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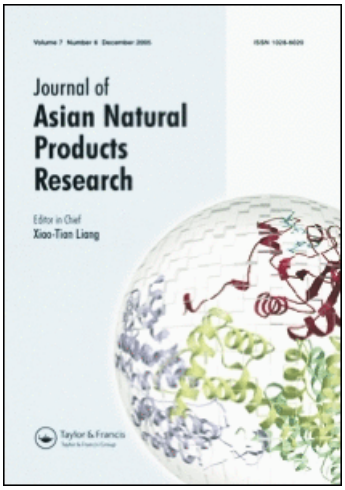

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A novel sulphur glycoside from the seeds of *Descurainia sophia* (L.)

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Note

A novel sulphur glycoside from the seeds of *Descurainia sophia* (L.)

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A new sulphur glycoside, named descurainoside (**1**), and the known compound sinapic acid (**2**) have been isolated from the seeds of *Descurainia sophia* (L.) Webb ex Prantl. The structure of **1** has been identified as (1*R*,6*S*,8*R*,9*S*,10*S*)-9,10-dihydroxy-4-[(4-hydroxy-3,5-dimethoxyphenyl)methylene]-8-(hydroxymethyl)-2,7-dioxa-5-thiabicyclo[4.4.0]decan-3-one by means of physico-chemical properties and spectroscopic methods (1D and 2D NMR, HRMS, ESI-MS).

Keywords: *Descurainia sophia* (L) Webb ex Prantl; Sulfur glycoside; (1*R*,6*S*,8*R*,9*S*,10*S*)-9; 10-Dihydroxy-4-[(4-hydroxy-3, 5-dimethoxyphenyl)methylene]-8-(hydroxymethyl)-2; 7-dioxa-5-thiabicyclo[4.4.0]decan-3-one

1. Introduction

Descurainia sophia (L.) Webb ex Prantl is widely distributed in the Northeast of China, and its seeds have been used as a Chinese traditional medicine to relieve cough, prevent asthma, reduce edema, promote urination and have a cardiotonic effect. Some cardiac glycosides from the seeds have been reported [1]. The present paper describes the isolation and structural elucidation of a new sulphur glycoside, descurainoside (**1**) [figure 1(A)].

2. Results and discussion

Descurainoside (**1**) was isolated as a yellow powder, mp 225–227°C. It responded positively to the Molish test and to $FeCl_3$. Acid hydrolysis in a test tube yielded glucose and H_2S that responded positively to a $Pb(Ac)_2$ test paper but, unfortunately, the aglycone

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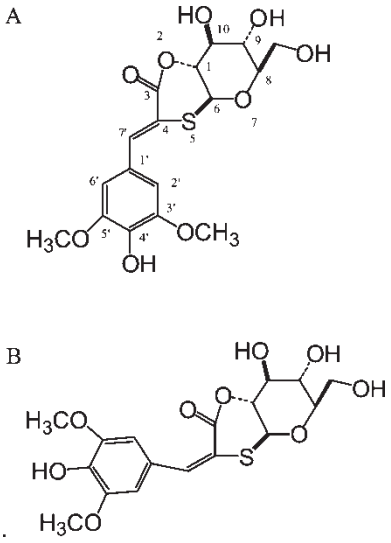

Figure 1. Structure of compound **1**.

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was not obtained. Its HRMS showed a quasi-molecular ion peak at *m/z* 401.0915 $[M + H]^+$. In the ESI-MS spectrum exhibits quasi-molecular ion peaks at *m/z* 401.0 $[M + H]^+$ and 399.2 $[M - H]^-$. The molecular formula of **1** was suggested to be $C_{17}H_{20}O_9S$.

The ^1H NMR spectrum of **1** shows the characteristic anomeric proton signal of sulphur glycosides at δ 5.25 (1H, d, J = 9.3 Hz, H-6) [2], indicating a β orientation of the anomeric center of the glucose. The spectrum also shows one phenol hydroxy signal at δ 9.19 (1H, s, 4-OH), two aromatic proton signals at δ 6.99 (2H, s, H-2,6), and one olefinic proton signal at δ 7.59 (1H, s, H-7), indicating that C-4 is substituted compared with *trans*-sinapic acid, and two methoxy groups at δ 3.80 (6H, s, 3,5-OCH_3). The ^{13}C NMR spectrum exhibits 17 carbon signals, of which six aromatic carbons in the downfield and two methoxy carbons correspond to those of the phenyl moiety of *trans*-sinapic acid. In addition, characteristic carbon signals [2] are ascribed to the sugar unit of sulphur glycoside at δ 75.3 (C-6), 80.7 (C-1), 73.9 (C-10), 69.7 (C-9), 82.0 (C-8) and 60.7 (8-CH_2OH) (table 1).

In the HMQC experiment, the correlated peaks are found between δ 7.59 (H-7) and 137.7 (C-7), δ 6.99 (H-2,6) and 108.6 (C-2, 6), δ 5.25 (H-6) and 75.3 (C-6), δ 3.92 (H-1) and 80.7 (C-1). In the $^1H—^1H$ COSY experiment, no correlated peaks of the aglycone moiety appear. In the HMBC experiment (figure 2), long-range correlations between δ 7.59 (H-7) and 108.6(C-2,6), δ 7.59(H-7) and 164.2(C-3), δ 5.25(H-6) and 115.6(C-4) indicate the connection

Table 1. NMR data of compound **1** (DMSO).

