Asian Journal of Chemistry Vol. 19, No. 2 (2007), 1535-1543

Sativalanosteronyl Glycoside and Oryzatriacontolide Constituents from the Hulls of *Oryza sativa*

ILL-MIN CHUNG, MOHD ALI†, SE-CHUL CHUN, OH-KYU LEE and ATEEQUE AHMAD*
Department of Applied Life Science, Konkuk University, Seoul, 143-701, South Korea
Fax: (82)(2)4467856; Tel: (82)(2)4503730; E-mail: aahmadc@yahoo.com

Three compounds sativalanosteronyl glycoside identified as lanost-5, 7, 9(11)-triene-3β-ol-24-one-3β-D-glucopyranoside (**1**), oryzatriacontolide characterized as n-triacontan-1, 5-olide (**2**), and tritriaconta-4, 12-diene (**3**) along with known compound octacosanoic acid were isolated and identified from the rice hulls of *Oryza sativa*. Their structures were elucidated with the help of 500 MHz NMR using 1D and 2D spectral methods *viz*., ^{1}H NMR, ^{13}C NMR, HMBC, ^{1}H-^{1}H COSY and HETCOR aided by EI MS, FAB MS and IR.

Key Words: *Oryza sativa*, Poaceae, Rice hull composition, Sativalanosteronyl glycoside, Oryzatriacontolide, Tritriaconta-4, 12-Diene.

INTRODUCTION

In continuation of our study on rice hulls of *Oryza sativa* constituents, we reported new and known compounds of inhibitory and cytotoxic activities[1-6]. This paper deals with the isolation and structural elucidation of three new compounds (**1-3**) and one known octacosanoic acid (**4**) on the basis of spectral methods, *viz.*, ^{1}H NMR, ^{13}C NMR, HMBC, COSY and HETCOR aided by EIMS, FABMS and IR. The complete spectral data of compound (**4**) are also reported in the experimental section. For all the molecules studied, relative configurations were suggested on the basis of biogenetic speculations.

EXPERIMENTAL

Melting points were determined on Electrochemical Eng. melting point apparatus and TLC was carried out on precoated silica gel plates (Merck). Spots were detected under UV (254 and 366 nm) before and after dipping in chamber with 1% vanillin sulfuric acid (ethanol solution). TLC glass plates used precoated silica gel (Merck), layer thickness 0.25 mm and column chromatography was carried out on silica gel (70-230 mesh, Merck) and Lichroprep RP-18 (ODS silica gel, Merck). Optical rotation was

†Faculty of Pharmacy, Hamdard University, New Delhi-110062, India.

measured on a AA-10 model polarimeter. Both ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra were obtained with a Bruker Avance (DRX-500) spectrometer operating at 500 and 125 MHz, respectively. NMR spectra were obtained in deuterated chloroform, methanol and pyridine using tetramethylsilane (TMS) as internal standard, with chemical shifts expressed in parts per million (δ) and coupling constants (*J*) in hertz. EI-Mass spectra were recorded on a Jeol JMS-SX 102 A spectrometer and FABMS on a Jeol JMS-AX 505 WA. IR spectra were recorded on a Thermo Mattson 60-AR spectrophotometer.

Plant Material: The hulls of *O. sativa* was collected from Konkuk university (experimental farm) Seoul, South Korea in October, 2002. The voucher specimen (No. KKU 96, HOCHOKJINDO) was deposited in the herbarium of our department.

