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LONG-CHAIN FATTY ACID ESTERS OF 3α-HYDROXY-LUP-20(29)-ENE-23,28-DIOIC ACID AND OTHER TRITERPENOID CONSTITUENTS FROM THE BARK OF *SCHEFFLERA OCTOPHYLLA**

JURGEN SCHMIDT, VŨ VIÊT NAM†, MANFRED LISCHEWSKI, HOANG VAN PHIÊT†, CHRISTINE KUHNT and GÜNTER ADAM

Institute for Plant Biochemistry, Academy of Sciences of the GDR, Halle/S., G.D.R.; †Institute of Chemistry, National Research Centre of the SRV, Hanoi, S.R.V

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Key Word Index—*Schefflera octophylla*, Araliaceae; triterpene fatty acid esters; oleanolic acid; 3α-hydroxy-lup-20(29)-ene-23,28-dioic acid.

Abstract—From the bark of *Schefflera octophylla* was isolated a series of triterpene fatty acid esters with the carbon numbers 16–21 and 23–29 in the fatty acid part. Oleanolic acid and 3α-hydroxy-lup-20(29)-ene-23,28-dioic acid were also identified.

INTRODUCTION

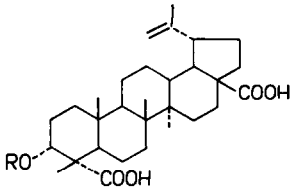
Recently, we described the structure elucidation of two new pentacyclic triterpenes of the lupane type isolated from the leaves of *Schefflera octophylla* (Araliaceae) [1, 2]. We now wish to report the isolation and identification of the triterpene constituents from the bark of the same plant.

RESULTS AND DISCUSSION

Dried powdered bark was extracted with petrol and ethanol. A yellow precipitate from the ethanolic extraction was separated and purified by preparative TLC on silica gel (petrol–chloroform, 1 : 1). Alkaline hydrolysis of the purified product **2** yielded 3α-hydroxy-lup-20(29)-ene-23,28-dioic acid (**1**) and a complex long-chain fatty acid mixture which was esterified with diazomethane and investigated by GC and GC/MS. Fourteen fatty acids with the carbon numbers 16–21 and 23–29 were identified (Table 1). The fatty acids C24 : 0 and C25 : 0 represent the main components. It is interesting to note that all fatty acids with an uneven carbon number (17, 19, . . . 29) are branched with a methyl at C-2 as shown by the base peak in the mass spectra. The corresponding ion originates by a McLafferty-rearrangement ($[R'–CH=C(OH)–OMe]^+$; R′ = H: *m/z* 74, R′ = Me: *m/z* 88). Furthermore, the mass spectra did not indicate any other branched positions [3].

The residue from the ethanolic extract was separated by CC on silica gel with chloroform–ethyl acetate gradient elution and checked by TLC-monitoring. Compound **1** [1] was identified by spectroscopic data and comparison with an authentic sample. A triterpene glycoside mixture was eluted with methanol. After acidic hydrolysis and CC on silica gel under the conditions given above oleanolic acid [4] and **1** were identified.

Further triterpenoid constituents from the leaves of this plant are betulinic acid and epibetulinic acid as identified after acidic hydrolysis by their spectroscopic data and by comparison with authentic samples (unpublished observations).



1 R = H

2 R = fatty acid moiety

Table 1 GC analysis of the fatty acids (as methyl esters) derived from **2**

Fatty acid	% Composition
16·0	2.0
17.0 (2-Me)	0.7
18:0	0.3
19:0 (2-Me)	0.3
20.0	0.8
21:0 (2-Me)	0 8
23:0 (2-Me)	3.5
24:0	50.6
25:0 (2-Me)	29.1
26:0 (2-Me)	0.2
26:0	4.8
27:0 (2-Me)	5.2
28:0	0.4
29:0 (2-Me)	1.3

*Part 10 in the series "Natural Products from Vietnamese Plants". For part 9 see Ngoc, Ph.H., Kutschabsky, L., Phuong, N. M and Adam, G., *Planta Med.* (in press)

EXPERIMENTAL

Schefflera octophylla (Lour.) Harms was identified by Dr. Ph. N. Nguyen, Institute of Biology, National Research Centre of the SRV, Hanoi, and a voucher specimen is kept there.

Extraction and separation. Air-dried powdered bark (190 g) of *S octophylla* was extracted with petrol in a Soxhlet for 8 hr. Further extraction with EtOH (24 hr) yielded 10.5 g ethanolic extract. During the ethanolic extraction a yellow precipitate (103 mg) settled down which was separated The precipitate was purified by preparative TLC (petrol–$CHCl_3$, 1:1) yielding **2**.