No.	δ_H	δ_C	*No.*	δ_H	δ_C
1	3.92 (1H, t, J = 9.3 Hz)	80.7	2′	6.99 (1H, s)	108.6
3		164.2	3′		147.8
4		115.6	4′		137.8
6	5.25 (1H, d, J = 9.3 Hz)	75.3	5′		147.8
8	3.37 (1H, m)	82.0	6′	6.99 (1H, s)	108.6
9	3.22 (1H, m)	69.7	7′	7.59 (1H, s)	137.7
10	3.56 (1H, m)	73.9	-CH_2OH	3.70 (1H, m) 3.44 (1H, m)	60.7
1′		124.3	-OCH_3	3.80 (6H, s)	56.2

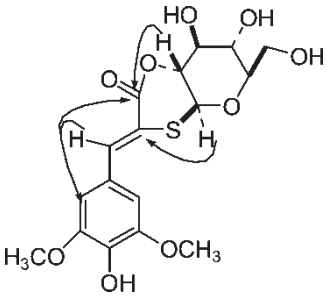

Figure 2. Important HMBC correlations of **1**.

of C-6 to C-4 by a sulphur atom. In addition, the long-range correlation between δ 3.92(H-1) and 164.2(C-3) indicates the connection of C-1 to C-3 by oxygen. From the above evidence, the structure of **1** was deduced as either (A) or its isomer (B) shown in figure 1. From searches of *Chemical Abstracts* and the Scifinder system, either A or B is a new compound. Unfortunately, an attempt to determine the stereochemical structure of **1** by a NOESY experiment revealed no valuable evidence. However, most derivatives of sinapic acid in nature possess a *trans*-configuration [3–5], similar to (A). Therefore the structure of **1** may well be (1*R*,6*S*,8*R*,9*S*,10*S*)-9,10-dihydroxy-4-[(4-hydroxy-3,5-dimethoxyphenyl)methylene]-8-(hydroxymethyl)-2,7-dioxa-5-thiabicyclo[4.4.0]decan-3-one, named descurainoside. However, further work is needed to confirm the stereochemical structure of **1**.

Compound **2** was identified as *trans*-sinapic acid (figure 3) by comparing its physicochemical properties and NMR data with those in the literature [3].

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3. Experimental

3.1 General experimental procedures

The melting point was measured on a Yamaco micro-hot-stage and is uncorrected. NMR spectra were recorded on a Bruker-ARX-300 spectrometer, using TMS as an internal standard. ESI-MS was performed on a Finnigan LCQ mass spectrometer. HRMS was performed on a QSTAR LCQ mass spectrometer. The optical rotation was measured using

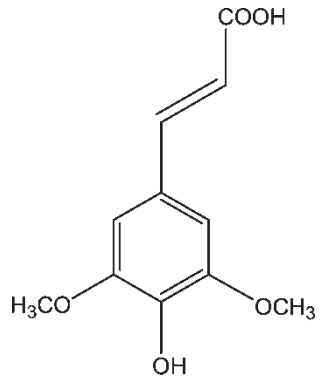

Figure 3. Structure of compound **2**.

a Perkin-Elmer 241 polarimeter. Silica gel for chromatography was produced by the Qingdao Ocean Chemical Group Co. Ltd., China. Macroporous resin D101 for chromatography was produced by Nankai University.

3.2 Plant material

The plant material was purchased from Shenyang TCM Corporation (Shenyang), and was identified by Professor Sun Qishi (Shenyang Pharmaceutical University). A voucher specimen (no. 20010321) has been deposited in the Research Department of Natural Medicine, Shenyang Pharmaceutical University.

3.3 Extraction and isolation

Air-dried seeds (10 kg) of *Descurainia sophia* were extracted with 70% ethanol, and the extract was then concentrated *in vacuo* and chromatographed on a D101 macroporous resin column, eluting gradiently with H_2O and 20%, 40%, 60% and 95% ethanol successively. The 20% eluate (120 g) was then subjected to column chromatography on silica gel and gradiently eluted with $CHCl_3$–MeOH. The so-obtained fourth fraction ($CHCl_3$–MeOH, 100:3) was then recrystallized from MeOH to give compound **2** (15.6 mg). The sixth fraction ($CHCl_3$–MeOH, 100:5) was recrystallized from MeOH to give compound **1** (20.0 mg).

Descurainoside (**1**): yellow powder (MeOH), mp 225–227°C. $[\alpha]_D^{20} = -34.0$ (c 0.001, MeOH). ^{1}H (300 MHz, DMSO-d_6) and ^{13}C (75 MHz, DMSO-d_6) NMR, see Table I. HRMS: m/z 401.0915 (calcd for $C_{17}H_{21}O_9S$, 401.0906). ESI-MS: m/z 401.0 $[M + H]^+$ and 399.2$[M - H]^-$.

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