Extraction and Isolation: The dried hulls of *O. sativa* (10 kg) were immersed in MeOH for a week at room temperature, filtered and the extract was concentrated in vacuum to give a semi-solid mass (150 g). It was suspended in H_2O and extracted with EtOAc and *n*-BuOH successively. The EtOAc extract (35 g) was subjected to normal phase column chromatography over silica gel column (70-230 mesh, 800 g 5.5 x 90 cm), yielded 40 fractions with the following eluants (each fraction 500 mL): fraction 1 in hexane, fractions 2-5 in hexane/EtOAc (9:1), fractions 6-11 in hexane/EtOAc (4:1), fractions 12-15 in hexane/EtOAc (7:3), fractions 16-20 in hexane/EtOAc (1:1), fractions 21-22 in EtOAc, fractions 23-28 in EtOAc/MeOH (9.5:0.5), fractions 29-32 in EtOAc/MeOH (9:1), fractions 33-36 in EtOAc/MeOH (7:3), fractions 37-40 in MeOH. fraction 1 (500 mg) with further CC and TLC over silica gel with n-hexane/EtOAc to yield one pure compound: hentriacontane (50 mg). Fractions 2-5 are same on TLC, after mixing (1.2 g) which was further CC and TLC over silica gel by using CH_2Cl_2, CH_2Cl_2/MeOH (99.8:0.2, 99.6:0.4, 99.4:0.6, 99.2:0.8, 99:1) as eluants to yield six fractions, 1-tetratriacontanol (50 mg) from intial fraction 1. The fraction 6 (2.8 g) was crystallized and after purification through column chromatography with hexane/EtOAc obtained β-sitosterol (200 mg) and confirmed by comparison to an authentic sample from Sigma. The fraction 11 (2.1 g) which was further purified by CC over silica gel with CH_2Cl_2/MeOH (99.8:0.2, 99.6:0.4, 99.4:0.6, 99.2:0.8, 99:1) afforded two pure compounds momilactone A (80 mg), momilactone B (70 mg). Fraction 12 (3.4 g) after CC over silica gel by using CH_2Cl_2/MeOH (99:1, 98:2, 97:3, 96:4, 95:5) as eluants to yield five fractions. Two compounds were obtained from fraction 1, identified as tricin (10 mg) and β-sitosterol-3-O-β-D-glucuronoside. The other impure fractions 2, 4 and 5 after mixing were rechromatographed over Lichroprep RP-18 (ODS silica gel) using sequential mixtures of H_2O/MeOH as eluants (elution order 80, 60, 40 20,

10% aqueous methanol, 100% methanol) to yield six fractions and obtained one new compound sativalanosteronyl glycoside (**1**, 25 mg) and known 3, 7- dimethyl-n-octan-1-yl benzoate (15 mg). The fraction 23 was subjected to silica gel with chromatography eluting with $CHCl_3$/MeOH to yield one pure compound β-sitosterol-3-O-β-D-glucopyranoside (50 mg).

The *n*-BuOH extract (19 g) was separated by column chromatography over silica gel with $CHCl_3$/MeOH mixtures of increasing polarity (9:1 to 1:1) to yield ten fractions. β-Sitosterol-3-O-β-D-glucoside (40 mg) was obtained from fraction 1 after purification on a silica gel column with $CHCl_3$/MeOH (9:1). Fractions 2 in $CHCl_3$ /MeOH (4:1) was separated on silica gel column with $CHCl_3$ /MeOH afforded *n*-triacontan-1,5-olide (**2**), (10 mg). Fractions 3-4 in $CHCl_3$/MeOH (7:3) was separated on an ODS silica gel (Lichroprep RP-18) with a H_2O/MeOH (8:2 to 2:8) gradient to yield two compounds as *n*-tritriacont-4,12-diene (**3**, 15 mg), octacosanoic acid (**4**, 12 mg). The several fractions of butanol extract contains sugar molecules on the basis of NMR, therefore is no need to analyze.

Lanost-5, 7, 9 (11)-triene-3β-ol-24-one-3β-D-glucopyranoside (1): Colourless gum; $[\alpha]_D^{20}$ +34.2º (MeOH; *c* 0.34); IR (KBr, ν, cm^{-1}): 3420, 3405, 3390, 2930, 2895, 1713, 1650, 1610, 1485, 1457, 1360, 1268, 1180, 1110, 1015, 810, 756 cm^{-1}; ^1H NMR (500 MHz, MeOD) and ^{13}C NMR (125 MHz, MeOD) (Table-1); EI MS m/z (rel. int.): 600 $[M]^+$ ($C_{36}H_{56}O_7$) (10.1), 584 (6.9), 501 (8.6), 339 (12.7), 293 (34.1), 286 (15.6), 222 (20.1), 168 (33.6), 154 (37.2), 147 (100), 134 (43.8), 127 (31.5), 71 (35.6), 55 (74.2); FAB MS (positive mode) m/z 601 $[M+H]^+$; FAB MS (negative mode) m/z 559 $[M - H]^-$.

Acid hydrolysis of 1: A solution of **1** (10 mg in 2N HCl-MeOH (2 mL) was refluxed for 6 h. After neutralization with NaOH, water layer was extracted with diethyl ether and concentrated under reduced pressure to afford the aglycone (lanostane-type triterpene) PC of the water extract layer with authentic sugar was identified as glucose.

