



Phytochemical and chemotaxonomic study on the whole plants of *Ligularia sagitta*



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ABSTRACT

The phytochemical study of the whole plants of *Ligularia sagitta* led to the isolation of 26 compounds, including a new eremophilane-type sesquiterpene (1) and twenty-five known compounds, i.e. eleven sesquiterpenes (2–12), one diterpene (13), one triterpene (14), one sterol (15), four flavonoids (16–19), one lignan (20), one cumarin (21), three phenylpropanoids (22–24), one benzene derivative (25), and one alkaloid (26). The structures of all isolated compounds were elucidated on the basis of extensive spectroscopic methods (HRESIMS, 1D and 2D NMR experiments), as well as by comparison with literature data. Among them, ten compounds (4–6, 8–9, 13, 16–17, 19, 26) were reported for the first time from the genus *Ligularia*. The chemotaxonomic significance of the isolated compounds is discussed.

1. Subject and source

The genus *Ligularia* (Asteraceae) contains about 130 species distributed widely in Asia and Europe. There are 111 species in China, mainly growing in the southwest mountainous area of China (Editorial Committee for Flora of the Chinese Academy of Science, 1989). More than 20 species have long been used as Chinese traditional folk medicine for cough phlegm, removing blood stasis, emetic, diuresis, cholangogue and removing jaundice, especially their root and rhizome, which are named as “Zi-Wan” in Chinese traditional folk medicine (Zhang et al., 2003). Phytochemical investigations of various *Ligularia* species showed the presence of monoterpenes, sesquiterpenes, diterpenes, triterpenes, alkaloids, steroids, flavonoids, lignans, and other skeleton type compounds, as well as some of these compounds showed various biological activities such as anticancer, antibacterial, anti-hepatotoxicity, antioxidant, and antithrombus (Yang et al., 2011).

Ligularia sagitta (Maxim) is a perennial herb growing throughout Tibet, Sichuan, Qinghai, Gansu, Ningxia, Shanxi, Hebei, and Inner Mongolia Provinces in China (Editorial Committee for Flora of the Chinese Academy of Science, 1989). It has been used as a Chinese traditional folk medicine to reduce phlegm, relieve cough, cure pulmonary tuberculosis, urinarytract blockages, common cold, and pharyngitis (Li et al., 2003). In this study, the whole plants of *L. sagitta* were collected in mountain areas at altitudes of 2100–2500 m in Beishan Forest, Huzhu County of Qinghai province, P. R. China, in September

2012 and authenticated by Prof. Guo-liang Zhang from the Department of Life Science, Lanzhou University. A voucher specimen (No. Lsa20120904) has been deposited at College of Chemistry and Chemical Engineering, Lanzhou University.

2. Previous work

Previous phytochemical studies on *L. sagitta* have reported the presence of sesquiterpenes, diterpenes and triterpenes (Chen et al., 1992, 2014; Zhao et al., 1994; Peng et al., 1997; Li et al., 2003, 2004, 2007, 2008; Li and Jia, 2008), which revealed that eremophilane sesquiterpenes are the main components of *L. sagitta*.

3. Present study

In this study, a new sesquiterpene (1) and twenty-five known secondary metabolites (2–26) were isolated from the whole plants of *L. sagitta*. Their structures were established by spectroscopic analysis, especially NMR experiments, and by comparison of their physical and spectroscopic data with the literature data. The chemical structures of 1–26 were shown in Fig. 1.

