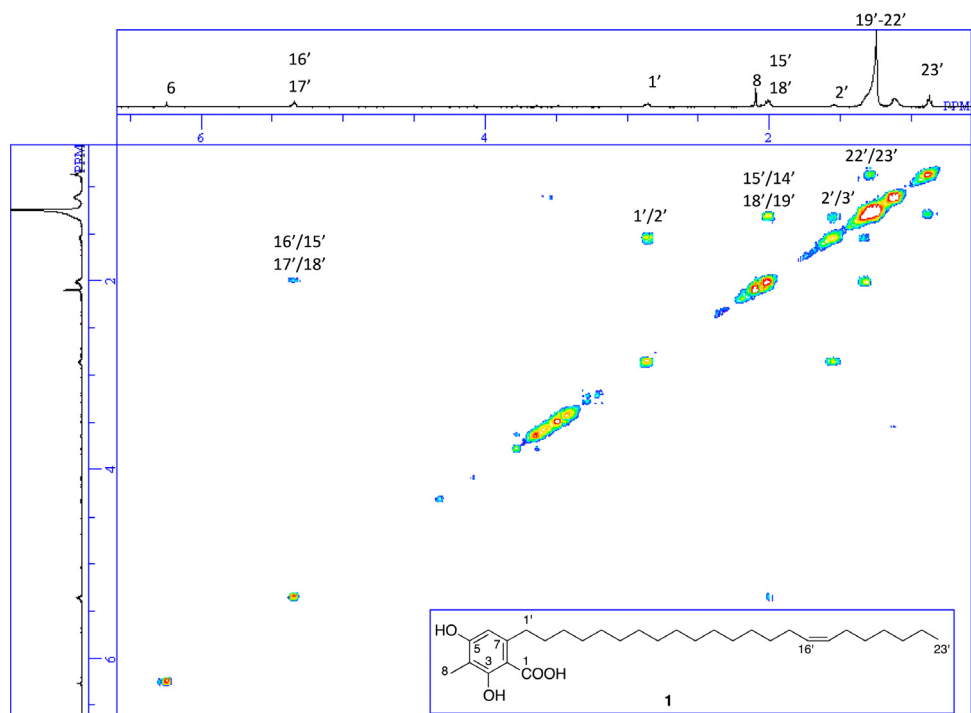


Fig. 3d. ^1H - ^1H COSY spectrum of janothigenin 23-1 (1).

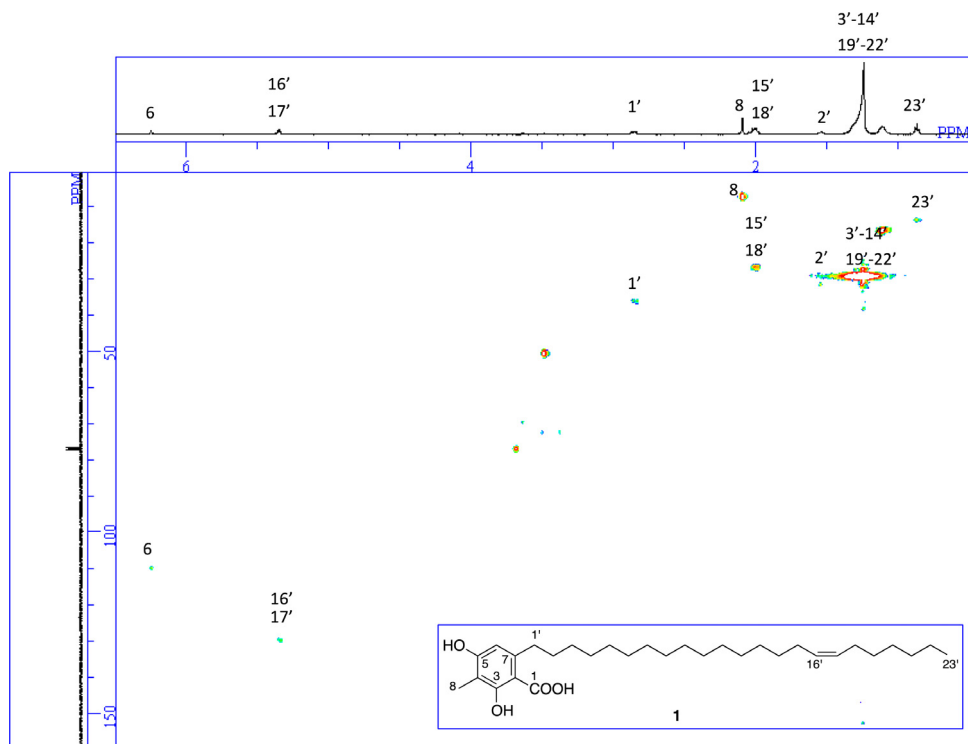


Fig. 3e. ^1H - ^{13}C HMQC spectrum of janhigenin 23-1 (**1**).

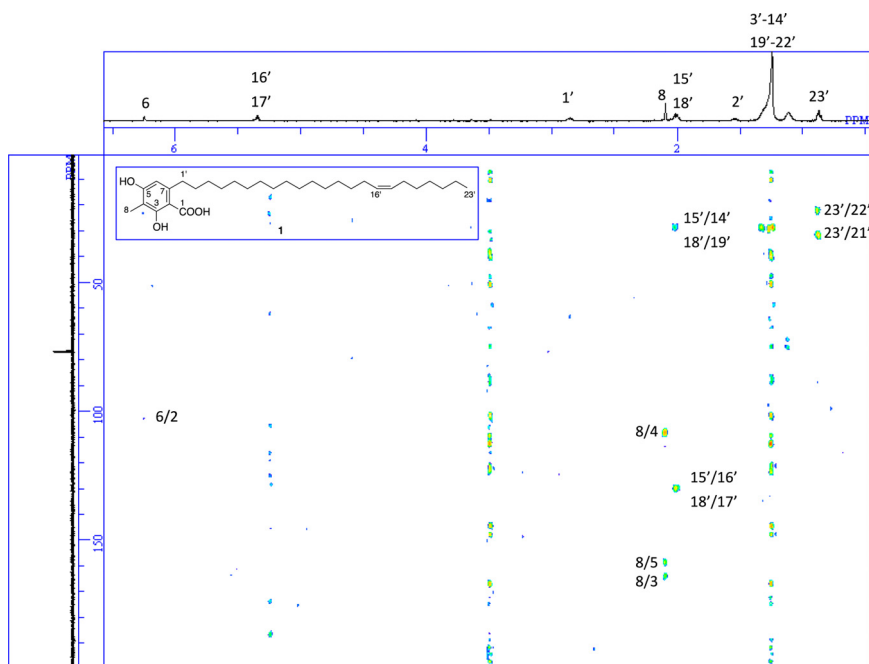


Fig. 3f. ^1H - ^{13}C HMBC spectrum of janhigenin 23-1 (**1**).

