Mongolian Journal of Chemistry 16 (42), 2015, 44-47

Mongolian Academy of Sciences

Mongolian Journal of Chemistry

Institute of Chemistry & Chemical Technology

Alkaloids from *Sedum telephium* L.

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ARTICLE INFO: Received 04 November 2015; revised 18 January 2016; accepted 21 January 2016

Abstract: The crude alkaloid mixtures from the aerial parts *S.telephium* was analyzed by GC-MS method. As a result 14 compounds, including 6 alkaloids were characterized. 3-methyl-2-carbethoxyindole (4.730%), 2-(2-hydroxyphenyl) benzothiazole (1.576%) and N,4, 5-trimethyl phenyl-1,2-diamine, (1.217%) were in higher contents. One sulfur-containing alkaloid 2-(2-hydroxyphenyl) benzothiazole has been identified. These six alkaloids are described for the first time from this plant.

Keywords: *Crassulaceae; morpholine, indole, pyridine, thio alkaloids, benzothiazoles, GC/MS*

INTRODUCTION

The plants *Sedum telephium* L. (Crassulaceae) is native to Eurasia. The members of the large genus are commonly known as stonecrops. This genus contains around 600 species of leaf succulents that are found throughout the Northern Hemisphere. *Sedum acre* was used to treat epilepsy and skin disease, and was used as an abortifacient in ancient Greece. Many plants of this genus are cultivated as garden plants, due to their attractive appearance and hardiness [1]. Anti-inflammatory, antitumor, and immunosuppressive activities of this genus have been investigated [2-5]. Also, antioxidant activity of total flavonoids from *S.sarmentosum* [6] and antimicrobial activity of essential oils from *S.pallidum var. bithynicum* and *S.spurium* [7] have been reported. Some of the *Sedum* species contain alkaloids. Leaves from 16 Asian species of Sedum were tested for the presence of alkaloids. Only seven species contained alkaloids. These species, *Sedum bulbiferum, S.japonicum, S.lepidopodium, S.orrisonensis, S.oryzifolium, S.polytrichoides* and *S.sarmentosum,* contain14 pyrrolidine and piperidine alkaloids [8]. Five species (*Sedum aizoon* L., *S.ewersii* Ledeb., *S.hybridum* L., *S.roseum* (L.) Scop. and *S.telephium* L.) are distributed in Mongolia. *S.telephium* is relatively widespread species in Mongolia [9]. Sedum genus are used in Mongolian traditional medicine for concealed hemorrhage, hematemesis, discharge of blood, metrorrhagia, wounds, traumatic injuries, painful oedema, and insomnia due to discomfort, emotion, alarm [10].

From Mongolian *Sedum* species only phytochemical and some pharmacological study on *Sedum hybridum* L. has been performed [11]. There is no report on alkaloid content of the genus Sedum growing in Mongolia. In this work we report our results on the GC-MS analysis of crude alkaloid mixtures from the aerial parts *S.telephium* L.

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DOI: http://dx.doi.org/10.5564/mjc.v16i0.670

EXPERIMENTAL

Plant material: The aerial parts of *S.telephium* were collected from Arkhangai Province (30 km from the Southwest of Tsetserleg) of Mongolia, during the flowering period in early August 2013. A voucher specimen (1326-2) is deposited at the Herbarium fund of the Institute of General and Experimental Biology, Mongolian Academy of Sciences. The plant material was identified by Prof. E.Ganbold, Institute of the General and Experimental Biology, Mongolian Academy of Sciences.

Extract of crude alkaloid from the aerial parts of S.telephium: The air-dried and powdered aerial parts (0.9 kg) of *S.telephium* were extracted exhaustively with 95% ethanol at room temperature for 3 times. The combined ethanolic solution was evaporated under reduced pressure and the residue was dissolved in 5% HCl (pH 1-2) and allowed to stand overnight at room temperature. Insoluble non-alkaloid materials were removed by filtration, and the filtrate was extracted with n-hexane to eliminate the rest of the non-alkaloid substances. Thus the purified acidic solution was made alkaline to pH 9-10 with 25% NH_4OH and extracted exhaustively with chloroform. The combined $CHCl_3$ solution was dried over anhydrous Na_2SO_4 and then concentrated to give crude alkaloid mixtures (0.56 g).

