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4(23)-DIHYDROROBURIC ACID FROM THE RESIN (INCENSE) OF *BOSWELLIA CARTERII*

ERNESTO FATTORUSSO,* CIRO SANTACROCE† and CABDI F. XAASAN

Faculty of Chemistry, National Somali University, P. O. Box 1081, Mogadishu Somalia

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Key Word Index—*Boswellia carterii*; Burseraceae; triterpenes; 3,4-secours-12-en-3-oic acid.

Abstract—In addition to α- and β-boswellic acids and their acetyl derivatives, 4(23)-dihydroroburic acid (3,4-secours-12-en-3-oic acid) has been isolated from the resin of *Boswellia carterii*.

INTRODUCTION

Incense, a costly resin produced by members of the Burseraceae which grow in tropical and subtropical regions, is widely used in perfumery, for religious purposes and as a folk medicine in the treatment of several diseases. It has been reported to be a rich source of terpenes, the major non-volatile constituents being cembrane diterpenoids [1–4] and ursane and oleanane triterpenoids [5–12]. Unfortunately in some papers the botanical origin of the material examined has not been indicated.

We have now investigated the acidic fraction of the incense produced by *Boswellia carterii*, a Burseracea growing mainly in North Somalia, and have found that it contains, in addition to β-boswellic acid acetate (**1**) and α-boswellic acid acetate (**2**) (previously reported as constituents of incense) and their desacetyl derivatives (**3** and **4**), 4(23)-dihydroroburic acid (**5**), a compound which has not been reported as a plant product, but has been synthesized [13].

RESULTS AND DISCUSSION

The acidic fraction from the ether-soluble material of the resin after methylation (diazomethane), was chromatographed on silica gel to give, in addition to pure **10**, mixtures A (**6** + **7**) and B (**8** + **9**). After separation by HPLC, the components of mixture A were identified on the basis of their ^{1}H NMR and mass spectra and by comparison of their physical properties with those reported in the literature [5, 6]. Mixture B was acetylated to give **6** and **7**, which were identified as above.

Compound **10** was identified by comparing its physical and spectral properties with those of an authentic sample obtained by catalytic hydrogenation of roburic acid methyl ester (**11**).

The occurrence of **5** in the exudate of *B. carterii* is of interest as 3,4-secotriterpenic acids carrying an isopropyl group at position 5 are very rare as naturally occurring compounds. Thus, until now, only casasequic and donanic acids have been isolated from fresh material [14]. 5-Isopropyl-3,4-secotriterpenic acids have been identified in sedimentary material [15], but they are considered to be degradation products originating through geochemical processes involving photochemical or photomimetic reactions.

EXPERIMENTAL

Mps: uncorr; MS: 70 eV; ^{1}H NMR: 270 MHz, $CDCl_3$, TMS as int. standard.

Extraction and purification of **6**–**10**. Incense from *B. carterii* (80 g), kindly supplied by Incense National Agency (Somalia), was extracted in a Soxhlet with Et_2O to yield an oily product (67.5 g) which was dissolved in Et_2O and extracted (× 3) with cold 2 M NaOH. The aq. layer, after acidification with conc. HCl, was extracted with Et_2O. Evaporation of the Et_2O gave 11 g of a residue which was methylated (CH_2N_2–Et_2O) and chromatographed on a silica gel column using CCl_4 as eluant with increasing amounts of Et_2O.