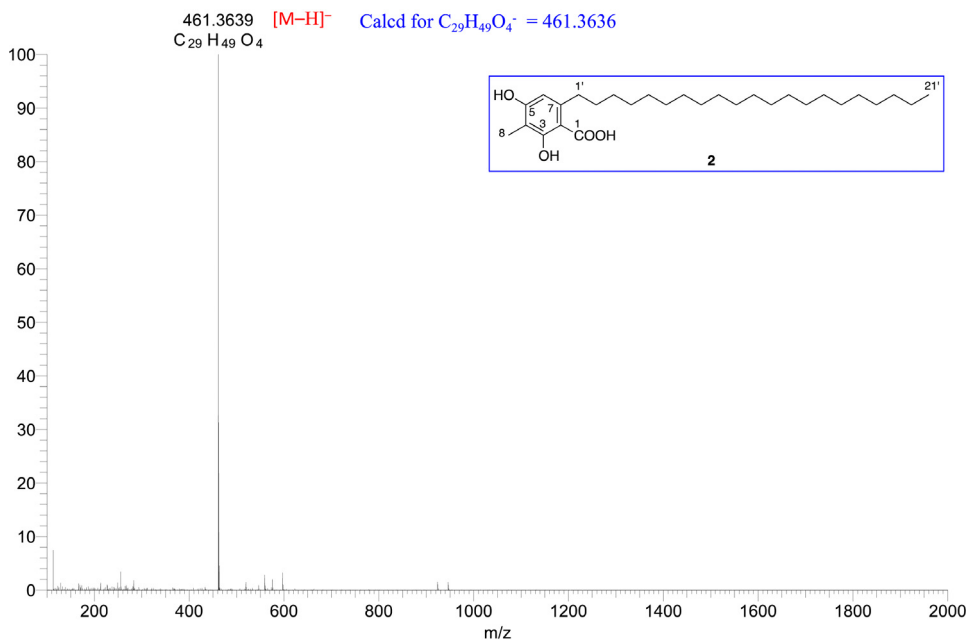


Fig. 4a. Negative ion mode HRESIMS of janohigenin 21-0 (2).

Although many anacardic acid derivatives have been isolated from the plants belonging to Anacardiaceae [2,3], Ginkgoaceae [4], and Fabaceae [5], anacardic acid derivatives possessing side-chains with lengths of C₂₁–C₂₅ are rare among them [1].

Janohigenins 1–4

(Z)-2,4-Dihydroxy-3-methyl-6-(tricos-16-en-1-yl)benzoic acid (janohigenin 23-1) (1)

HRESIMS, ESI-MS/MS, 1D and 2D NMR spectra of compound 1 are provided in Fig. 3a–f. 6-Henicosyl-2,4-dihydroxy-3-methylbenzoic acid (janohigenin 21-0) (2)

HRESIMS, ESI-MS/MS, 1D and 2D NMR spectra of compound 2 are provided in Fig. 4a–f.

(Z)-2,4-Dihydroxy-3-methyl-6-(pentacos-16-en-1-yl)benzoic acid (janohigenin 25-1) (3)

HRESIMS, ESI-MS/MS, 1D and 2D NMR spectra of compound 3 are provided in Fig. 5a–f. 2,4-Dihydroxy-3-methyl-6-tricosylbenzoic acid (janohigenin 23-0) (4)

HRESIMS, ESI-MS/MS, 1D and 2D NMR spectra of compound 4 are provided in Fig. 6a–f. Supplementary material includes the raw data for ESIMS and NMR spectra of 1–4.

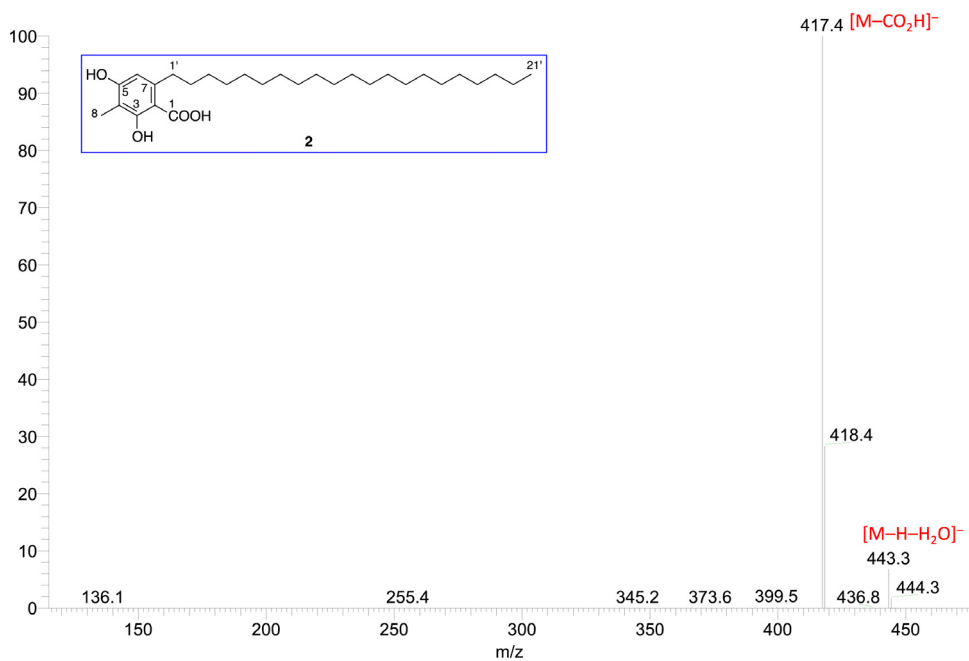


Fig. 4b. Product ion spectrum (ESI-MS/MS; precursor ion: m/z 461 $[M-H]^-$) of janhigenin 21-0 (**2**).

