Data Article

Two-dimensional NMR data of a water-soluble β-(1→3, 1→6)-glucan from Aureobasidium pullulans and schizophyllan from Schizophyllum commune

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A R T I C L E   I N F O

Article history:
Received 31 July 2017
Accepted 26 September 2017
Available online 1 October 2017

Keywords:
NMR
β-(1→3, 1→6)-glucan
Aureobasidium pullulans
Schizophyllan
Spectral data

A B S T R A C T

This article contains two-dimensional (2D) NMR experimental data, obtained by the Bruker BioSpin 500 MHz NMR spectrometer (Germany) which can used for the determination of primary structures of schizophyllan from Schizophyllum commune (SPG) and a water-soluble β-(1→3, 1→6)-glucan from Aureobasidium pullulans. Data include analyzed the 2D NMR spectra of these β-glucans, which are related to the subject of an article in Carbohydrate Polymers, entitled “NMR spectroscopic structural characterization of a water-soluble β-(1→3, 1→6)-glucan from

DOI of original article: http://dx.doi.org/10.1016/j.carbpol.2017.07.018
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http://dx.doi.org/10.1016/j.dib.2017.09.067
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A. pullulans” (Kono et al., 2017) [1]. Data can help to assign the \(^1\)H and \(^{13}\)C chemical shifts of the structurally complex polysaccharides.

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### Specifications Table

| Subject area                | Chemistry                                      |
|-----------------------------|-----------------------------------------------|
| More specific subject area  | Structural analysis                           |
| Type of data                | NMR spectra                                   |
| How data was acquired       | NMR, Bruker BioSpin AVIII 500 MHz spectrometer|
| Data format                 | Analyzed                                       |
| Experimental factors        | About 30 mg of each sample dissolved in 600 \(\mu\)L of 99.9% dimethylsulfoxide (DMSO)-d6. |
| Experimental features       | All NMR experiments were performed at 363 K.   |
| Data source location        | National Institute of Technology, Tomakomai College, Nishikioka 443, Tomakomai, Hokkaido 059 1275, Japan |
| Data accessibility          | Data are with this article.                    |

### Value of the data

- The following data detail NMR characterization of a novel water-soluble \(\beta\)-(1\(\rightarrow\)3, 1\(\rightarrow\)6)-glucan and schizophyllan from Schizophyllum commune.
- The NMR data can be helpful to estimate the branching patterns of other \(\beta\)-glucans.
- NMR parameters for the data can be useful for structural characterization of complex polysaccharides.

### 1. Data

The presented data include 2D NMR spectra of schizophyllan from Schizophyllum commune (SPG) and a water-soluble \(\beta\)-(1\(\rightarrow\)3, 1\(\rightarrow\)6)-glucan from Aureobasidium pullulans (A. pullulans) whose primary structures are shown in Fig. 1. \(^1\)H–\(^{13}\)C heteronuclear single quantum coherence (HSQC), 2D \(^1\)H–\(^{13}\)C heteronuclear multiple-bond correlation (HMBC), and 2D \(^1\)H–\(^1\)H rotating frame Overhauser effect spectroscopy (ROESY) spectra of SPG are shown in Figs. 2–4, and those of the water-soluble A. pullulans \(\beta\)-(1\(\rightarrow\)3, 1\(\rightarrow\)6)-glucan are in Figs. 5–7, respectively.

### 2. Experimental design, materials and methods

The experiment’s planning, design, and data processing correspond to the protocol given in Refs. [1,2].
Fig. 2. HSQC spectrum of SPG in DMSO-\(d_6\) at 363 K. The vicinal \(^1\text{H}\)--\(^{13}\text{C}\) spin couplings of the A1, B1, B2, and C1 residues in SPG (Fig. 1) are denoted by solid red, solid and dashed blue, and solid green lines, respectively. \(^1\text{H}\) and \(^{13}\text{C}\) NMR spectra of SPG are shown in horizontal and vertical axes in the HSQC spectrum, respectively, and the \(^1\text{H}\) and \(^{13}\text{C}\) resonance assignments are indicated in the \(^1\text{H}\) and \(^{13}\text{C}\) spectra.

