Data Article

Nuclear magnetic resonance spectroscopy and mass spectrometry data for sulfated isoguanine glycosides

Yuri Uyama, Emi Ohta, Yui Harauchi, Tatsuo Nehira, Hisashi Omura, Hiroyuki Kawachi, Aya Imamura-Jinda, Mylene M. Uy, Shinji Ohta

Graduate School of Integrated Sciences for Life, Hiroshima University, 1-7-1 Kagamiyama, Higashi-Hiroshima, 739-8521, Japan
Graduate School of Biosphere Science, Hiroshima University, 1-7-1 Kagamiyama, Higashi-Hiroshima, 739-8521, Japan
Nagahama Institute of Bio-Science and Technology, 1266 Tamura-cho, Nagahama, Shiga, 526-0829, Japan
Department of Chemistry, Mindanao State University-Iligan Institute of Technology, Iligan City, 9200, Philippines

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ABSTRACT

The data presented here are related to the research paper entitled “Rare sulfated purine alkaloid glycosides from Bruchidius dorsalis pupal case” [1]. In this data article, we provide 1D and 2D nuclear magnetic resonance (NMR) spectroscopy and electrospray ionization mass spectrometry (ESIMS) data of three undescribed sulfated purine alkaloids, locustoside A disulfate, saikachinoside B disulfate, and saikachinoside A trisulfate isolated from the pupal case of the wild bruchid seed beetle Bruchidius dorsalis (Chrysomelidae, Bruchinae) infesting the seed of Gleditsia japonica Miquel (Fabaceae).

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1. Data

The data set presented in this article focuses on characterization of the sulfated purine alkaloids described in [1]. The article provides the information on the spectroscopic data of the sulfated purine alkaloids isolated from the pupal case produced by the bruchid beetle *Bruchidius dorsalis* inside the seed of *Gleditsia japonica* (Fig. 1). The $^1$H NMR spectra of 1–3 are shown in Figs. 2a, 3a and 4a, respectively. The $^{13}$C NMR and DEPT spectra of 1–3 are shown in Figs. 2b, 3b and 4b, respectively. 2D $^1$H–$^1$H COSY spectra of 1–3 are shown in Figs. 2c, 3c and 4c, respectively. 2D $^1$H–$^1$H NOESY spectra of 1–3 are shown in Figs. 2d, 3d and 4d, respectively. 2D $^1$H–$^{13}$C heteronuclear single quantum coherence (HSQC) spectra of 1–3 are shown in Figs. 2e, 3e and 4e, respectively. 2D $^1$H–$^{13}$C heteronuclear multiple-bond correlation (HMBC) spectra of 1–3 are shown in Figs. 2f, 3f and 4f, respectively. ESIMS data of 1–3 are shown in Figs. 2g, 3g and 4g, respectively. Analyses of the spectra of 1–3 are described in the research article [1]. It has been reported that 3 inhibited starfish blastulation during embryonic development [1].

Although some sulfated guanosine analogs, such as the kainate receptor inhibitor HF-7 [2], have been isolated from the venom of spiders [3], sulfated nucleoside derivatives from natural sources other than spiders are rare [1,4].

![Fig. 1. Structures of sulfated isoguanine glycosides isolated from pupal case produced by bruchid beetle *Bruchidius dorsalis* inside *Gleditsia japonica* seeds.](image-url)
2. Experimental design, materials, and methods

2.1. Samples

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

2.2. Description of the NMR experiments

Compounds 1–3 were dissolved in 0.6 mL of a mixture of CD$_3$OD and D$_2$O (1:9). All NMR spectra were acquired using a JEOL A400 spectrometer (400 MHz for $^1$H, 100 MHz for $^{13}$C). NMR analysis was performed using the ALICE2 software (JEOL, Tokyo, Japan). $^1$H and $^{13}$C NMR chemical shifts were referenced to residual solvent peaks: $\delta_H$ 3.30 (residual CHD$_2$OD) and $\delta_C$ 49.0 for CD$_3$OD. HRESIMS were carried out using a Thermo Fisher Scientific LTQ Orbitrap XL mass spectrometer at the Natural Science Center for Basic Research and Development (N-BARD), Hiroshima University.