***n*-Triacontan-1, 5-olide (2):** Colourless powder; mp 160-162ºC; $[\alpha]_D^{20}$ + 1.32º (MeOH; *c* 0.21); IR (KBr, ν_{max}, cm^{-1}): 2918, 2845, 1740, 1579, 1465, 1437, 1120, 1105, 1080, 1015, 1005, 735, 703 cm^{-1}; ^1H NMR (500 MHz, MeOD;): δ 3.92 (1H, dd, *J* = 6.5, 6.5 Hz, H_2-5α), 3.89 (1H, dd, *J* = 6.5, 6.5 Hz, H_2-5b), 2.13 (1H, br m, $w_{1/2}$ = 6.0 Hz, H-2β), 1.79 (1H, m, H_2 - 4a), 1.75 (1H, m, H_2-4b), 1.55 (2H, m, H_2-4), 1.36 (4H, br s, 2 x CH_2), 1.33 (20 H, br s, 10 x CH_2), 1.30 (12H, br s, 6 x CH_2), 1.28 (8H, br s, 4 x CH_2), 0.88 (3H, t, *J* = 7.0 Hz, Me-30); ^{13}C-NMR: (125 MHz, MeOD): δ 176.21 (C-1), 60.11 (C-5), 43.4 (C-2), 31.79 (C-3), 30.09 (C-4), 28.01 (20 x CH_2), 27.89 (CH_2), 27.57 (CH_2), 24.52 (CH_2), 20.90 (CH_2), 12.24 (Me-30); EIMS m/z (rel.int): 450 $[M]^+$ ($C_{30}H_{58}O_2$) (2.3), 428 (6.1), 421 (32.1), 393 (6.6), 379 (2.4), 365 (6.7), 351 (3.0), 337 (3.9), 320 (4.1), 306 (4.4),

292 (4.6), 264 (5.2), 237 (5.4), 223 (6.0), 209 (6.9), 195 (8.0), 181 (9.9), 167 (12.3), 153 (3.4), 125 (35.3), 111 (58.8), 97 (100), 85 (41.7), 83 (92.3), 71 (59.0), 69 (65.3), 57 (89.6); FABMS m/z. 451 $[M+H]^+$.

TABLE-1
^1H AND ^{13}C NMR ASSIGNMENTS OF **1**

Position	^1H NMR (MeOD, 500MHz)		^{13}C NMR (MeOD, 125 MHz)
	α	β	
1	1.42 m	2.04 m	34.6
2	1.83 m	2.01 m	32.5
3	3.68 dd (11.5, 3.7)	-	76.0
4	-	-	40.7
5	-	-	147.1
6	7.38 d (11.9)	-	128.1
7	7.18 d (11.9)	-	127.7
8	-	-	131.9
9	-	-	130.7
10	-	-	43.3
11	6.82 m	-	125.7
12	2.41 dd (11.2, 7.1)	2.19 br s	38.8
13	-	-	52.9
14	-	-	56.9
15	1.01 m	1.62 m	31.0
16	1.67 m	1.35 m	36.8
17	1.60 m	-	56.7
18	0.69 br s	-	11.7
19	1.29 br s	-	19.4
20	1.70 m	-	36.4
21	0.93 d (3.9)	-	19.2
22	1.73 m	1.73 m	32.5
23	2.41 d (5.2)	2.39 d (5.2)	46.5
24	-	-	203.6
25	2.34 m	-	30.0
26	0.85 d (4.7)	-	21.8
27	0.87 d (6.7)	-	24.7
28	1.30 br s	-	23.5
29	1.16 br s	-	12.5
30	0.91 br s	-	14.4
1'	5.13 d (7.7)	-	104.6
2'	3.91 dd (5.9, 7.7)	-	76.4
3'	3.87 m	-	76.0
4'	3.56 m	-	68.9
5'	4.25 m	-	79.4
6'	3.35 d (11.5)	3.33 d (11.5)	62.1