Analysis of volatile compounds of crude alkaloid mixtures by GC-MS: The Gas Chromatography-Mass Spectrometry (GC-MS) analyses were carried out on Hewlett Packard 6890/MSD5973 instrument operating in EI mode at 70 eV, in the laboratory in the Centre of Phytochemistry, Institute of Organic Chemistry, Bulgarian Academy of Sciences. An HP–5 MS column (30 m×0.25 mm×0.25 μm) was used. The

temperature program was 70 to 290ºC at 6ºC min^{-1} and 10 min hold at 300ºC. Injector temperature was 280ºC. Nitrogen was used as a carrier gas at 0.8 ml/min^{-1}. The identification of components was accomplished by computer searches in the HP Mass Spectral Library NIST 98 (Hewlet-Packard, Palo Alto, California, USA).

RESULTS AND DISCUSSION

The crude alkaloid mixtures from the aerial parts *Sedum telephium* was analyzed by GC-MS. The results were summarized in Figure 1 and Table 1.

The crude alkaloid fraction has a complex chemical composition. We have tried to identify the alkaloids and other components with larger contents. Some of the GS-MS peaks remained unidentified, because of the lack of reliable data and references of the corresponding compounds.As a result of our GC-MS analysis, 14 substances, including 6 alkaloids were identified. In addition to the alkaloids, 8 non-alkaloids were characterized. From these compounds relative large quantities was: dehydromevaloniclastone (5.992%); 4-(3-hydroxy-1-butenyl)-3,5,5-trimethyl-2-cyclohexen-1-one, (4.413%); 4-((1E)-3-hydroxy-1-propenyl) -2-methoxyphenol, (4.315%) and 4-(3-hydroxybutyl)-3,5, 5-trimethyl-2-cyclohexen-1-one, (3.809%).

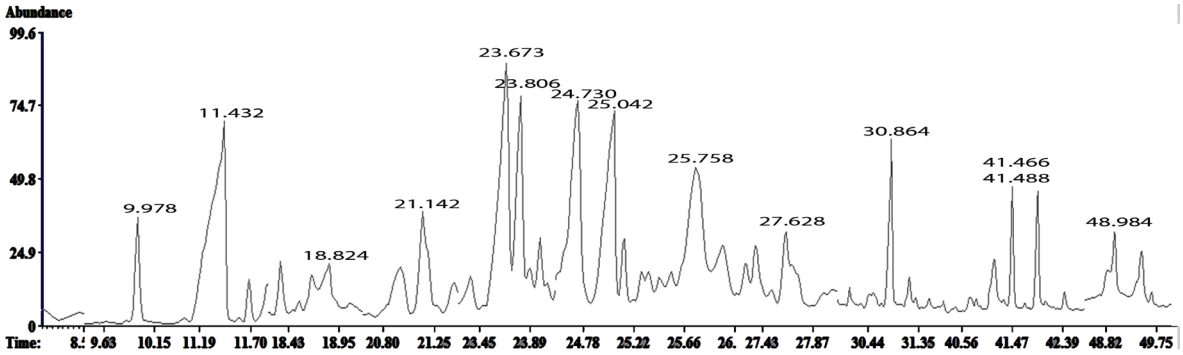
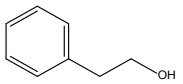
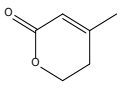
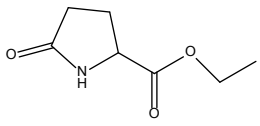
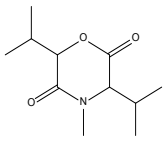
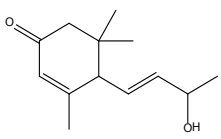
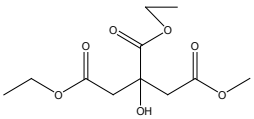
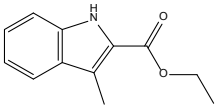
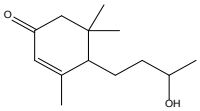
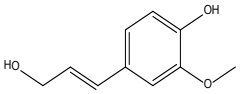
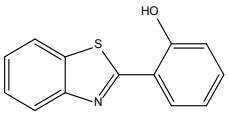
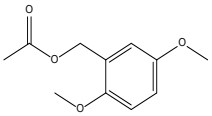
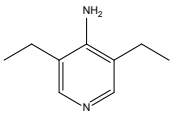
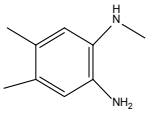
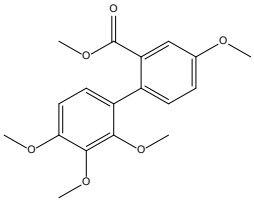


Fig. 1. GC-MS profile of the fourteen constituents in crude alkaloid mixtures of *Sedum telephium* L.