Fig. 1. Primary structures of schizophyllan (SPG) and the water-soluble \(\beta-(1\rightarrow3, 1\rightarrow6)\)-glucan from \textit{Aureobasidium pullulans}. The A1, B1, B2, and C1 residues in SPG and A1, A2, B1, B2, C1, and C2 residues in the \(\beta-(1\rightarrow3, 1\rightarrow6)\)-glucan are magnetically inequivalent in their structures.
Fig. 3. HMBC spectrum of SPG in DMSO-$d_6$ at 363 K. The arrows indicate the interresidual correlations between $A_1H_1$–$B_1C_3$, $B_1H_1$–$B_2C_3$, $B_2H_1$–$A_3C_3$, and $C_1H_1$–$A_1C_6$ via glycosidic bonds.

Fig. 4. ROESY spectrum of SPG in DMSO-$d_6$ at 363 K. The arrows indicate the interresidual correlations between $B_1H_3$–$A_1H_1$, $A_1H_3$–$B_2H_1$, $B_2H_3$–$B_1H_1$, $A_1H_6a$–$C_1H_1$, and $A_1H_6b$–$C_1H_1$ via glycosidic bonds.
Fig. 5. HSQC spectrum of the water-soluble β-(1→3, 1→6)-glucan from A. pullulans in DMSO-d$_6$ at 363 K. The vicinal $^1$H–$^{13}$C spin couplings of the A1, A2, B1, B2, C1, and C2 residues in the β-(1→3, 1→6)-glucan (Fig. 1) are denoted by solid and dashed red, solid and dashed blue, and solid and dashed green lines, respectively. $^1$H and $^{13}$C NMR spectra of the β-(1→3, 1→6)-glucan are shown in horizontal and vertical axes in the HSQC spectrum, respectively, and the $^1$H and $^{13}$C resonance assignments are indicated in the $^1$H and $^{13}$C spectra.

Fig. 6. HMBC spectrum of the water-soluble β-(1→3, 1→6)-glucan from A. pullulans in DMSO-d$_6$ at 363 K. The arrows indicate the inter-residual correlations between C1C1–A1H6a, C1C1–A1H6b, C2C1–A2H6a, C2C1–A2H6b, CH1–A1C6, and C2H1–A2C6 via glycosidic bonds.
2.1. Samples

SPG was purchased from InvivoGen (USA). The water-soluble *A. pullulans* \(\beta-(1\rightarrow3, 1\rightarrow6)\)-glucan was prepared according to a previously reported method[1,2].

2.2. Description of the NMR experiments

Each sample was dissolved in 600 \(\mu\text{L}\) of DMSO-\(d_6\) (99.9% isotropic purity, Sigma-Aldrich (USA)). All NMR spectra were recorded on a Bruker AVIII 500 MHz spectrometer at 363 K. HSQC data were acquired on a 2048 \(\times\) 256-point matrix for the full spectrum, with 96 scans per increment, and the interpulse delay which corresponded to \(1/4J_{CH}\) was set to 3.44 ms. HMBC) data were acquired on a 1024 \(\times\) 256-point matrix for the full spectrum, with 128 scans per increment, and the delay time for the evolution was set to 62.5 ms. ROESY data were acquired on a 2048 \(\times\) 256-point matrix for the full spectrum with 64 scans per increment, and the mixing time was 200 ms. The repetition time of each 2D NMR experiment was 2 s, and all 2D NMR data were zero-filled to 2k in both dimensions prior to Fourier transformation. \(^1\text{H}\) and \(^{13}\text{C}\) chemical shifts were calibrated using the methyl resonances of DMSO at 2.52 ppm for \(^1\text{H}\) and 39.52 ppm for \(^{13}\text{C}\).

Acknowledgements

This work was supported by the Japan Society for Promotion of Science (JSPS) (Grant no. JP16K05802).
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