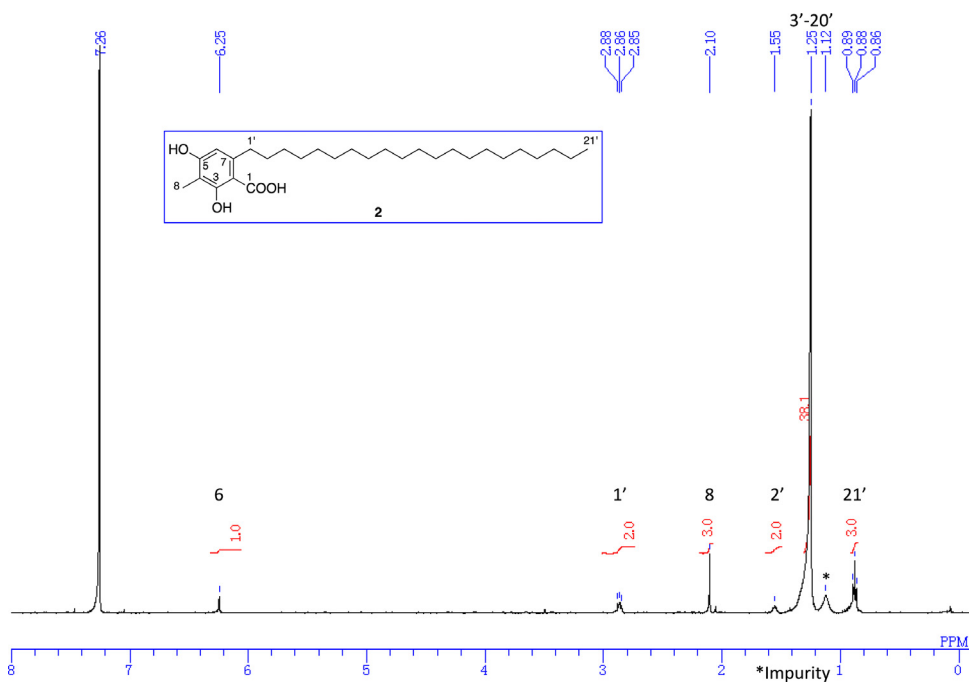


Fig. 4c. ^1H NMR spectrum of janhigenin 21-0 (**2**) (500 MHz, CDCl_3).

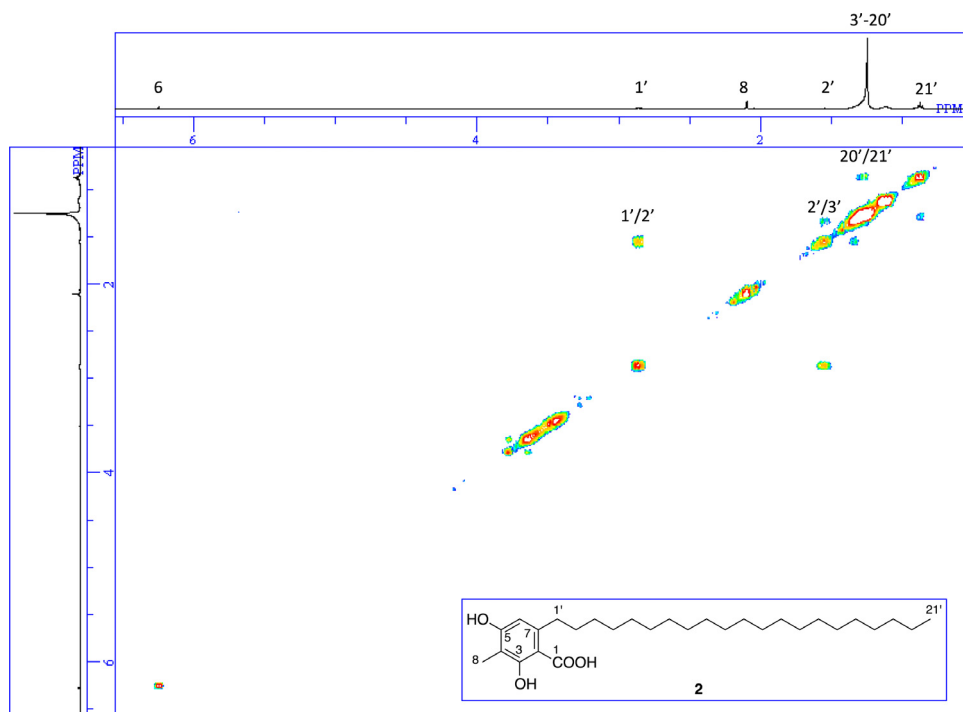


Fig. 4d. ^1H - ^1H COSY spectrum of janohigenin 21-0 (2).

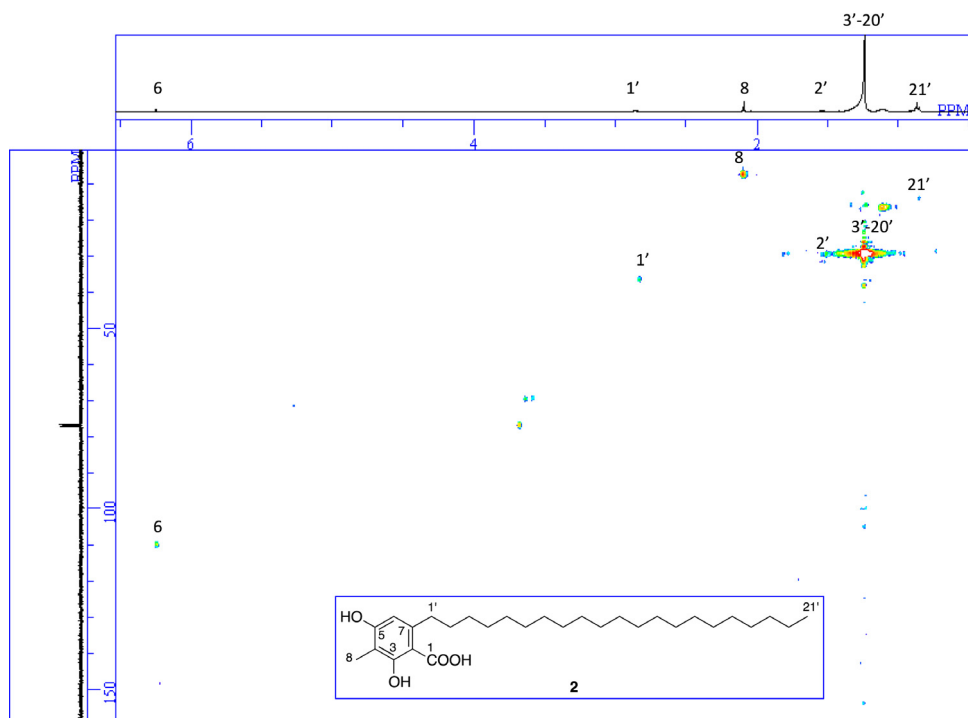


Fig. 4e. ^1H - ^{13}C HMQC spectrum of janohigenin 21-0 (2).