Coupling constant (*J*) values in Hertz (Hz) are in parenthesis

***n*-Tritriaconta-4, 12-diene (3):** Yellow semi-solid; m.f. $C_{33}H_{65}$ IR (KBr, ν_{max}, cm^{-1}): 2924, 2854, 1590, 1460, 1365, 1215, 1130, 1090, 725 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 5.37 (1H, br s, H - 4), 5.33 (2H, br s, H - 5, H-12), 5.30 (1H, br s, H -13), 2.81 (1H, br m, H_2 - 6a), 2.76 (1H, br s, H_2 - 6b), 2.29 (2 H, br s, H_2-3), 2.03 (2 H, br s , H_2 -11), 2.01 (2 H, br s, H_2 -14), 1.57 (2 H, br m, H_2 -7), 1.28 (10 H, br s, 5 x CH_2), 1.25 (26 H, br s, 13 x CH_2), 1.22 (8 H, br s, 4 x CH_2), 0.87 (6 H, t, *J* = 6.0 Hz, Me-1, Me - 33); ^{13}C-NMR ($CDCl_3$; 125 MHz): δ 130.44 (C - 5), 130.15 (C -12), 128.23 (C - 4), 128.11 (C -13), 32.02 (C - 6), 30.41 (C -11), 29.92 (C -14), 29.57 (12 x CH_2), 29.21 (C - 3), 28.96 (CH_2), 28.20 (CH_2), 27.45 (CH_2), 27.41 (CH_2), 26.93 (CH_2), 25.85 (CH_2), 25.74 (CH_2), 23.39 (2 x CH_2), 23.30 (CH_2), 15.98 (Me-1), 14.32 (Me-33); EIMS m/z (rel.int.): 460 $[M]^+$ ($C_{33}H_{64}$) (15.5), 419 (2.2), 391 (5.3), 307 (25.9), 289 (12.4), 179 (2.5), 167 (5.6), 153 (100), 149 (10.5), 139 (12.5), 136 (64.9), 127 (3.5), 125 (3.7), 113 (3.02), 111 (2.5), 99 (2.0), 97 (3.7), 85 (3.9), 83 (6.0), 71 (10.6), 69 (10.8), 57 (13.4), 55 (17.7), 43 (12.7). FABMS; m/z 461 $[M + H]^+$.

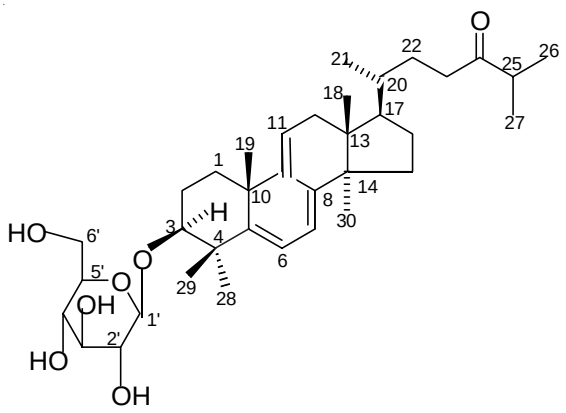
Octacosanoic acid (4): m.f. $C_{28}H_{57}O_2$; IR (KBr, ν_{max}, cm^{-1}): 3350, 2918, 2849, 1705, 1464, 1455, 1425, 725 cm^{-1}; ^1H NMR (500 MHz, C_5D_5N): δ 2.56 (1H,d, *J* = 7.3 Hz, H_2-a), 2.53 (1H, d, *J* = 7.4 Hz, H_2-2b), 2.13 (2H, m, H_2-3), 1.84 (2H, m, H_2 - 4), 1.81 (2H, m, H_2 - 5), 1.42 (2H, m, H_2 - 6), 1.36 (18 H, br s, 9 x CH_2), 1.28 (24 H, br s, 12 x CH_2), 0.88 (3H, t, *J* = 6.2 Hz, Me-28); ^{13}C-NMR (C_5D_5N, 125 MHz): δ 176.4 (COOH-1), 36.7 (CH_2), 35.3 (CH_2), 32.5 (CH_2), 30.4 (10 x CH_2), 30.3 (6 x CH_2), 30.2 (CH_2), 30.1 (CH_2), 30.0 (CH_2), 29.91 (CH_2), 28.0 (CH_2), 26.6 (CH_2), 26.1 (CH_2), 23.3 (CH_2), 14.7 (Me -28); EIMS m/z (rel.int): 424 $[M]^+$ ($C_{28}H_{56}O_2$) (1.7), 396 (15.1), 381 (22.1), 367 (100), 353 (72.8), 339 (78.5), 325 (17.5), 311 (15.6), 297 (15.3), 283 (11.0), 269 (13.3), 255 (10.4), 241 (15.4), 227 (10.8), 213 (10.1), 199 (7.8), 185 (23.8), 171 (13.6), 157 (6.6), 143 (8.5), 129 (57.2), 115 (17.4), 113 (12.9), 97 (41.3), 85 (35.4), 83 (44.3), 73 (70.1), 71 (48.9), 57 (79.5); FABMS: m/z 425 $[M+H]^+$.

