J. SOACHIM (1999) **008**, 123 - 128

STUDY OF TRITERPENOIDS FROM ALSTONIA BOOSEI

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(Reçu le 5 novembre 1997 - Revisé le 19 mai 1999)

Résume : L'extraction et caractérisation de deux composés : Lanosta-7,21-dien-3-one et l'acétate correspondant au composés de l'extrait éthéré de l'écorce de *L'Alstonia boonei*
Mots clés : *Alstonia boonei*, Triterpènes.

I - INTRODUCTION

The *alstonia boonei* De Wild (Apocynaceae), called Kla as local names in the west central region (Daloa/Bahigbeu) of Ivory Coast, are big long trees that grow throughout tropical west Africa.

The leaves and barks are extracted with alcohol, or water (hot or cold), to obtain medicinals that have extensive therapeutic applications including antimalarial, antihelminthic, antihypertension, antihepatitis and antidiabetic activities, etc. This paper deals with the isolation of compounds belonging to the triterpenoids acetate, ketone and alcohol classes, and those compounds were identified by comparison of their ^{1}H and ^{13}C NMR data with the published values [1-5]. Investigative studies on *the alstonia boonei* has been extensive and led to the isolation of alkaloids, most of which were α-amyrin palmitate, echitamine and echitamidine[1'].

II - RESULTS AND DISCUSSION

Diethyl ether soxhlet extraction of 200 g of dry barks afforded 450 mg of crude viscous yellow oils. The crude viscous yellow oils were subjected to chromatographix fractionator to yield pure three compounds ; **1**, **2** and **3** from *alstonia boonei*. The compound **3** is β-Amyrin[1] and Its mass spectrum had MS : m/z 468 ; m.p. 180°C ; IR ν 360 cm^{-1} (OH). The tabulated data of NMR ^{1}H and ^{13}C led to the identification of compound 3 as β-Armyrin.

The two first compounds were characterized and by comparison of NMR ^{1}H and ^{13}C data with those of the published values [2-5]. These two compounds, on the vacuum pump, changed from viscous oil to semi-solid aspect.

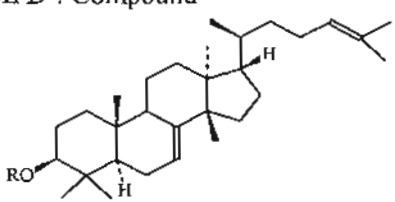
Compound **1** : MS : m/z 424, mp 93 - 103°C, IR ν (cm^{-1}) 1707 and 1456 (C = O) in position 3.Compound **2** MS : m/z 468 : NMR spectrum data indicated the presence of an acetate group comfirmed by δ 5.50 ppm (m, 1H, H-C-OAc) peak in ^{1}H NMR spectrum and δ 21.32 ppm (CH_3-CO) and δ 170.4 ppm ($\underline{C}H_3$-CO) in ^{13}C NMR spectrum. IR

ν (cm^{-1}) 1733 and 1451 (O=C-CH_3). The α and β-Amyrin acylated with fatty acides have interesting biological activities [6-11].

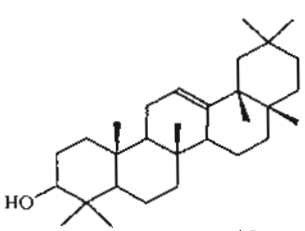
Table of Carbon-13 chemical shifts for the terpenoids compounds 1, 2 and 3 ($CDCl_3$ solution, ppm from internal tetramethylsilane).

Carbon n°	COMPD 1	COMPD 2	COMPD 3	Carbon n°	COMPD 1	COMPD 2	COMPD 3
1	35.90	35.70	38.73	16	30.90	30.90	28.16
2	27.82	27.90	27.29	17	50.23	50.23	33.77
3	218.60	81.40	78.97	18	15.80	15.80	59.19
4	38.56	38.56	38.84	19	18.30	18.30	39.73
5	50.18	50.18	55.29	20	37.02	37.02	39.65
6	19.20	19.20	19.45	21	18.80	18.80	31.29
7	125.30	105.70	32.80	22	36.68	36.68	41.59
8	134.40	152.60	38.78	23	25.54	25.54	28.16
9	50.50	50.50	47.74	24	125.30	124.70	15.65
10	37.20	37.20	37.00	25	130.80	140.02	15.65
11	21.10	21.10	23.57	26	17.60	17.60	16.95
12	27.04	27.04	121.80	27	25.80	25.80	28.75
13	43.40	43.40	145.10	28	24.30	24.30	17.48
14	50.16	50.16	41.80	29	28.02	28.02	17.48
15	31.10	31.10	26.22	30	15.40	15.40	21.36