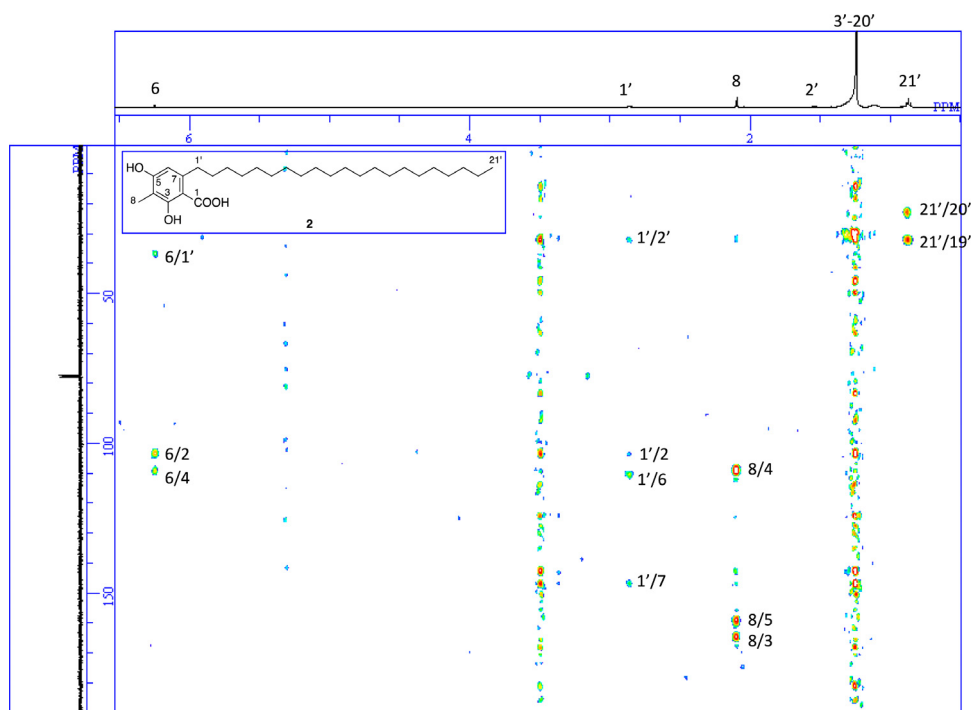


Fig. 4f. ^1H - ^{13}C HMBC spectrum of janohigenin 21-0 (2).

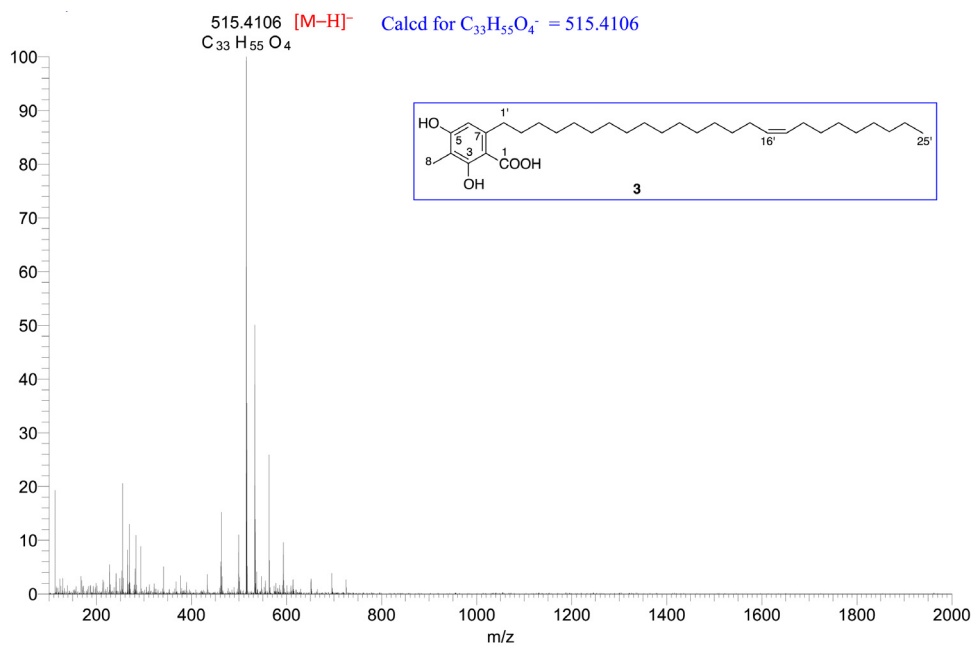


Fig. 5a. Negative ion mode HRESIMS of janohigenin 25-1 (3).

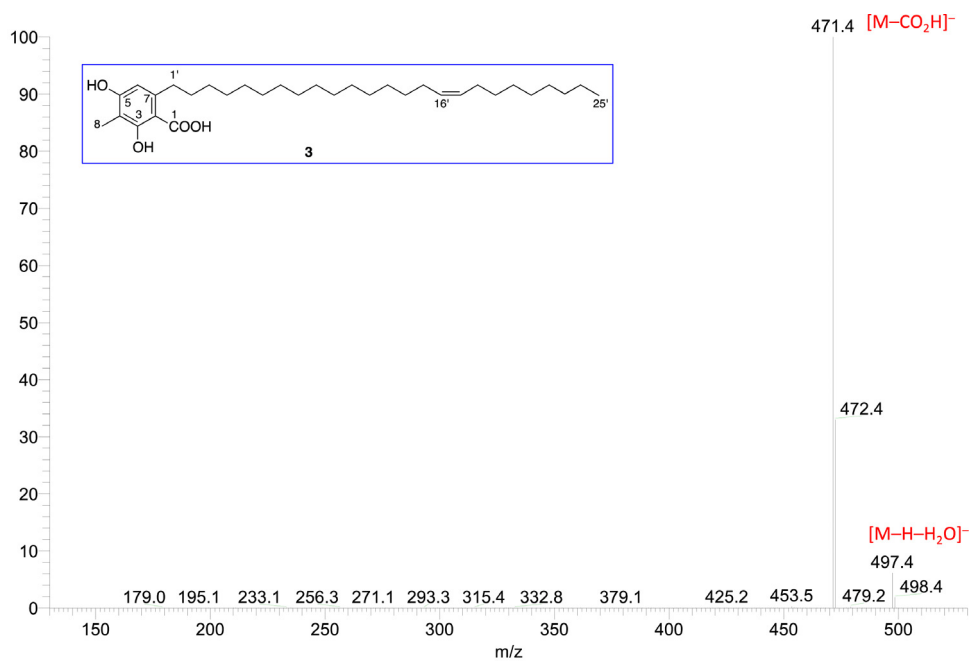


Fig. 5b. Product ion spectrum (ESI-MS/MS: precursor ion: m/z 515 $[M-H]^-$) of janhigenin 25-1 (**3**).