Table1. GS-MS analysis of the crude alkaloid mixtures from *Sedum telephium* L.

Retention time, min	% of total ion current	Name, formula, MW, MS, RSI	Structure
9.978	0.997	Phenylethyl Alcohol (**1**), $C_8H_{10}O$, 122 122 (27.2), 92 (57.8),91 (99.9), 65 (16.6), 39(6.6) 938	
11.432	5.992	Dehydromevalonic lactone (**2**) $C_6H_8O_2$, 112 112 (49.0),82 (99.9),39 (33.5), 54 (32.9), 53 123 925	
18.824	0.893	2-Pyrrolidinecarboxylic acid-5-oxo-,ethyl ester (**3**) $C_7H_{11}NO_3$, 157 157 (3.2),84 (99.9),41 (12.7), 56 (74), 85(53) 924	
21.142	1.632	4-Methyl-3,6-diisopropyl-2,5-diketo- morpholine (**4**) $C_{11}H_{19}NO_3$, 213 171 (99.9), 84 (33.5), 42 (91.8), 83 (79.9), | 69 (43.7) 854	
23.673	4.413	2-Cyclohexen-1-one, 4-(3-hydroxy-1-butenyl)-3,5,5-trimethyl (**5**) $C_{13}H_{20}O_2$, 208 152 (14.8),109 (28.7), 108 (99.9), 43 (14.6), 107 (12.0) 876	

23.806	2.447	Ethyl citrate (**6**) $C_{12}H_{20}O_7$, 276 203 (18.8), 158 (8.0), 157 (99.9), 115 (30.3),43 (9.9) 948	
24.730	4.730	3-Methyl-2-carbethoxyindole (**7**) $C_{12}H_{13}NO_2$, 203 203 (50.2), 174 (23.8), 157 (99.9), 129 (85.4),101 (45.8) 739	
25.042	3.809	2-Cyclohexen-1-one, 4-(3-hydroxybutyl)-3,5,5-trimethyl (**8**) $C_{13}H_{22}O_2$, 210 135 (99.9), 109 (62.4), 108 843 93 (80.50, 95 (66.8) 912	
25.758	4.315	4-((1E)-3-Hydroxy-1-propenyl)-2-methoxyphenol (**9**) $C_{10}H_{12}O_3$, 180 180 (75.2), 137 (99.9), 124 (55.1), 91 (32.7) 119 (19.9) 891	
27.628	1.576	2-(2-Hydroxyphenyl) benzothiazole (**10**) $C_{13}H_9NOS$, 227 226 (96.4), 198 (31.3), 181 (99.9), 211 (13.5), 147 (21.2) 693	
30.864	2.304	Benzenemethanol, 2,5-dimethoxy-, acetate (**11**) $C_{11}H_{14}O_4$, 210 (99.9), 167 (83.8), 154 (31.5), 149 (24.5),182 (24.3) 685	
41.466	1.117	4-Amino-3,5-diethylpyridine (**12**) $C_9H_{14}N_2$, 150 150(99.9), 135(13.4), 151 (3.3), 107 (3.6), 77(3.2) 798	
41.488	1.217	N,4,5-trimethylphenyl-1,2-diamine, (**13**) $C_9H_{14}N_2$, 150 150 (99.9), 135 (13.1), 151(13.0), 107 (3.5), 152 (2.9) 834	
48.984	2.588	Benzoic acid, 5-methoxy-2-(2,3,4-trimethoxyphenyl)-, methyl ester(**14**) $C_{18}H_{20}O_6$, 332 334(2.8), 333 (18.8), 332 (99.9), 331(2.8), 197 (2.4) 707	

Six alkaloids were described. Among these alkaloids **7** (4.730%), **10** (1.576%) and **13** (1.217%) were in relatively higher contents. One sulfur-containing alkaloid 2-(2-hydroxyphenyl) benzothiazole (**10**) has been identified and could be an interesting fact. Previously the thioalkaloids have been isolated from plants. For example immunosuppressive, anti-metastatic, anti-inflammatory and anticancer active thioalkaloids were isolated from Nuphar [12-14]. Our data show that Sedum genus may contain not only pyrrolidine and piperidine alkaloids, but also morpholine, indole, pyridine, thioalkaloid and other class of alkaloids.

CONCLUSIONS

Six alkaloids are described for the first time from *S.telephium* L. growing in Mongolia. The present study determined the chemical characteristic profiles and identified the probable phytochemicals. According to their structure, some of them could be considered as active ingredients. Furthermore it is necessary to isolate these compounds in pure form and to analyze their bioactivities.

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