COMPD : Compound



1 : RO = O ; lanosta-7,24 dien-3 one, cryst
2 R = $COCH_3$; lanosta-7,24 dien-3 acetate



compound 3

III - EXPERIMENTAL

Unless specified otherwise, all extractions or purification were carried out under an atmosphere of dry air and the samples were stocked under an atmosphere of nitrogen. All solvents were appropriately distilled an degassed prior to use. The TLC separations were performed in air by using florisil 100-200 mesh from Fischer Scientific Company or by using silica gel (60A, F254) on plates (Whatman, 0.25 mm). IR spectra were recorded on a Nicolet 5DXB FT-IR spectrometer. The NMR (^1H and ^{13}C) spectra were taken at AM 500 Bruker Spectrometer. 5 mm $^1H/^{13}C$ using CPD decoupling # 1 watt of power. Mass spectra were carried out on a VG Model 70 SQ Hybrid mass spectrometer (direct inlet, electron impact ionization). The rotations were carried out on PERKIN-ELMER 243 B polarimeter.

About 200 g of crushed, dried bark of *Alstonia boonei* were extracted separately in 2000 ml of diethyl ether in soxhlet apparatus in 24 h. After evaporating the ether, 450 mg of viscous yellow oil were obtained. The purification of each was done on florisil gel column and using ether/hexane as eluting solvent.

Compound 1 : Eluting solvent 20 % ethyl ether/hexane, TLC solvent 20 ethyl ether/hexane to give 150 mg of viscous oil qhich was diluted in 150 ml of methanol and stored in freezer for 24 h. The first solution gave 100 mg of needles solid (mp 93 - 103°C) ; $[\alpha]^{25}D = +16$ ($CHCl_3$; c=2). MS : m/z 424, 409, 313, 218, 95 and proposed Lanosta-7, 24 dien-3-one, $C_30H_{48}O$. IR (CCL_4) : ν cm^{-1} 2933.3 ; 1707.7 ; 1456.1 ; 1379.5 ; 1241.0 ; 1117.9 ; 779.4 ; 620.5. 1H NMR : 0.75 (s, 3H), 0.87 (s, 12H), 0,97 (s, 3H), 1.,60 (s, 3H), total of 8C-methyls ; 4.50 (m, 1H, H-C-OAc), 5.01 (m, 1H, HC=C) and 4.55 (d, 1H, HC=C, lateral).

Compound-2 : Eluting solvent 20 % éthyl ether/hexane, TLC solvent 20 % éthyl acetate/hexane to give 45 mg of white solid (m 140 - 150°C) ; $[\alpha]^{25}D = - 9$ ($CHCl_3$ c=1.5). MS : m/z 468, 453, 408, 393,

365, 339, 218, 189, 135, 95, 69, Lanosta-7,24-dien-3-cetate, $C_{32}H_{52}O_2$. IR (CCl_4) : ν cm-1 2933.3 ; 1733.3 ; 1450.3 ; 1379.5 ; 1241.0 ; 1117.9 ; 1025.6 ; 984.6 ; 784.6. 1H NMR : 0.75 (s, 3H, O.87 (s, 12H), O.97 (s, 3H), 1.67 (s, 3H), total of 8C-methyls ; 2.01 (s, 3H, CH_3OCO), 4.50 (m, 1H, H-C-OAc), 5.01 (m, 1H, HC=C) an 4.55 (d, 1H, HC=C, lateral).

Compound-3 : Eluting solvent 20 % ethyl acetate/hexane, TLC solvent 20 % ethyl acetate/hexane, gave an oil which crystallized in hexane in 200 mg of white solide (mp 180°C ; $[\alpha]^{25}D$ = +69.5 ($CHCl_3$; c=2) ; MS : m/z 426, 411, 393, 218, 203, 175 and proposed molecular $C_{30}H_{50}O$, β - Amyrin. IR (Ccl_4) : n cm-1 3630.5 ; 2949.7 ; 2855.6 ; 1463.8 ; 1380.3 ; 1363 ; 1030 ; 995.9. 1H NMR ($CDCl_3$) : 0.8 - 1.3 (s, 8xMe), 2.37 (ddd, J = 15.9, 6.8 and 3.4 and 3.4 Hz, H-2) and 2.55 (ddd, J = 15.9 ; 7.3 and 11.0 Hz, H-2) and 5.15 (m, 1h, HC=C).

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