RESULTS AND DISCUSSION

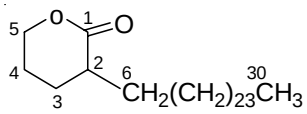
Sativalanosteronyl glycoside (**1**) was obtained as a colourless gum from methanol extract. It gave positive tests of triterpene glycosides. Its IR spectrum showed characteristic absorption bands for hydroxyl groups (3420, 3405, 3390 cm^{-1}), carbonyl group (1713 cm^{-1}) and unsaturation (1650 cm^{-1}). A molecular formula of $C_{36}H_{56}O_7$ for compound **1** was established from its FAB MS and NMR data. The fragmentation pattern of aglycone part 1 are shown in Fig. 2.

The ^1H NMR spectrum of **1** displayed two one-proton doublets at δ 7.38 (*J* = 11.9 Hz) and 7.18 (*J* =11.9 Hz) assigned to vinylic H-6 and H-7, respectively. A one-proton multiplets at δ 6.82 was attributed to olefinic H-

11. A one-proton double doublet at δ 3.68 (J = 11.5, 3.7 Hz), was accounted to carbinol H-3α. Five three-proton broad signals at δ 0.69, 1.29, 1.30, 1.16 and 0.91 were ascribed to tertiary C-18, C-19, C-28, C-29 and C-30 methyl protons all attached to saturated carbons. Two three-proton doublets at δ 0.85 (*J* = 4.75 Hz) and 0.87 (*J* = 6.7 Hz), were associated with correspondingly to C-26 and C-27 secondary methyl protons. A one-proton doublet at δ 5.13 (J = 7.7 Hz) was assigned to anomeric H-1'. A one proton doublet doublet at δ 3.91 (J = 5.9, 7.7 Hz) and three multiplets at δ 3.87, 3.56, 4.25, were ascribed to carbinol H-3', H-4' and H-5', respectively. Two one-proton doublets at δ 3.55 (J = 11.5 Hz) and 3.33 (J = 11.5 Hz) were accounted to H_2-6'.



1



2

$$CH_3(CH_2)_2CH{=}CH(CH_2)_6CH{=}CH(CH_2)_{19}CH_3$$

3

$$CH_3(CH_2)_{24}CH_2CH_2COOH$$

4

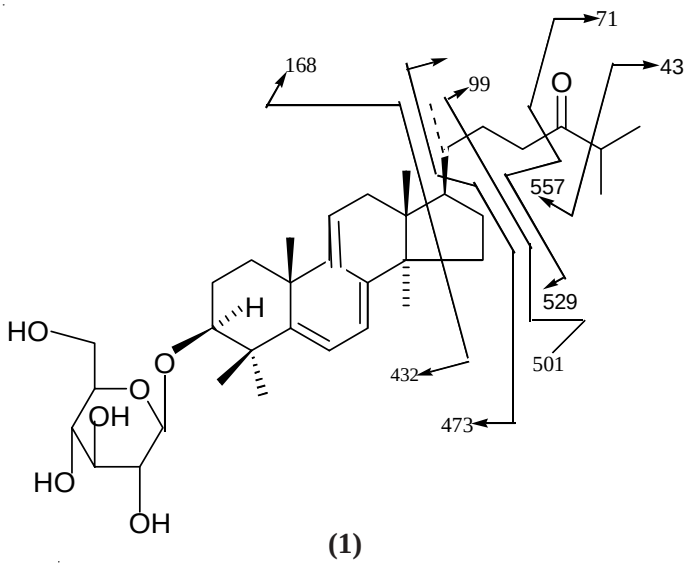
Fig. 1. Chemical structures of sativalanosteronyl glycoside (**1**), oryzatriacontolide (**2**) tritriaconta-4,12-diene (**3**) and octacosanoic acid (**4**)

The ^{13}C NMR spectrum of **1** exhibited vinylic carbon at δ 147.1 (C-5), 128.1 (C-6), 127.7 (C-7), 131.93 (C-8), 130.7 (C-9) and 125.7 (C-11). The C-24 carbonyl carbon resonated at δ 203.6. The C-3 carbinol carbon and