Alkaline hydrolysis of **2**. Compound **2** (100 mg) was boiled with 10 ml 5% KOH in MeOH for 14 hr. The soln was evaporated to dryness and the residue, after addition of H_2O, extracted with $CHCl_3$ to remove the triterpene. The triterpene was identified by its MS, ^{1}H NMR, IR and TLC data, as well as by direct comparison, as 3α-hydroxy-lup-20(29)-ene-23,28-dioic acid (**1**) [1]. The alkaline soln was acidified with HCl and extracted with EtOAc to remove the fatty acids The soln was evaporated to dryness and esterified with CH_2N_2 in C_6H_6. The methyl esters were investigated by GC (steel column 2.0 m × 4 mm, 3% SE 30, gaschrom Q 125–150 μm, temperature programming 190–280°, 2°/min, N_2 at 21 ml/min) and GC/MS (3% SE 30, temperature programming 205–250°, 2°/min, He at 20 ml/min, 80 eV). The residue from the ethanolic extract was separated by CC on silica gel with $CHCl_3$–EtOAc gradient elution and TLC-monitoring ($CHCl_3$–EtOAc–AcOH, 9:1:0.5). Compound **1** [1] (0.7%) was identified by ^{1}H NMR, MS, IR, $[\alpha]_D$ and TLC data, as well as direct comparison with an authentic sample. Using MeOH as eluent a triterpene glycoside mixture (2.9 g) was isolated.

Acidic hydrolysis of the triterpene glycoside mixture. The triterpene glycoside mixture (2.9 g) was boiled with 50 ml 1 N HCl in MeOH for 4 hr. The soln was evaporated to dryness and the residue, after addition of H_2O, extracted with EtOAc. The EtOAc-phase was dried over Na_2SO_4 and concd. The triterpene components were separated by CC on silica gel with gradient elution ($CHCl_3$–EtOAc) and TLC monitoring ($CHCl_3$–EtOAc–AcOH, 9:1:0.5). Oleanolic acid [4], (0.1%) and 3α-hydroxy-lup-20(29)-ene-23,28-dioic acid [1] (0.2%) were identified by MS, ^{1}H NMR, IR and TLC data, as well as by comparison with authentic samples.

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TRITERPENE CONSTITUENTS OF *CALTHA PALUSTRIS**

PRABHA BHANDARI and R. P. RASTOGI

Central Drug Research Institute, Lucknow, India

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Key Word Index—*Caltha palustris*; Ranunculaceae; palustrolide; 3β,23-dihydroxylupan-13β→28-lactone; hederagenin; 16,17-dihydroxykauran-19-oic acid; hederagenic acid.

Abstract—The structure of a new triterpene lactone, palustrolide, has been elucidated as 3β,23-dihydroxylupan-13β→28 lactone on the basis of physico-chemical studies. In addition, sitosterol, its glucoside, hederagenin, 16,17-dihydroxykauran-19-oic acid and hederagenic acid have been characterized.

INTRODUCTION

In a preceding paper [1] we described the isolation and structure elucidation of two new 24-norlupane lactones along with the isolation of the chemical constituents from the chloroform-soluble fraction of the alcoholic extract of *Caltha palustris*. The present paper describes the characterization of substance B as sitosterol, substance G as hederagenic acid, substance J as hederagenin, substance K as 16,17-dihydroxykauran-19-oic acid, substance L as sitosterol-β-D-glucoside and the structure elucidation of a new triterpene lactone (substance H), named as palustrolide.

*CDRI Communication No. 3447.

RESULTS AND DISCUSSION

Palustrolide, $C_{30}H_{48}O_4$, gave positive colour tests for a triterpenoid. The IR absorptions at 3300 and 1760 cm^{-1} suggested it to be a hydroxy-γ-lactone. The acetylation of palustrolide afforded a diacetate whose ^{1}H NMR spectrum displayed signals for six methyls in the region δ0.78–1.20, two acetoxymethyls at 1.95 and 1.98 and the corresponding carbinolic protons as an AB*q* (J = 12 Hz) at 3.72 for a primary acetoxymethylene group and the signal for a methine geminal to an acetoxy group appeared as a *dd* (J = 10, 6 Hz) at δ4.7 which confirmed one primary and one secondary hydroxyl group in the molecule.

Its mass spectrum showed major fragments at *m/z* 236, 223, 218, 205 (corresponding ions at *m/z* 320, 307, 260 and