3. Sulfated isoguanine glycosides 1–3

3.1. 6-Amino-7-(2,4-di-O-sulfo-$\beta$-D-glucopyranosyl)-3,7-dihydro-3-(3-methyl-2-buten-1-yl)-2H-purin-2-one (locustoside A disulfate) (1)

1D NMR, 2D NMR, and HRESIMS spectra of the compound 1 are shown in Fig. 2a–g.

3.2. 6-Amino-7-(6-O-$\alpha$-apio-$\beta$-D-furanosyl-2,4-di-O-sulfo-$\beta$-D-glucopyranosyl)-3,7-dihydro-3-[(2Z)-4-hydroxy-3-methyl-2-buten-1-yl)]-2H-purin-2-one (saikachinoside B disulfate) (2)

1D NMR, 2D NMR, and HRESIMS spectra of the compound 2 are shown in Fig. 3a–g.

![Fig. 2a. $^1$H NMR (400 MHz, CD$_3$OD–D$_2$O, 1:9) of 1.](image-url)
Fig. 2b. $^{13}$C NMR and DEPT (100 MHz, CD$_3$OD–D$_2$O, 1:9) of 1.

Fig. 2c. $^1$H–$^1$H COSY of 1.
Fig. 2d. $^1$H–$^1$H NOESY of 1.

Fig. 2e. $^1$H–$^{13}$C HSQC of 1.
Fig. 2f. $^1$H–$^{13}$C HMBC of 1.

Fig. 2g. (−-) HRESIMS of 1.

Calded for $C_{16}H_{21}N_2O_5S_2Na^- = 562.0531$

Calded for $C_{16}H_{21}N_2O_5S_2^- = 540.0712$  

Calded for $C_{16}H_{21}N_2O_5S_2^2^- / 2 = 269.5320$
**Fig. 3a.** $^1$H NMR (400 MHz, CD$_3$OD–D$_2$O, 1:9) of 2.

**Fig. 3b.** $^{13}$C NMR and DEPT (100 MHz, CD$_3$OD–D$_2$O, 1:9) of 2.
Fig. 3c. $^1$H–$^1$H COSY of 2.

Fig. 3d. $^1$H–$^1$H NOESY of 2.
Fig. 3e. $^1$H–$^{13}$C HSQC of 2.

Fig. 3f. $^1$H–$^{13}$C HMBC of 2.
Fig. 3g. (-)HRESIMS of 2.

Fig. 4a. $^1$H NMR (400 MHz, CD$_3$OD–D$_2$O, 1:9) of 3.
Fig. 4b. $^{13}$C NMR and DEPT (100 MHz, CD$_3$OD–D$_2$O, 1:9) of 3.

Fig. 4c. $^1$H–$^1$H COSY of 3.
Fig. 4d. $^1$H--$^1$H NOESY of 3.

Fig. 4e. $^1$H--$^{13}$C HSQC of 3.
Fig. 4f. $^1$H–$^{13}$C HMBC of 3.

Fig. 4g. (–)HRESIMS of 3.

T: FTMS - p ESI Full ms [100.00-2000.00]

$^{[M-2H]}^+$: Caled for C$_{16}$H$_{12}$N$_2$O$_{10}$S$_4^+ / 2 = 317.5078$

$^{[M-3H]}^+$: Caled for C$_{16}$H$_{10}$N$_2$O$_{9}$S$_4^+ / 3 = 211.3361$

$^{[M-H]}$:

Caled for C$_{16}$H$_{12}$N$_2$O$_{10}$S$_4^-$ = 636.0229

$^{[M-2H+Na]}^+$: Caled for C$_{16}$H$_{12}$N$_2$O$_{10}$S$_4$Na$^+$ = 658.0049
3.3. 6-Amino-3,7-dihydro-3-[(2Z)-4-hydroxy-3-methyl-2-buten-1-yl]-7-(2,4,6-tri-O-sulfo-β-D-glucopyranosyl)-2H-purin-2-one (saikachinoside A trisulfate) (3)

1D NMR, 2D NMR, and HRESIMS spectra of the compound 3 are shown in Fig. 4a–g.

Acknowledgments

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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.

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