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A Pentacyclic Triterpenoid from *Rubus sanctus*

Rubus sanctus' tan Elde Edilen Pentasiklik bir Triterpenoit

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Abstract

Rubus species are used for their analgesic and antidiabetic activities in traditional folk medicine. In this work, a triterpenic compound related with analgesic activity has been isolated. The compound is 2α,3α,19α,24 tetrahydroxyurs-12-en-28-oic acid-28-O-β-glucopyranosyl ester.

Key words: *Rubus sanctus*, Rosaceae, Triterpenoid

Introduction

Nine *Rubus* species (Rosaceae) grow widely in the Turkish flora (Davis,1965). Different parts of *Rubus* species have been used in traditional folk medicine as antibacterial, antiinflamatory, antidiabetic and for the treatment of diseases such as arthritis, rheumatism (Baytop,2000). Flavonoids, coumarins, terpenic acids and terpenoids are mainly found in the aerial parts of the species. Previously, we investigated the plant extracts from the microbiological point of view and the found that the extracts showed antibacterial activity (Ulusoylu, *in press*). In this work a triterpenoid compound was isolated and identified.

Materials and Methods

*Plant material:*The plant was collected from Northern Turkey. It was identified by Prof. Dr. Ertan Tuzlacı. The voucher specimens are deposited in the Marmara University Faculty of Pharmacy Herbarium (MARE 8146).
Extraction and isolation: The aerial parts of *R. sanctus* were air dried (2.5 kg), powdered and macerated with MeOH at room temperature. The MeOH macerate was evaporated to dryness. The residue was dissolved in warm water and the solution was successively extracted with PE (9.1g), $CHCl_3$ (15g), EtOAc (17g). All extracts were controlled with TLC and glycosidic compounds were identified in the EtOAc extract. The purification of EtOAc extract was started

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by silicagel column. The column was eluted with $CHCl_3$, EtOAc and MeOH. The compound was separated from EtOAc: MeOH (60:40) fraction. The preparative TLC was used for further purification

Results and Discussion

Many chemical investigations on *Rubus* species showed that ursane - oleanane triterpenoids are the major compounds and these are found to be pharmacologically effective (Bin-Gui *et al.*,2000, Nierro *et al.*,1999). The compound is an amorphous white powder, hydroxyl group (3295 cm^{-1}), ester carbonyl group (1721 cm^{-1}), trisubstituted double bond (1658 cm^{-1}) and glycosidic linkage (1054 cm^{-1}) are seen in its IR spectrum. The FABMS spectrum shows peaks at m/z 689$[M+Na]^+$ and 504$[M\text{-glu}]^+$, which suggests the molecular formula is $C_{36}H_{58}O_{11}$, which is further confirmed by ^{13}C NMR and DEPT data in Table 1. The ^{13}C NMR spectrum of the compound shows 30 signals, a triterpenoid structure, six peaks in the range at δ60-98 (97.8, 79.2, 73.7, 72.3, 60.3) corresponding to the presence of a glucose moiety and the anomeric carbon signal at δ 96.3 (CH) shows an ester linkage with the aglycone. The charecteristic signal for the H-18 of ursane type triterpenoids with 19α-hydroxyl substitution seen at 2.83 singlet in the ^1H NMR spectrum, together with a pair of double bond signals at δ 126.8 (CH, C-12) and 134.8 (C-13) in the ^{13}C NMR spectrum, suggest a 19α-hydroxyurs-12-en skeleton for the aglycone of the compound (Lien *et al.*, 1999). On alkaline hydrolysis the compound gave D-glucose as the sugar component which was identified by direct comparison with an authentic sample. The ^1H NMR spectrum of the compound showed signals at 3.78 (m, H-2β), δ 3.46 (br s, H-3β), which suggested the α-configuration for the two hydroxyl groups on ring A. The compound which has chemical shifts for C-2 and C-3, is the same with those which are reported to have 2α,3α-diol system (Zhi, 1998). This also confirmed the configuration of 2α,3α-diol for the compound.

The ^1H NMR spectrum showed that the glycosyl group was linked with the aglycone in the β-configuration by the anomeric proton at δ6.25 (d, J=8.4 Hz). Five methyl signals were seen at δ 1.02, 1.20, 1.38, 1.50, 1.58 as singlets, a methyl signal at δ 1.02 as a doublet and one olefinic proton signal at δ 5.45 (1H, br s, H-12). Thus, the structure of the compound was elucidated as 2α,3α,19α,24 tetrahydroxyurs-12-en-28-oic acid-28-O-β-glucopyranosyl ester (Fig.1). This compound was previously isolated from *R. xanthocarpus* (Zhi,1998). All spectral data are confirmed with the literature.

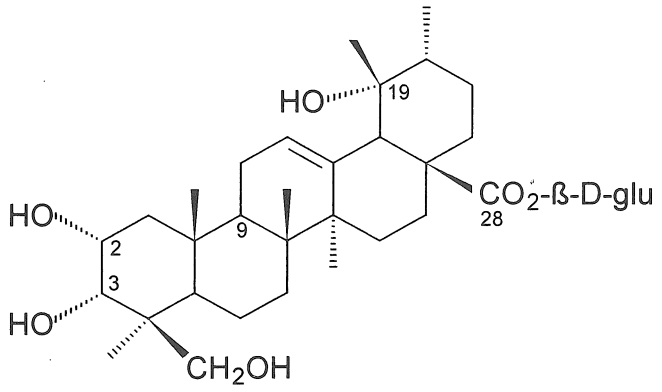

Figure 1

Table 1: ^{13}C NMR and DEPT data

C	^{13}C	DEPT
1	43.1, t	CH_2
2	65.4, d	CH
3	77.6, d	CH
4	44.8, s	C
5	49.3, d	CH
6	18.7, t	CH_2
7	34.3, t	CH_2
8	42.2, s	C
9	46.4, d	CH
10	37.5, s	C
11	25.4, t	CH_2
12	126.8, d	CH
13	134.8, s	C
14	43.1, s	C
15	30.4, t	CH_2
16	24.7, t	CH_2
17	47.8, s	C
18	55.5, d	CH
19	73.2, s	C
20	43.0, d	CH
21	25.6, t	CH_2
22	35.8, t	CH_2
23	23.4, q	CH_3
24	64.2, t	CH_2
25	14.2, q	CH_3
26	15.0, q	CH_3
27	23.4, q	CH_3
28	177.4, s	C
29	27.2, q	CH_3
30	15.8, q	CH_3
Glu-1	97.8, d	CH
2	73.7, d	CH
3	79.2, d	CH
4	72.3, d	CH
5	77.8, d	CH
6	60.3, t	CH_2

Özet

Rubus türleri analjezik özellikleri ve antidiyabetik aktiviteleri dolayısıyla geleneksel halk tıbbında kullanılmaktadırlar. Bu çalışmada, analjezik aktivite gösterebilecek triterpenik yapıda bir madde izole edilmiştir. Bileşik 2α,3α,19α,24 tetrahidroksiurs-12-en-28-oik asid-28-O-β-glukopiranizil ester'dir.

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