Fractions eluted with CCl_4–Et_2O (9:1) afforded **10** (90 mg) which was recrystallized from EtOH; mp 121–123°; $[\alpha]_D + 66°$ (*c* 1.2; $CHCl_3$); IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1730; ^{1}H NMR: δ 5.15 (1H, *m*), 3.67, 1.07, 1.04, 0.93 and 0.81 (each 3H, *s*) and 2.20 (2H, *t*, *J* = 8 Hz); EIMS *m/z*: 456 $[M]^+$, 441 $[M - Me]^+$, 425 $[M - OMe]^+$, 413 $[M - C_3H_7]^+$, 369 $[M - CH_2CH_2COOMe]^+$, 218 $[C_{16}H_{26}]^+$, arising from a retro-Diels–Alder fragmentation. Fractions eluted with CCl_4–Et_2O (4:1) afforded 3 g of a white solid. An aliquot (200 mg) was further chromatographed by HPLC on a μ Bondapack C-18 column (Waters) using MeCN–H_2O (4:1) as eluant, to give **6** (153 mg) and **7** (19 mg) which were identified by comparison of their physical properties with those reported in the literature [5, 6].

6: ^{1}H NMR: δ 5.35 (1H, *m*), 5.15 (1H, *m*), 3.68, 2.09, 1.18, 1.12, 1.04, 0.91 and 0.81 (each 3H, *s*). EIMS in addition to $[M]^+$ at *m/z* 512, showed intense peaks at *m/z* 234 and 218 arising from a retro-Diels–Alder fragmentation in ring C.

7: ^{1}H NMR: δ 5.33 (1H, *m*), 5.21 (1H, *m*), 3.66, 2.08, 1.18, 1.12, 1.03, 0.99, 0.84 (each 3H, *s*) and 0.87 (6H, *s*). EIMS was very similar to that of **6**.

Fractions eluted with CCl_4–Et_2O (7:3) afforded 1.45 g of a mixture of **8** and **9** which was recrystallized from aq. MeOH.

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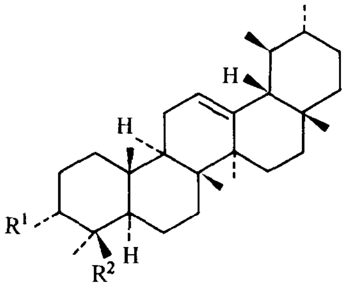
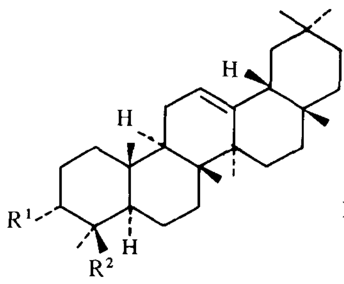
*Present address: Istituto di Biorganica, Università di Napoli, Via L. Rodinò 22, I-80138 Napoli, Italy.

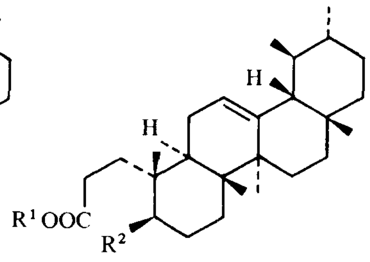
†Present address: Istituto di Chimica Organica e Biologica, Università di Napoli, Via Mezzocannone 16, I-80134 Napoli, Italy.

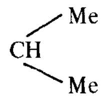
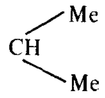
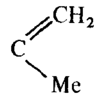


	R^1	R^2
1	OAc	COOH
3	OH	COOH
6	OAc	COOMe
8	OH	COOMe



	R^1	R^2
2	OAc	COOH
4	OH	COOH
7	OAc	COOMe
9	OH	COOMe



	R^1	R^2
5	H	 Me CH Me
10	Me	 Me CH Me
11	Me	 CH_2 C Me

After acetylation with Ac_2O–pyridine (1:1) overnight at room temp., the resulting acetates (**6** and **7**) were resolved as just described (**8** 792 mg; **9** 204 mg).

Catalytic hydrogenation of roburic acid methyl ester (**11**). **11** (100 mg) in HOAc (10 ml) was hydrogenated over PtO_2 overnight at room temp. After removal of the catalyst by filteration, the soln was evapd to dryness and the residue was chromatographed on a silica gel column using CCl_4 as eluant with increasing amounts of Et_2O. Fractions eluted with CCl_4–Et_2O (9:1) were evapd to dryness to give 76 mg of **5**.

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