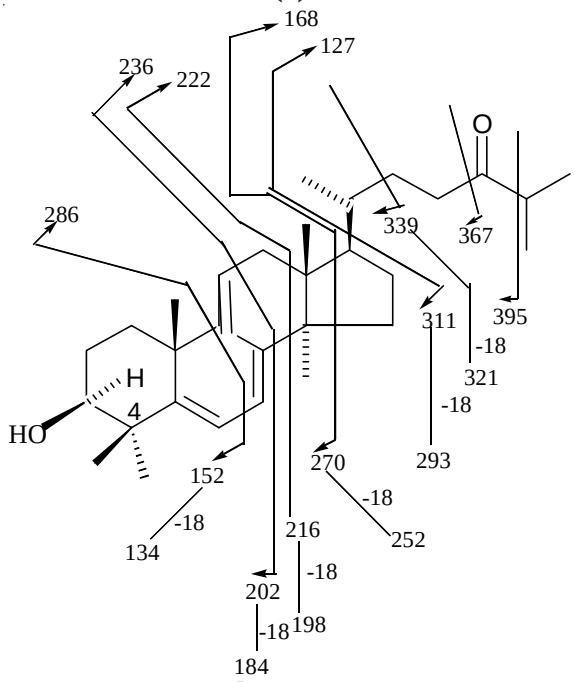
anomeric carbons appeared at δ 76.0 (C-3), 104.6 (C-1'). The remaining carbons signals of the sugar moieties appeared between δ 79.4 - 62.1 (Table-1). The ^{1}H and ^{13}C-NMR signals of **1** were compared with the other related lanostane type triterpenes[7]. The ^{1}H-^{13}C NMR spectrum of **1** showed correlation of C-6 with H-7, H_3-28; C-11 with H_2-12; C-3 with H_2-2, H_2-1 and H-1'; C-1' with H-2', H-3' and H-4'. The ^{1}H-^{1}H COSY spectrum exhibited correlation of H-1' with H-2', H-3' and H-3; H-3 with H_2-2; H-6 with H-7 and H-11with H_2-12. In HMBC spectrum, the anomeric proton signal at δ 5.13 correlated with signals at δ 3.68 (C-3) which suggested that the glucose unit was connected to the C-3 hydroxyl group in **1**. Acid hydrolysis of **1** yielded glucose sugar which was identified by comparison with the aunthetic sample. On the basis of these evidences the structure of **1** has been elucidated as lanost-5,7,9(11)-triene-3β-ol-24-one-3β-D-glucopyranoside.

Oryzatriacontolide (**2**) (m.f. $C_{30}H_{58}O_2$) was obtained as a colourless amorphous mass from butanol extract of the rice hull. Its IR spectrum displayed characteristic absorption bands for δ lactone (1740 cm^{-1}), and long aliphatic chain (735, 703 cm^{-1}). Its mass spectrum showed a molecular ion peak at m/z 450 corresponding to δ-lactone having C_{25} long aliphatic side chain. Its indicated two degrees of unsaturation adjustable in the lactone ring system. The generation of base peak at m/z 97 due to expulsion of the side chain m/z 351 from the molecular ion peak indicated that C_{25} side chain was attached to the lactone ring.

The ^{1}H NMR spectrum of **2** showed two one-proton double doublets at δ 3.92 (*J* = 6.5, 6.5 Hz) and 3.89 (*J* = 6.5, 6.5 Hz), assigned to oxygenated C-5 methylene protons. A one-proton broad multiplet at δ 2.13 (J = 6.0 Hz) was ascribed to β-oriented H-2 adjacent to the carbonyl group. A three-proton triplet at δ 0.88 (*J* = 7.0 Hz), was attributed to terminal primary methyl protons. The remaining methylene protons appeared between δ 1.79 - 1.28. The ^{13}C NMR spectrum of **2** displayed signals for carbonyl carbon (δ 176.2), oxygenated methylene carbon (δ 60.1), methyl carbon (δ 12.2), and methylene carbons between δ 31.7 - 20.9. The absence of ^{1}H NMR signals beyond δ 3.92 and ^{13}C NMR signals between δ 176.21-60.11 ruled out the location of any vinylic carbon in the molecule. In ^{1}H-^{13}C HETCOR NMR spectrum correlation of H_2-5 with H_2-4, H_2-3 and H-4 were observed. The HSQC spectrum showed correlation of H_2-5 with H-2. In ^{1}H-^{1}H COSY spectrum, a correlation of H_2-5 was observed with H_2-4a. On the basis of these informations the structure of **2** has been elucidated as *n*-triacontan-1, 5-olide.