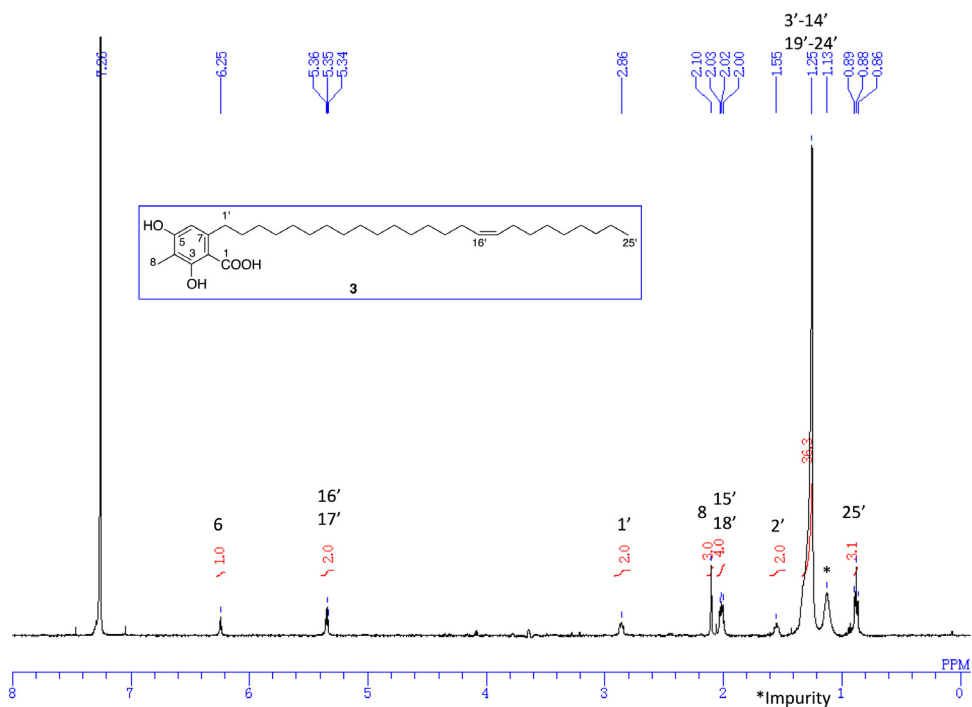


Fig. 5c. 1H NMR spectrum of janhigenin 25-1 (**3**) (500 MHz, $CDCl_3$).

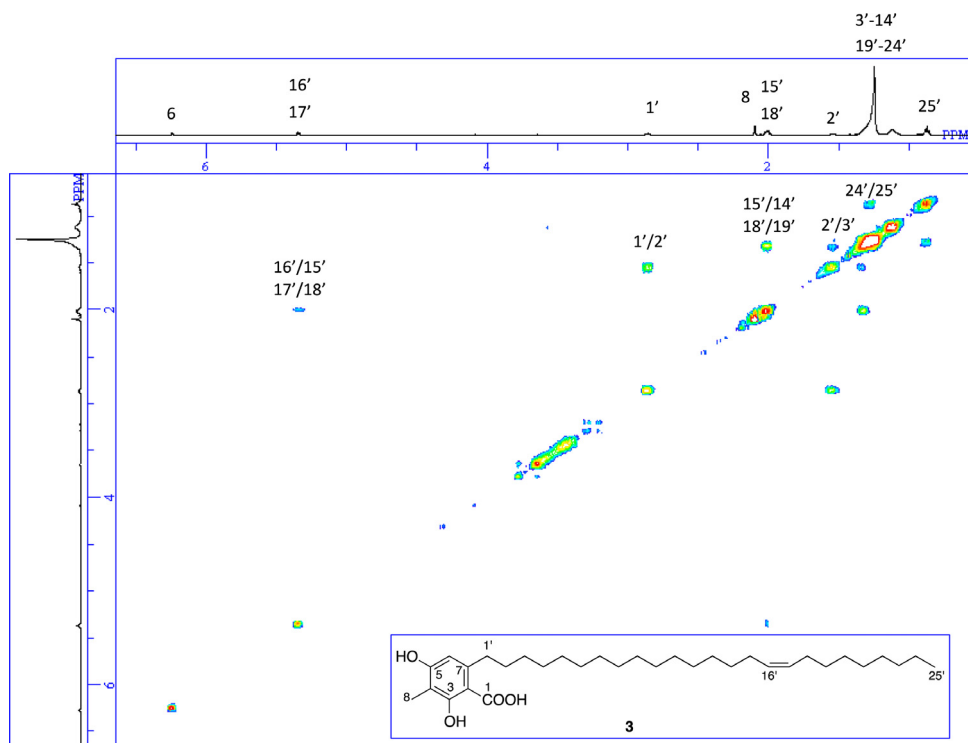


Fig. 5d. ^1H - ^1H COSY spectrum of janohigenin 25-1 (**3**).