3.1. Extraction and isolation

The air-dried and powdered whole plants of *L. sagitta* (9.5 kg) were

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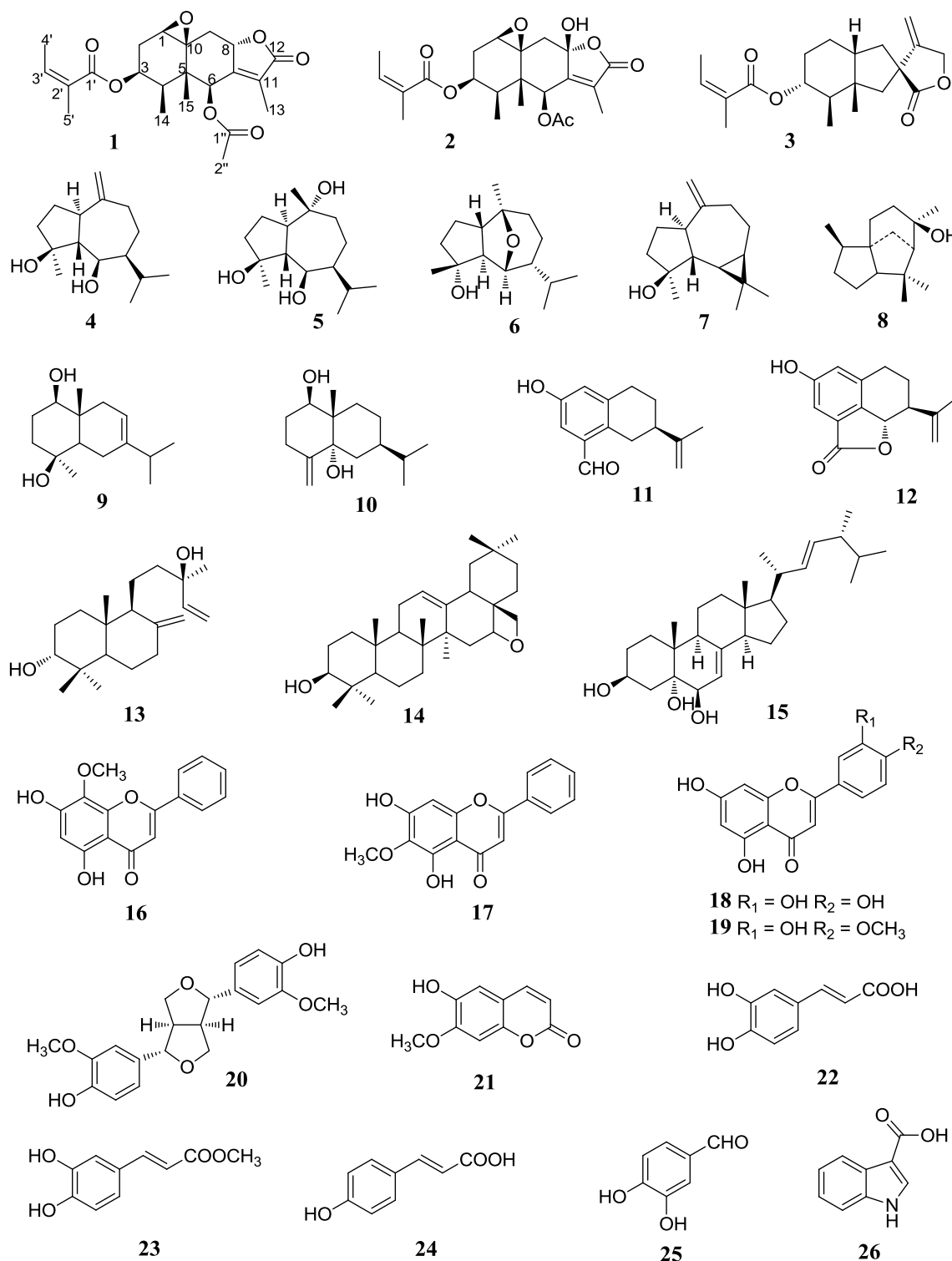


Fig. 1. Structures of compounds 1–26 from *L. sagitta*.

extracted successively with mixed solvent of petroleum ether (PE, 60–90 °C)/Et₂O/MeOH (1:1:1, v/v/v, 65 L × 3) and MeOH (55 L × 3) for three times (5 days each time) at room temperature, and then boiled under reflux with MeOH (45 L × 3) for 12 h. The filtrate was concentrated and yielded dried residue 403.0 g, 355.0 g, and 344.0 g after removing the solvent at reduced pressure, respectively. The residue was suspended in distilled H₂O and then partitioned sequentially with PE, ethyl acetate (EtOAc), and *n*-butyl alcohol (*n*-BuOH), yielding PE fraction (220.0 g), EtOAc fraction (96.0 g), *n*-BuOH fraction (129.0 g), and

aqueous fraction (426.0 g), respectively.

The PE fraction (220.0 g) was subjected to silica gel column chromatography (CC) by gradient elution with PE/acetone (100:0 to 0:1, v/v) as the eluent, and finally washed with MeOH, to give 16 fractions (Fr-1 – Fr-16) based on TLC analysis. Fr-2 (3.6 g) was subjected to silica gel CC eluted with PE/EtOAc (30:1 to 0:1, v/v) to obtain five fractions (Fr-2-1 – Fr-2-5). Fr-2-4 (301.0 mg) was subjected to Sephadex LH-20 column with CHCl₃/MeOH (1:1) as the eluent, then subjected to silica gel preparative thin-layer chromatography (PTLC) using CHCl₃/EtOAc

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