(1)



Aglycone part

Fig.2 Fragmentation of pattern of 1

n-Tritriaconta-4,12-diene (**3**) was obtained from the butanol extract of rice hull. The compound decolourized bromine water and did not react with any acetylating, methylation or oxidizing reagents indicating that the compound is an alkene. Its IR spectrum showed characteristic absorption bands for unsaturation (1590 cm^{-1}) and long aliphatic chain (725 cm^{-1}). Its

mass spectrum displayed a molecular ion peak at m/z 460 corresponding to the molecular formula of an alkene, $C_{33}H_{64}$. It indicated two double bond equivalents which were adjusted in two vinylic bonds. The mass spectrum gave C_nH_{2n+1}, C_nH_{2n} and C_nH_{2n-1} ion fragments in higher abundance for lower fragments. Most of the fragments were separated by 14 mass units and decreased in abundance with increasing molecular weight of long straight chain hydrocarbons. The absence of $[M^+$-Me] ion suggested its straight nature whereas the presence of $[M^+$+1] ion arose due to its unsymmetrical nature. The generation of prominent ion fragments at m/z 43 [C_3 - C_4 fission]$^+$, 69, 391 [C_5 - C_6 fission]$^+$, 153, 307 [C_{11} - C_{12} fission]$^+$ and 179 [C_{13} - C_{14} fission]$^+$, indicated the location of the vinylic bonds at C- 4 and C-12 positions.

The ^{1}H-NMR spectrum of **3** exhibited two one proton broad signals at δ 5.37 and 5.30 assigned to vinylic H- 4 and H-13, respectively. A two-proton broad signal at δ 5.33 was ascribed to vinylic H-5 and H-12. The ^{1}H signals at δ 2.81 (1H, br m), 2.76 (1H, br s), 2.29 (2H, br s), 2.03 (2H, br s) and 2.01 (2H, br s) were associated with the methylene protons adjacent to the vinylic carbons. Two six-proton triplets at δ 0.87 (J = 6.0 Hz) were accounted to C-1 and C-33 primary methyl protons, respectively. The remaining methylene protons resonated at δ 1.57 (2H), 1.28 (10 H), 1.25 (26 H) and 1.22 (8 H). The ^{13}C-NMR spectrum displayed signals for vinylic carbons at δ 130.44 (C-5), 130.15 (C-12), 128.23 (C-4), and 128.11 (C-13), methyl carbons at δ 15.98 (C-1) and 14.32 (C-33) and the remaining carbons signals between δ 34.33 - 22.80. On the basis of these informations the structure has been elucidated as *n*-tritriacont-4, 12-diene.

ACKNOWLEDGEMENTS

One of the authors (A. Ahmad) is grateful to Korean Federation of Science and Technology Societies (KOFST) for the award of fellowship as a Senior researcher in Konkuk University, Seoul, South Korea.

REFERENCES

1. I.M. Chung, S.J. Hahn and A. Ahmad, *J. Chem. Eco.*, **31**, 1339 (2005)
2. I.M. Chung, M. Ali, S.J. Hahn, N.A. Siddique and A. Ahmad, *Chem. Nat. Comp.*, **41**, 182 (2005).
3. I.M. Chung, M. Ali and A. Ahmad, *Asian J.Chem.*, **17**, 2467 (2005).
4. I.M. Chung, M. Ali and A. Ahmad, *Asian J. Chem.*, **17**, 2616 (2005).
5. I.M. Chung, M. Ali, A. Ahmad, J.D. Lim, C.Y. Yu and J.S. Kim, *Phytochem. Anal.*, **17**, 36 (2006).
6. I.M. Chung, M. Ali, T.D. Khan, M.G. Choung, H.J. Park and A. Ahmad, *Bull. Korean Chem. Soc.*, **27**, 93 (2006).
7. M. Ali, Techniques in Terpenoid Identification, Birla publications, New Delhi, India, p. 352 (2001).

(*Received*: 24 March 2006; *Accepted*: 20 September 2006) AJC-5125