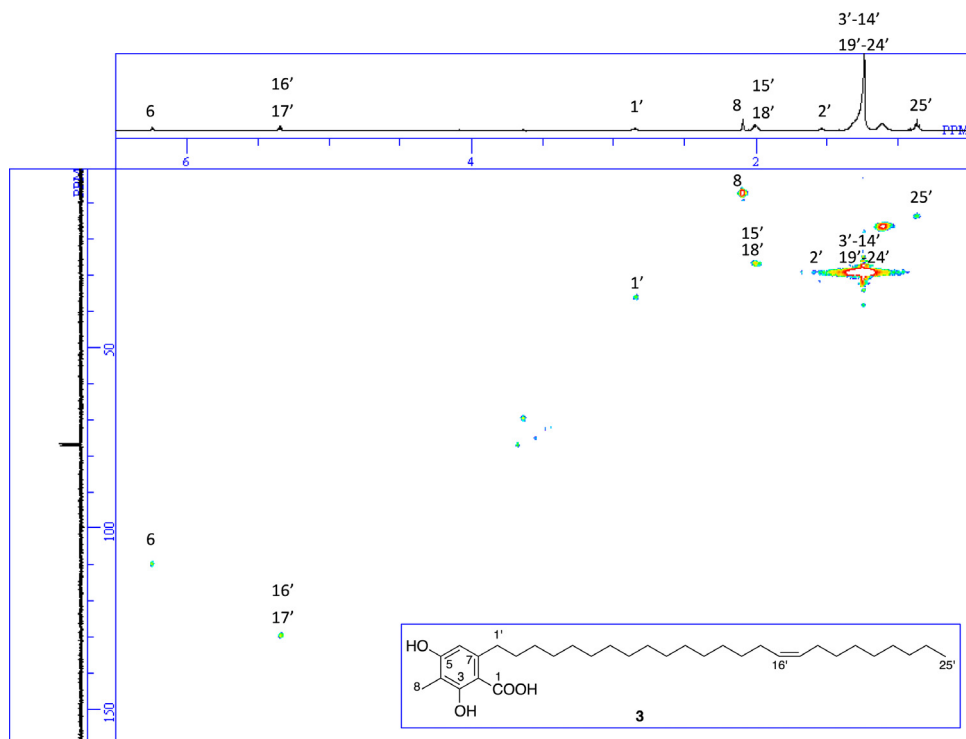


Fig. 5e. ^1H - ^{13}C HMQC spectrum of janohigenin 25-1 (3).

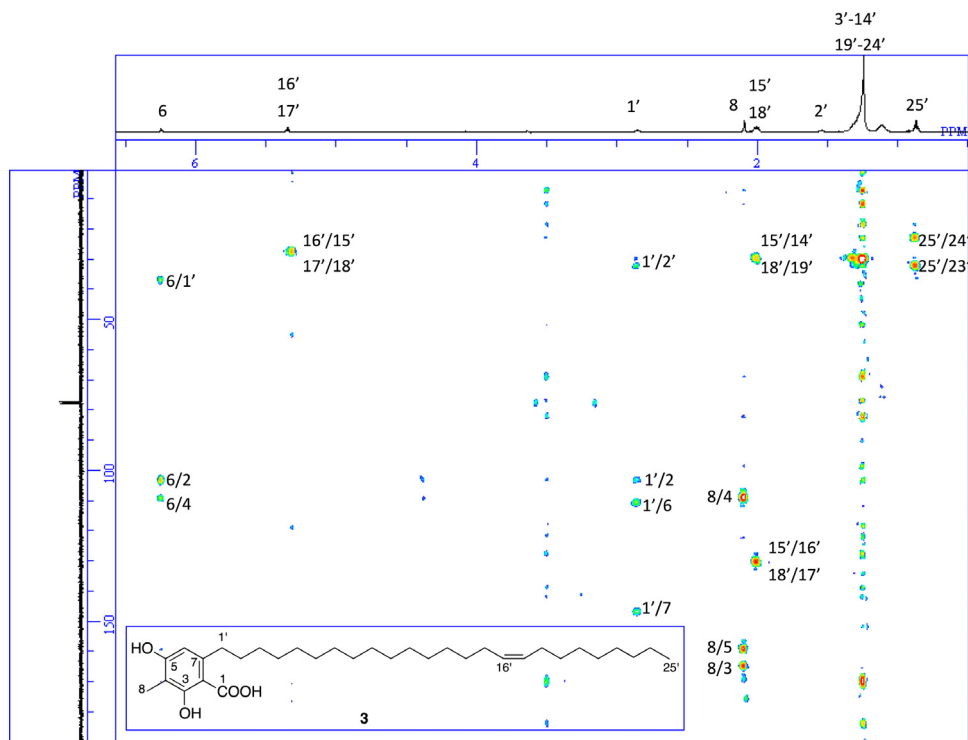


Fig. 5f. ^1H - ^{13}C HMBC spectrum of janothigenin 25-1 (3).

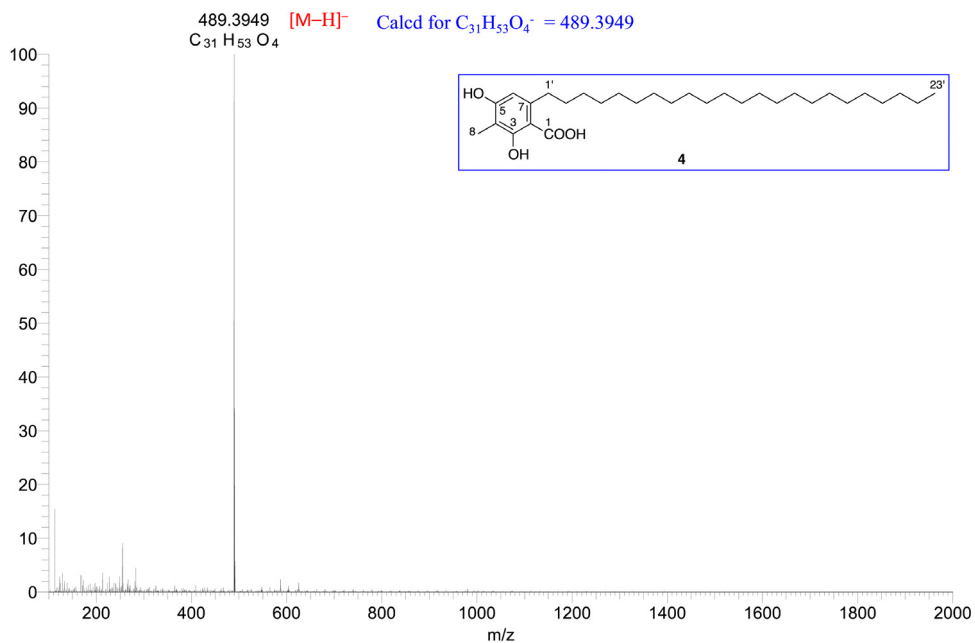


Fig. 6a. Negative ion mode HRESIMS of janothigenin 23-0 (4).

