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4-(*p*-HYDROXYPHENYL)-(2*S*)-BUTANOL FROM THE NEEDLES OF *TAXUS BACCATA*

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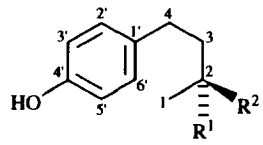
Key Word Index—*Taxus baccata*; Taxaceae; 4-(*p*-hydroxyphenyl)-(2*S*)-butanol.

Abstract—4-(4′-hydroxyphenyl)-(2*S*)-Butanol has been isolated from the needles of Himalayan Yew. Its structure has been established by spectral methods and its stereochemistry determined by comparing its optical rotation with the optical rotation of its *R* isomer.

INTRODUCTION

In the course of a chemical investigation of the needles of the Himalayan yew, *Taxus baccata*, as a source of 10-deacetylbaccatin III (10-DAB), we have isolated 4-(4′-hydroxyphenyl)-(2*S*)-butanol (**1**), the glucoside of which has been isolated from *Betula pendula* [1] and the needles of *T. baccata* and *T. brevifolia* [2, 3]. Das *et al.* [4] have obtained **1** in 65% enantiomeric excess by oxidation and enzymatic reduction of **2**.



1 $R^1 = H$; $R^2 = OH$
2 $R^1 = OH$; $R^2 = H$

RESULTS AND DISCUSSION

Compound **1** was isolated as viscous oil by CC followed by preparative TLC. Its mass spectrum showed a molecular ion peak at *m/z* 166 in accordance with the molecular formula $C_{10}H_{14}O_2$. A prominent fragmentation ion at *m/z* 148 $[M - H_2O]^+$ and IR absorption at 3351 cm^{-1} indicated that the compound contained a hydroxyl group. In addition, the IR spectrum exhibited an absorption at 1596 cm^{-1} revealing the presence of an aromatic ring in **1**. Its ^1H NMR spectrum showed the presence of a secondary methyl group (δ1.30, *d*, $J = 7.0$ Hz), two aliphatic protons (δ1.75 *m*), two benzylic protons (δ2.65 *m*), one methine proton attached to an oxygen function (δ3.85, *m*) and two pairs of *ortho* coupled aromatic protons (δ6.80, *d*, $J = 8.5$ Hz and 7.05, *d*, $J = 8.5$ Hz). The ^{13}C NMR spectrum showed the signals of 10 C atoms: one methyl, two methylenes, five methines and two tetrasubstituted carbons. The base and diagnostic peaks at *m/z* 107 due to the *p*-hydroxy benzyl cation in the mass spectrum further confirmed the structure. The ^1H NMR spectral data of both **1** and **2** showed close similarity. However, in the ^{13}C NMR spectra the chemical shifts of C-1 and C-2 were strikingly different. This could be attributed to the change in the configuration of the side-chain. This was confirmed when the optical rotation of **1** was found {$[\alpha]_D^{25}$ + 21.5° ($CHCl_3$; *c* 0.348)} to be opposite to that of **2** {lit. [4] $[\alpha]_D^{25}$ − 17.8° (EtOH; *c* 0.3151)}

EXPERIMENTAL

IR: $CHCl_3$; ^1H and ^{13}C NMR: $CDCl_3$ with TMS as int. standard; MS: Finnigan Mat-1020, Automated GC–MS.

Plant material. Needles of *T. baccata* L. were collected from the Himalayan region in Himachal Pradesh in May 1993. A voucher speciman is deposited in our laboratory.

Extraction and isolation. Air-dried and powdered needles (1 kg) were extracted with MeOH for 3 days at room temp. The mixture was then filtered and the solvent removed under red. pres. to yield a dark green extract (32 g). This extract was chromatographed over silica gel (200 g, 60–120 mesh) using Me_2CO–petrol as the elution gradient to collect six broad fractions: A (8 g), B (4 g), C (6 g), D (3.5 g), E (3 g) and F (7 g).

Fraction D (3.5 g) on repeated CC coupled with prep. TLC with $CHCl_3$–MeOH (10:1) yielded **1** (45 mg) as a viscous oil.

Compound **1**. Viscous oil, $[\alpha]_D^{25}$ + 21.5° ($CHCl_3$; *c* 0.348). IR_{max} cm^{-1}: 3351, 1596 and 1514; ^1H NMR

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(200 MHz, $CDCl_3$): δ7.05 (2H, *d*, $J = 8.5$ Hz, H-3′ and H-5′), 6.80 (2H, *d*, $J = 8.5$ Hz, H-2′ and H-6′), 3.85 (1H, *m*, H-2), 2.65 (2H, *m*, H-4), 1.75 (2H, *m*, H-3), 1.30 (3H, *d*, $J = 7.0$, Me); ^{13}C NMR (50.2 MHz, $CDCl_3$): δ 153.9 *s* (C-4′), 133.6 *s* (C-1′), 129.2 *d* (C-2′ and C-6′), 115.2 *d* (C-3′ and C-5′), 23.3 *q* (C-1), 67.7 *d* (C-2), 40.8 *t* (C-3) and 31.0 *t* (C-4); MS *m/z* (rel. int.): 166 $[M]^+$ (9), 148 (15), 133 (96), 107 (100).

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