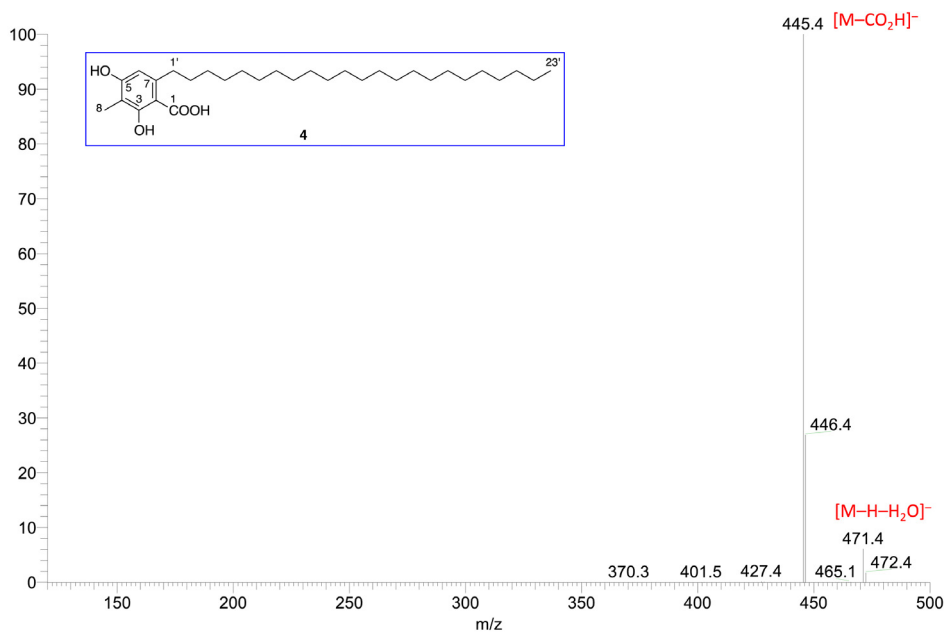


Fig. 6b. Product ion spectrum (ESI-MS/MS: precursor ion: m/z 489 $[M-H]^-$) of janhigenin 23-0 (**4**).

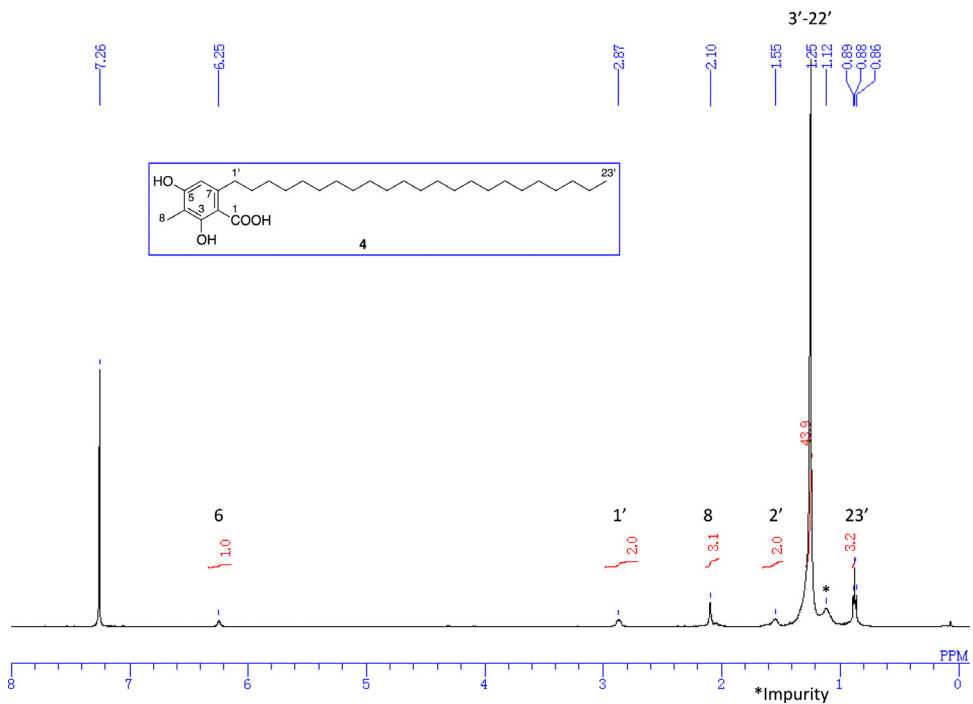


Fig. 6c. 1H NMR spectrum of janhigenin 23-0 (**4**) (500 MHz, $CDCl_3$).

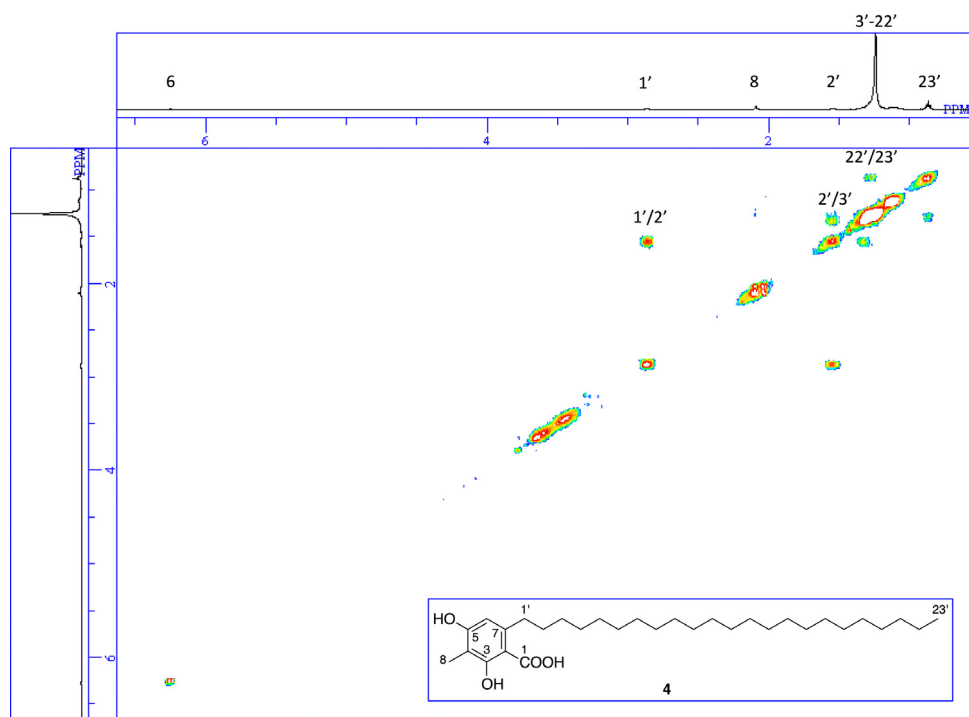


Fig. 6d. ^1H - ^1H COSY spectrum of janohigenin 23-0 (4).

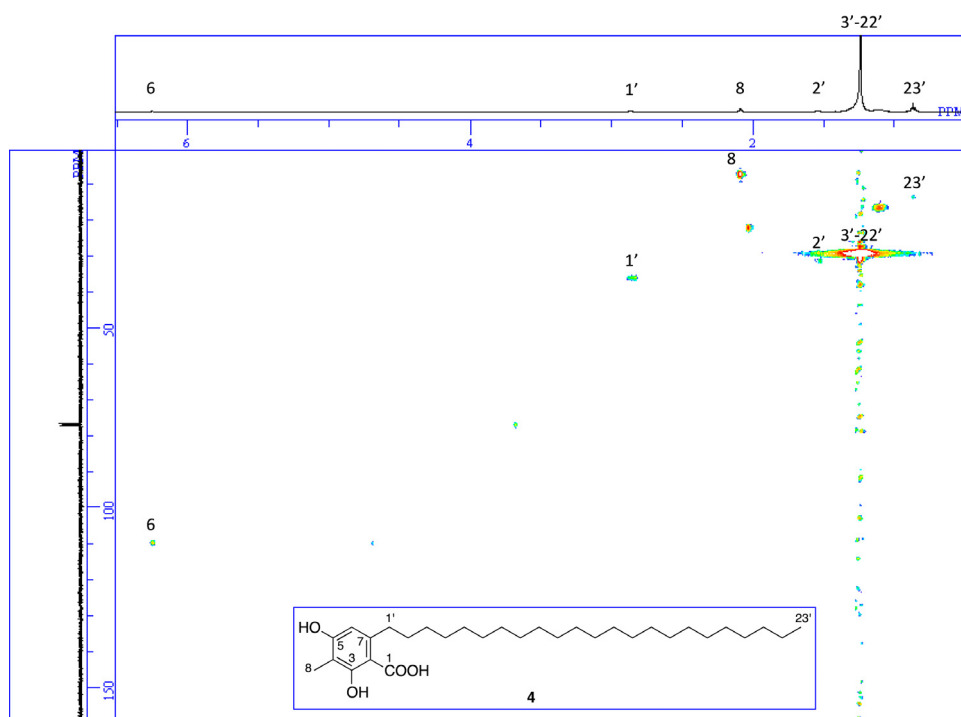


Fig. 6e. ^1H - ^{13}C HMQC spectrum of janhigenin 23-0 (4).

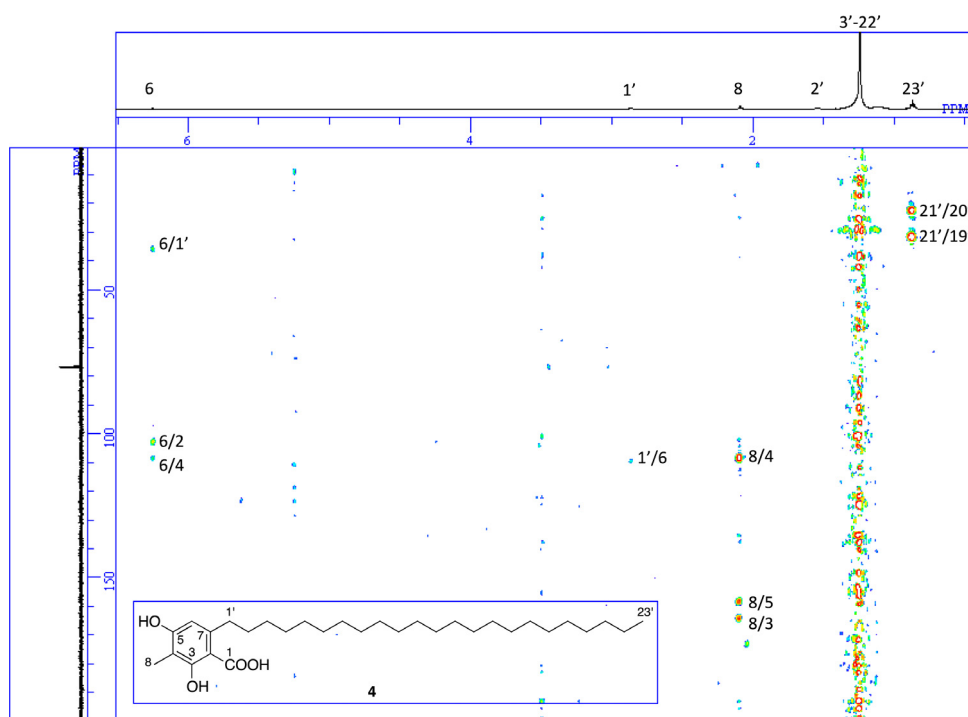


Fig. 6f. ^1H - ^{13}C HMBC spectrum of janohigenin 23-0 (4).

2. Experimental Design, Materials and Methods

2.1. Samples

Samples were isolated according to a previously reported method [1].

2.1.1. Description of the HPLC-PDA analysis

HPLC-PDA analyses were performed using an Inertsil ODS-3 column (150 × 4.6 mm i.d., 5 μm) on a JASCO LC-2000 instrument equipped with a JASCO MD-2015 multiwavelength detector. CH₃CN containing 0.1% trifluoroacetic acid was used as the mobile phase with a flow rate of 0.6 mL/min.

2.1.2. Description of the ESIMS experiments

All HRESIMS and ESIMS/MS were carried out using a LTQ Orbitrap XL mass spectrometer (Thermo Fisher Scientific, Bremen, Germany) operated in negative ion mode at the Natural Science Center for Basic Research and Development (N-BARD), Hiroshima University. Helium was employed as the collision gas at a pressure of 1 mTorr for collision-induced dissociation (CID) experiments.

2.1.3. Description of the NMR experiments

Dried samples of **1–4** were dissolved in 0.6 mL of CDCl₃ and transferred to 5 mm i.d. × 180 mm NMR tubes for data acquisition. NMR spectra were acquired using a JEOL Lambda500 spectrometer (500 MHz for ¹H, 125 MHz for ¹³C) at the Natural Science Center for Basic Research and Development (N-BARD), Hiroshima University. Standard methods and pulse sequences were used to acquire 1D and 2D NMR spectra including 1D ¹H NMR spectra, 2D ¹H–¹H COSY spectra, 2D ¹H–¹³C HMQC spectra, and 2D ¹H–¹³C HMBC spectra. The NMR spectra were analyzed using the ALICE2 software (JEOL, Tokyo, Japan). ¹H and ¹³C NMR chemical shifts were referenced to residual solvent peaks: δ_H 7.26 (residual CHCl₃) and δ_C 77.7 for CDCl₃.

Ethics Statement

None.

Declaration of Competing 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.

CRediT Author Statement

Shinji Ohta: Conceptualization, Methodology; **Manami Takeda:** Investigation; **Emi Ohta:** Investigation; **Hisashi Ômura:** Investigation; **Mylene M. Uy:** Writing – original draft.

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Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi:[10.1016/j.dib.2022.107889](#).

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